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# Comprehensive characterization of the liquid fraction of digestates from full-scale anaerobic co-digestion

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ABSTRACT

Waste management by anaerobic digestion generates a final byproduct, the digestate, which is usually separated into solid and liquid fractions to reduce the volume for transportation. The composition of the solid fraction has been recently studied to allow its valorization. However, full composition of liquid fraction of digestate and its size fractionation are less considered in the literature for efficient post treatment and valorization purposes. Therefore, here we characterized in detail liquid fraction of digestate obtained after solid-liquid separation from 11 full-scale co-digestion plants. The liquid fraction has a high concentration in organic matter with Chemical Oxygen Demand (COD) from 9.2 to 78 g/L with 60–96% of COD in suspended particles (>1.2 μm), 2–27% in colloids (1.2 μm to 1 kDa) and 2–18% in dissolved matter (<1 kDa). Besides, it contained from 1.5 to 6.5 g/L total nitrogen and high ions concentrations (0.5–3.1 g/L $NH_4^+$, 1.05–5.48 g/L $K^+$, 0–2.13 g/L $PO_4^{3-}$). In addition, liquid fraction of digestate has poor biodegradability due to presence of humic substances making aerobic treatment inefficient. Only physico-chemical post treatment can be proposed for organic matter removal.



## 1. Introduction

Anaerobic digestion (AD) of solid waste has gained a lot of interest, and achievements in applications for biogas and renewable energy production stimulating new research areas. This is due to feasibility of AD for a broad type of waste, such as livestock manure, lignocellulosic biomass (crop residues), food waste, waste activated sludge, organic fraction of municipal solid waste (OFMSW), fruit and vegetable waste, industrial waste and bio-waste as summarized in a review by Mao et al. and Mata-Alvarez et al. (Capson-Tojo et al., 2016; Mao et al., 2015; Mata-Alvarez et al., 2014). As a result, there has been a strong development of AD plants all around the world, especially in China, with 26.5 million plants in 2007 (Mao et al., 2015), Europe with 17,240 plants in 2014 (European Biogas Association, 2015) and USA with 1497 plants in 2013 (Edwards et al., 2015). High difference in number of biogas plants in Europe and USA was due to the political will particularly on the feed-in-tariffs for electricity from biogas in Europe (Torrijos, 2016). Increasing number of AD plants means simultaneous increase of the quantity of the final byproduct, the digestate. Digestate withdrawn from anaerobic digester is a liquid to thick slurry that contains a significant quantity of remaining solids. Tremendous volumes of digestate produced daily can become a major problem for local and regional transportation of digestate as it consumes huge amount of fuel oil (Rehl and Müller, 2011). Therefore, the solid-liquid separation of raw digestate is often performed on-site (Delzeit and Kellner, 2013). The most common solid-liquid separation applied in full-scale plants are screw press, screening drum press (vibrating screen) and centrifuge (Al Seadi et al., 2013; Delzeit and Kellner, 2013). Solid fraction of digestate has gained more interest in research and development. They can be composted or applied directly as organic fertilizer (Tambone et al., 2015; Zeng et al., 2015). In recent research, new routes have been proposed for solid digestate valorization (Monlau et al., 2015a,b) such as production of bio-fuel for use in domestic furnaces (Pedrazzi et al., 2015), production of biochar (Monlau et al., 2015a,b; Stefaniuk and Oleszczuk, 2015) as well as post treatments for methane recovery (Sambusiti et al., 2015). For liquid fraction of digestates, generally they contain very low residual biogas potential (Gioelli et al., 2011) but high concentration of COD (Ganesh et al., 2013, 2014; Li et al., 2015; Xia and Murphy, 2016). Total Nitrogen (TN) and ammonia nitrogen (Xia and Murphy, 2016) as well as nutrient concentrations can limit digestate application into soils as reported in the European Nitrates Directive (Pedrazzi et al., 2015). The disposal of liquid fraction of digestate by land application can generate issues such as nitrogen leaching (Svoboda et al., 2013) and infiltration into the

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groundwater, polluting nearby rivers and affecting aquatic life. The interests of research on liquid fraction of digestates have rapidly increased recently such as microalgae cultivation with liquid digestates (Xia and Murphy, 2016), ammonia stripping and vacuum evaporation (Li et al., 2016) vacuum thermal stripping for ammonia recovery (Ukwuani and Tao, 2016), liquid digestate treatment with application of direct contact membrane distillation process (Kim et al., 2016), struvite recovery (Tao et al., 2016), liquid digestate treatment to produce fertilizer products with low water and concentrated nutrient contents (Tampio et al., 2016a), liquid digestate recirculation in continuous stirred tank processes (Peng et al., 2016) and anaerobic biogasification of corn stover with biological pretreated liquid fraction of digestate (Hu et al., 2015). However, they only focused on nutrients removal. Besides, the full composition of liquid fraction of digestates in the literature is less considered. Full composition in liquid fraction of digestate is necessary to understand for efficient post treatment and valorization. For example, high solids content in liquid fraction of digestate which contributed to dark color and high turbidity cannot be used for algae cultivation due to the necessity of light penetration for algae growth (Marcilhac et al., 2014). Therefore, on-site treatment of liquid fraction of digestate is necessary before cultivating with microalgae for an efficient nutrient removal and reducing the nutrient cost for cultivation (Xia and Murphy, 2016). Proper management of the liquid fraction of digestate will be improved when thorough knowledge of this effluent composition is available. Besides, liquid fraction of digestates can vary from one to another. This could be due to the origins of substrates, operating parameters used in the digester and type of solid-liquid separation. Hence, the objective of this study was to characterize the liquid fraction of digestate obtained after solid-liquid separation from full-scale co-digestion plants treating different types of substrates, with different process parameters and different separation processes. In particular, this study focused on size fractionation of the liquid fraction of digestate, to quantify the contribution of suspended particles, colloids and dissolved matter fraction on the physico-chemical and biological parameters.

## 2. Materials and methods

### 2.1. Digestate collection

Raw digestates, solid and liquid fractions of digestates after separation were collected from 11 full-scale co-digestion plants treating solid wastes. Nine AD plants were operated in continuous stirred tank reactor (CSTR) digesters at mesophilic condition (35 °C). The two other ones were operated using plug-flow (PF) digesters at thermophilic condition (55 °C). The plants were operated at different operating parameters: feeding (11–43 kg of raw substrate/day/$m^3$) and hydraulic retention time (HRT) (24–80 days). The digestates were separated on-site into solid and liquid fractions either by screw press, centrifugation or vibrating screen. Data of the industrial plants are shown in Table 1. In order to have representative samples, plants with different types of co-substrates fed were selected (Table 1). In this study, raw digestate and solid fraction of digestates were collected for determination of total solids (TS) and volatile solids (VS) contents in order to have some information on the efficiency of on-site solid-liquid separation. Only liquid fractions were characterized in detail.

### 2.2. Sample filtration for size fractionation

Dilution with milliQ water was performed in order to facilitate filtration and prevent a rapid clogging of the filters. The dilution factor varied from 0 to 1/20 and was chosen based on COD range from 1 to 5 g/L. 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, 1 kDa) were successively performed on liquid fraction of digestates at room temperature in order to have full understanding of different sizes. Permeates of each size were collected and stored at 4 °C for characterization. The sizes were then grouped as suspended particles (>1.2 μm), coarse colloids (1.2–0.45 μm), fine colloids (0.45 μm to 1 kDa) and dissolved matter (<1 kDa) (Ziyang and Youcai, 2007) to have simplified sizes fractions.

### 2.3. Chemical analysis

pH was measured using WTW series inoLab pH720 calibrated with pH 4 and pH 7 buffer solutions. Alkalinity was performed by 0.1 N hydrochloric acid titration as described elsewhere (APHA, 2012). Total solids (TS), volatile solids (VS) and mineral solids (MS) were determined using standard methods (APHA, 2012). Chemical Oxygen Demand (COD) was analyzed using commercial Aqualytic 420721 COD Vario Tube Test MR 0–1500 mg/L. Total Kjeldahl Nitrogen (TKN) was determined by Buchi AutoKjeldahl Unit K-370 after mineralization with BUCHI Digest Automat K-438. Ammonium ($NH_4^+$) was determined in dissolved matter with Buchi AutoKjeldahl Unit K-370. Total Organic Carbon (TOC) and Inorganic Carbon (IC) were measured in dissolved matter using Shimadzu TOC-$V_{CSN}$ Total Organic Carbon Analyzer with Shimadzu ASI-V auto sampler as described elsewhere (Battimelli et al., 2010). Metabolites and simple sugars (carboxylic acids, sugars and alcohols, i.e. saccharose, lactose, glucose, pyruvate, xylose, arabinose, succinate, lactate, glycerol, formate, fumarate, acetate, 1,3-propanediol, propionate, ethanol and butyrate) were measured in dissolved matter using HPLC at a flow rate of 0.7 mL/min as described elsewhere (Moscoviz et al., 2016). Cations and anions were analyzed in dissolved matter using ion chromatograph (ICS 3000, Dionex, USA) as described elsewhere (Uggetti et al., 2014). Volatile fatty acids (VFA) including acetic, propionic, isobutyric, butyric, isovaleric, valeric, isocaproic, caproic and enanthic acids concentrations were measured in dissolved matter using Perkin Elmer Clarus 580 Gas Chromatograph as previously described (Cazier et al., 2015). All the chemical analyses were performed in duplicate.

### 2.4. Physical analysis

Particle size distribution (in the range 0–2000 μm) was analyzed using Beckman Coulter LS200 granulometer as described elsewhere (Ganesh et al., 2013). Turbidity was performed on all permeates using HACH portable turbidimeter model 2100P with formazin for standard calibration. Absorbance spectra were measured in dissolved matter using a UV-2501PC UV–vis spectrophotometer as described elsewhere (Battimelli et al., 2010). The ratio $SUVA_{254}$ (A254/TOC) of the specific UV absorbance at wavelength 254 nm to the dissolved total organic carbon (TOC) concentration is an indicator of the aromatic carbon content in the dissolved organic matter as well as the degree of humification. This ratio is also linked to biological degradability (Zheng et al., 2014). 3D Fluorescence spectroscopy analysis was performed in dissolved matter using a Perkin Elmer LS55 fluorescence spectrometer. Fluorescence spatialization integration for spectra interpretation and quantification was 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) glycolated protein-like; (6) Melanoidin-like; lignocellulose-like; (7) Humic acid-like (Jimenez et al., 2014). Fluorescence 1, 2 and 3 were grouped as protein-like. The conductivity was measured in dis-

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