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Abstract
Samples of the liquid fraction of digestate from 6 mesophilic anaerobic co-digestion plants after solid-liquid separation were characterised. The samples represented different types of substrates, different AD process parameters and types of solid-liquid separation devices. Fractionation of the liquid fraction of digestate was performed according to different size in coarse filtration (100 µm, 41 µm, 10 µm) followed by microfiltration (1.2 µm, 0.45 µm, 0.2 µm) and ultrafiltration (100 kDa, 10 KDa and 1 KDa). The fractions were then grouped as suspended particles (> 1.2 µm), coarse colloids (1.2-0.45 µm), fine colloids (0.45µm-1 kDa) and dissolved matter (< 1kDa). The results highlighted that COD was high in liquid fraction of digestates (9-78 g/L) with most of the COD as suspended particles (67.5% to 94.1%) and only 3.2% to 21.5% as dissolved matter. The characterisation of 5 more AD digestates are ongoing and statistical analysis will be performed to assess the correlation between the digestate characterisation and the types of substrates used, process parameters and type of solid-liquid separation used.

Keywords
Anaerobic digestion; biodegradability, digestate; liquid fraction; solid-liquid separation

INTRODUCTION

Anaerobic digester plants in Europe have increased to 14,563 plants with total installed capacity of 7,857 MWel in 2013 (European Biogas Association, 2014) generating a tremendous increase in the quantity of digestate produced. For digestate with high water content, solid-liquid separation is generally carried out in order to reduce the transportation costs of digestate. The solid-liquid separation can be done by either bow sieve, double circle bow sieve, sieve belt press, sieve drum press, press screw / auger separator, sieve centrifuge, decanter centrifuge (Burton & Turner, 2003). Solid fraction of the digestate, possibly after composting, can be used for land application as fertilizer. Few data are available on the characteristics of the liquid fraction after solid-liquid separation. However, this liquid fraction can have quite a high remaining COD content making its treatment or its disposal problematic. The objective of this research is to characterise the liquid fraction obtained after liquid-solid separation of the digestate at full scale co-digestion plants and the recalcitrant compounds produced during anaerobic digestion (AD). In particular, this study focusses on size fractionation of the liquid fraction of digestate.

Methodology

Six samples of the liquid fraction of digestate after solid-liquid separation from 6 different full-scale anaerobic digester plants all over France were investigated (fractionation and characterization). The 6 digesters were operated in mesophilic conditions but with different types of substrates, AD process parameters and type of solid-liquid separation as shown in Table 1. Coarse filtration (100 µm, 41 µm, 10 µm) followed by microfiltration (1.2 µm, 0.45 µm, 0.2 µm) and ultrafiltration (100 kDa, 10 KDa and 1 KDa) were performed on the liquid fraction of all AD digestates to get fractionation according to different sizes. The sizes were then grouped as suspended particles (> 1.2 µm), coarse colloids (1.2-0.45 µm), fine colloids (0.45 µm-1 kDa) and dissolved matter (< 1 kDa)

(Ziyang & Youcai, 2007). A dilution was first made to make COD range is between 3-7 g/L so better filtration can be performed. 3D Fluorescence spectroscopy was performed to determine the complexity of the liquid fraction of AD digestates in soluble phase. Fluorescence regionalisation integration for spectra interpretation and quantification were according to: 1) Protein-like (Tyrosine, Tyr); 2) Protein-like (Tyrptophane, Trp); 3) Protein-like (Tyr and Trp, microbial products); 4) Fulvic acid-like; 5) Inner filter, glycolated protein-like; 6) Melanoidin-like; lignocellulose-like; 7) Humic acid-like (Jimenez et al., 2014). Fluorescence 1, 2 and 3 are grouped as low complexity while 4, 5, 6 and 7 as high complexity. Other parameters were analysed according to standard methods for the examination of water and wastewater (Rice et al., 2012).

Table 1. Types of substrates, AD process parameters and type of solid-liquid separation of the full scale AD plants.

	Types of Substrates	Retention time (days)	Size of reactor (m^3)	Feeding (tons/day)	Methane production (m^3/day)	Type of solid-liquid digestate separation
A	Sludge (22%), septage (14%), manure (16%), shrinkage (7%), fat (22%), blood (7%), pet food (5%), fruits and vegetables waste (7%)		2800	120		Screw Press
B	cattle manure (60%), pig slurry (20%), cereals (10%), fats (8%), rainwater (2%)	60	1370	15	1500-1600	Screw Press
C	Fruits and vegetables waste					Drum Filter
D	Horse manure	60	-	batch	-	Centrifugation
E	Sludge (40%), Fats from restaurant (30%), waste from food industries (30%)	37	3300	90	4500	Centrifugation
F	Liquid cattle manure (49.7%), raw cattle manure (2%), poultry manure (8%), catch crops and vegetable products (14.5%), cereal residues (10.9%), fruits and vegetables waste (10.9%), grass clippings (4%)		1206	15	1230	

Results and Discussion

Characterisation of AD digestate. Table 2 shows the characterisation of raw, solid fraction and liquid fraction of AD digestate. In raw digestate, total solids were in the range 14 – 108 g TS/kg depending on the type of substrates used, the type of process and the operating conditions. Correlations between these parameters and characteristics of the digestate are under investigation. B has the highest TS and VS due to manure and slurry used as main substrates, followed by F, A, E and C. C has the lowest TS and VS due to fruits and vegetables used as feed. Solid liquid separation generated a solid phase with high TS with values in the range 173 – 310 g TS/kg. F had the highest TS, followed by E, A, B and C. Different types of solid-liquid separation and their efficiency could have contributed to the value of TS in the solid fraction of the digestate and this will be further investigated too. B which uses mostly manure and slurry as feedstock has the highest COD with 78 ± 4 g/L followed by F, D, A, E and the lowest is C, with 9.2 ± 0.1 g/L, which used fruits and vegetables, the most biodegradable substrate of the study. VFA was not found in any samples confirming no overloading in the digesters. The pHs of liquid fraction of the digestates are between 7.88 to 8.42. In soluble phase, all liquid fraction of the digestate showed the presence of sodium (0.08 to 3.21 ± 0.01 g/L), ammonia (0.61 ± 0.01 to 3.52 ± 0.02g/L), potassium (1.64 ± 0.04 to 7.39 ± 0.01), magnesium (0.03 to 0.30g/L), calcium (0.04 to 0.53 ± 0.01 g/L), chlorine (0.72 to 4.09 ± 0.02 g/L), phosphate (0 to 2.17 ± 0.05 g/L) and sulphate (0.01 to 0.84 ± 0.03 g/L). No nitrate nor nitrite was detected.

Table 2. Characterisation of raw, solid fraction and liquid fraction of the digestates

	A	B	C	D	E	F
Raw Digestate						
Total Solids (TS) (g/kg)	71 ± 1	108 ± 1	14.36 ± 0.03	-	55.85 ± 0.03	105 ± 2
Volatile Solids (VS) (g/kg)	43 ± 1	74.07 ± 0.01	5.38 ± 0.03	-	27.35 ± 0.02	64 ± 2
VS/TS	0.61	0.69	0.37	-	0.49	0.61
Solid fraction of digestate after solid-liquid separation						
TS (g/kg)	235 ± 2	229 ± 12	173 ± 3	-	238.9 ± 0.1	310 ± 1
VS (g/kg)	208 ± 1.2	183 ± 6	161.1 ± 1.3	-	117 ± 2	243 ± 2
VS/TS	0.89	0.8	0.93	-	0.49	0.79
Liquid fraction of digestate after solid-liquid separation						
TS (g/kg)	56.85 ± 0.02	80.9 ± 0.2	13.70 ± 0.1	51 ± 1	16.68 ± 0.04	82.7 ± 0.1
VS (g/kg)	31.9 ± 0.1	52.3 ± 0.2	4.7 ± 0.2	35.54 ± 0.04	7.49 ± 0.03	44.5 ± 0.1
VS/TS	0.56	0.65	0.35	0.7	0.45	0.54
Total Alkalinity (g/L $CaCO_3$)	24.8 ± 0.03	17.07 ± 0.04	7.40 ± 0.04	15.5 ± 0.2	21.5 ± 0.1	23.6 ± 0.4
Initial COD (g/L)	47 ± 1	78 ± 4	9.2 ± 0.1	69.3 ± 0.4	12.1 ± 0.3	70 ± 1
Initial Turbidity (NTU)	43300	51400	6160	13200	3780	49400
Soluble Total Organic Carbon (sTOC) (g/L)	0.78	3.11 ± 0.02	0.90	3.53 ± 0.09	3.91 ± 0.05	3.45
Inorganic Carbon (IC) (g/L)	2.95 ± 0.01	2.81 ± 0.01	1.36	2.95 ± 0.04	3.32 ± 0.01	3.72 ± 0.03
sCOD/sTOC (gCOD/gTOC)	3.5	3.04	0.7	3.3	1.0	3.7

Fractionation of liquid fraction of digestate. Figure 1-a shows that COD highly decreased after 1.2µm filtration for all the samples, indicating the presence of a high quantity of suspended solids. Indeed, 67.5% to 94% of the COD was in the form of suspended particles. 0% to 1% of the COD was in coarse colloids, 1.8% to 13.8% in fine colloids and 3.2% to 21.5% in dissolved matter. Previous study also found 10-20% of COD in dissolved matter of the digestate of a 2-stage anaerobic digestion process treating a mixture of cow manure, grass cuttings and fruits and vegetable (Ganesh et al., 2013).

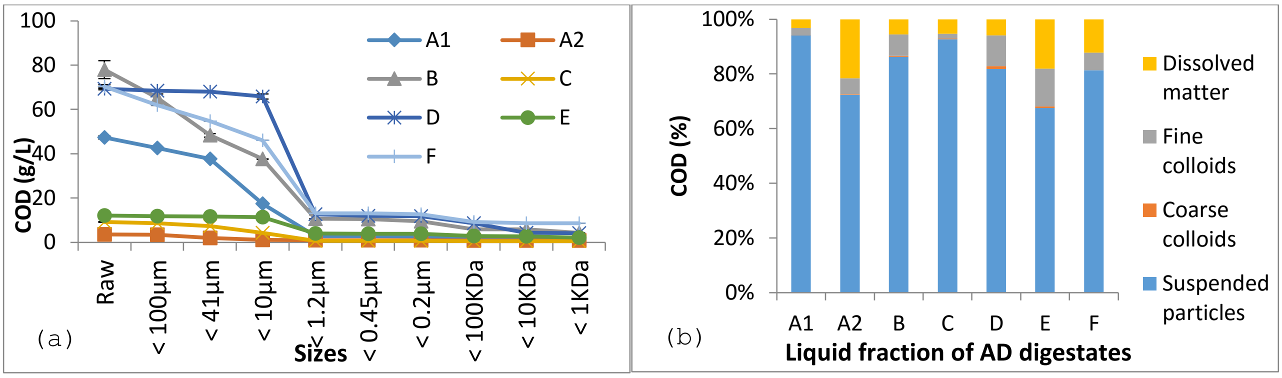

Figure 1. COD fractionation of liquid fraction of AD digestate (a) and COD percentage (%) in different group sizes (b).

3D Fluorescence spectroscopy analyses. 3D fluorescence spectra of soluble phase showed that fluorescence ratio of low complexity/high complexity compounds was around 50/50 for all samples, except B (37/63) and E (55/45). Once again the highest complexity of soluble compounds might be related to the presence of poorly biodegradable substrates such as manure and slurry.

Biodegradability. BOD_{21} tests are ongoing for liquid fraction of the digestate to determine the biodegradability of the samples.

Conclusion

COD (g/L) values were found high in liquid fraction of full scale AD digestate (9-78g/L) mostly under the form of suspended particles with: 67.5% to 94% of COD in suspended particles, 0% to 1% in coarse colloids, 1.8% to 13.8% in fine colloids and 3.2% to 21.5% in dissolved matter. The characterisation of 5 more AD digestate are ongoing and statistical analysis will be performed to determine the origin of remaining COD and the impact of different types of substrates used, AD process parameters and type of solid-liquid separation used. The removal of these compounds which are recalcitrant in the AD process will be further studied by combined physico-chemical and biological treatments.

ACKNOWLEDGEMENTS

Authors would like to thank Majlis Amanah Rakyat (MARA) Malaysia for the scholarship received to conduct this research.

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