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Physical-chemical traits, phytotoxicity and pathogen detection in liquid anaerobic digestates

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ABSTRACT

Anaerobic digestates, which are co-products from biogas production, have been recognised as potential biofertilisers for their benefits in nutrient recovery and recycling of different types of organic wastes. Due to the increasing number of different types of organic wastes being used to produce biogas, it is necessary to identify how different types of anaerobic digestates vary in their physical-chemical traits, and how these can impact upon their use as fertilisers. In addition, safe land spreading of anaerobic digestates must be within recommended limits for potentially toxic elements (PTEs) and pathogens. This study analysed physical-chemical traits, phytotoxicity, PTEs and indicator pathogens in a set of eleven different commercial liquid anaerobic digestates from Ireland and the UK, and compared them to the Irish draft standard for digestate. Liquid anaerobic digestates exhibited significant differences ($P < 0.001$) for most of the physical and chemical traits evaluated, with higher variability found for dry matter (DM) and K ($CV = 17.2$ and 16.8 respectively), and lower variation for pH and P ($CV = 1.78$ and 3.55 respectively). PTE concentrations were in general within recommended limits; nevertheless, some digestates showed higher concentrations than the recommended limits for Pb, Zn and Cu. Digestate from wastewater treatment feedstock was shown to be high in PTEs. Anaerobic digestates were found to negatively affect early stages of seed germination, but phytotoxicity effects were decreased by dilution in water. Levels of *Salmonella* spp. and *E. coli* were within recommended limits for most of the anaerobic digestates analysed.

1. Introduction

The use of renewable energy derived from biogas has risen all over the world stimulated by benefits such as the generation of green energy, low-cost treatment for organic wastes from households, industry and agriculture, reduction of GHG emissions from organic waste degradation, associated methane capture from biological systems, and co-production of potential biofertilisers (Mao et al., 2015). These resulting residues, known as anaerobic digestates, are rich in nutrients and have been recognised as potential sustainable alternatives to conventional inorganic and other undigested organic fertilisers (Tambone et al., 2010; Alburquerque et al., 2012; Möller and Müller, 2012; Walsh et al., 2012a).

The utilisation of anaerobic digestates as fertiliser still faces many challenges in terms of uses such as land spreading, due to a broad range of physical-chemical compositions (Alburquerque et al., 2012; Möller and Müller, 2012; Nkoa, 2014), which make it difficult to establish standard management practices such as fertilisation rates. Physical-chemical and microbiological traits of anaerobic digestate depend on several factors; however, most can be attributed to the type of feedstock utilised (Amani et al., 2010), pre-treatment of the feedstock (Appels et al., 2008), the effect of physical-chemical traits of the feedstocks used for digestion on the activity of microbial community within the reactor (Dai et al., 2016), and post-treatment and storage after digestion (Pell Frichmann consultants, 2012). Differences between anaerobic digestates directly impact the management practices related to them.

In Europe, many different regulations and guidelines for anaerobic digestate production and use can be found (Holm-Nielsen et al., 2009). In the United Kingdom (UK), the utilisation of anaerobic digestates is subjected to environmental permitting or licenses (BSI, 2010). In Ireland, the Irish Bioenergy Association, in consultation with industry, has developed a draft standard for anaerobic digestate use (IrBEA, 2012), based on reviews of standards and quality assurance throughout Europe. These standards deal with environmental impacts, health risks and waste management practices. In order to develop useful standards, it is necessary

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to better understand how different types of anaerobic digestates vary in their physical-chemical and microbiological composition. Such information can lead to improvements in the regulations about their use and land spreading, and also improve agriculture management practices related to them.

The aim of this study was to analyse physical-chemical traits, total nutrients, PTEs, phytotoxicity and indicator pathogens in a set of eleven different types of commercial liquid anaerobic digestates, and compare them to the concentrations recommended in the draft Irish digestate standards.

2. Material and methods

2.1. Digestate sampling

Liquid anaerobic digestate samples from eleven different types of biogas plants were collected from Ireland and United Kingdom in October 2015 (Table 1). DM, ODM, pH, EC and N analyses were carried out within four days of sampling. For microbial analysis, all anaerobic digestate samples were kept refrigerated at 4 °C and analysed a maximum of of one week from sampling. Samples for elemental composition and PTEs were kept at −20 °C and processed within two months. Samples were analysed according to the methods outlined in the draft IrBEA industry standard for digestate (IrBEA, 2013). They were prepared in accordance with European standard EN 16179 (2012). For elemental analysis, samples were air-dried at 40 °C until a constant weight.

2.2. Physical-chemical, elemental composition and PTEs

For DM analysis, samples were oven dried at 105 °C according to European standard EN 13040 (2007). The organic dry matter (ODM) was determined by loss on ignition according to European standard EN 15935 (2012). Total organic carbon (TOC) was calculated based on the OM analysis, estimating that TOC was approximately 58% of the OM (Bernal et al., 1998). Total Kjeldahl nitrogen (TKN) was measured using a Buchi Kjeldahl apparatus according to European standard EN 16169 (2012). The C/N ratio was calculated using the ratio of the TOC and the TKN. For pH and electrical conductivity (EC), samples were extracted with deionised water at a ratio of 1:5 (v/v) according to European standard EN 15933 (2012). pH was measured with by probe (Mettler Toledo, Switzerland). After pH measurement, samples were centrifuged at 4500 rpm for 10 min, then the supernatant was filtered and measured for EC using a probe (CON-700, EUTECH), according to CEN/TS 15937 (2013). Total concentrations of the following chemical elements (P, Ca, K, Mg, Na, Mn, B, Co, Se, Al, Fe) and PTEs (Cd, Cr, Cu, Pb, Ni, Zn) were analysed using ICP-OES (Inductively Coupled Plasma-Optical Emission Spectrometry) VARIAN model 710-ES, according to guidelines of CEN/TS 16170 (2012). The extracts analysed were produced after total digestion of dried anaerobic digestates (Section 2.1) in aqua regia (6 ml HCl + 2 ml HNO_3) using a microwave digester (Mars 240/50, CEM) in accordance with the guidelines described in European standard EN 16174 (2012). For Hg, samples were sent to an external laboratory and analysed using ICP-MS.

2.3. Phytotoxicity

Seed germination assays were carried out by adapting the methodology described by Alburquerque et al. (2012). Seed germination tests were performed in square Petri dishes, where two filter papers moistened with 1 ml of solution served as an environment for seed germination. Ten cress (*Lepidium sativum*) seeds were sown in between filter papers. The dishes were sealed with parafilm and incubated in darkness at 23 °C for 72 h. Anaerobic digestates were diluted with deionised water to solution concentrations of 10%, 25%, 50%, and 100%. After incubation, the number of germinated seeds was noted, and germination was calculated as a percentage of the control (deionised water).

2.4. Detection of pathogens

Salmonella spp. were enumerated in digestate samples by enrichment in selenite-cystine broth, followed by most-probable-number (MPN) analysis using Rappaport-Vassiliadis broth, and confirmed by streaking on XLD and Rambach agar, in accordance with CEN/TR 15215-2 (2006). *Escherichia coli* were enumerated by most-probable-number (MPN) analysis in Fluorocult lauryl sulphate broth confirmed by Kovac's reagent, according to CEN/TR 16193 (2013).

2.5. Statistical analysis

Physical-chemical trait data were tested for normality and equal variance (Levene's test), and analysed using one-way ANOVA. Seed germination data were analysed by descriptive statistics. Phytotoxicity was correlated with physical-chemical traits of anaerobic digestates using Pearson's correlation test ($P > 0.05$ and $P > 0.01$) in SPSS. Magnitudes of correlation follow: if $|r| < 0.20$, non-existent correlation; $0.20 < |r| < 0.40$, weak correlation; $0.40 < |r| < 0.60$, moderate correlation; $0.60 < |r| < 0.80$ strong correlation; if $|r| > 0.80$ very strong correlation. Relationships among physical-chemical characteristics were analysed by principal component analysis (PCA) using XLSTAT (Addinsoft Software).

3. Results and discussion

3.1. Physical-chemical traits

All traits related to organic matter (DM, ODM, N, C, TOC, C/N) exhibited significant differences between anaerobic digestates

Table 1
Feedstock composition and operational aspects of biogas plants supplying the set of anaerobic digestates evaluated. HRT = Hydraulic retention time.

Digestate	Feedstock	Operation	Temperature (°C)	HRT (days)	Volume (m^3)	Pasteurisation
AD1	Food waste (dairy industry)	Continuous	Mesophilic	70	1200	Pre-digestion
AD2	Food waste, pig slurry	Continuous	40	90	2000	Post-digestion
AD3	Food waste (farm and food)	Continuous	38	54	600	No
AD4	Food waste, municipal sludge	Continuous	37–42	60	1850	Post-digestion
AD5	Waste water treatment	Batch	Mesophilic	14	1700	Pre-digestion
AD6	Food waste, garden waste	Continuous	Mesophilic	26	5200	Pre-digestion
AD7	Whole cattle slurry	Continuous	27	22	220	No
AD8	Whole grass	Continuous	40	70	0.2	No
AD9	Cattle slurry, chicken manure, food waste	Continuous	Mesophilic	40	265	No
AD10	Whole cattle slurry	Continuous	Mesophilic	40–50	870	No
AD11	Food waste (kitchen), garden waste	Continuous	Mesophilic	70	0.2	No