Is anaerobic digestion effective for the removal of organic micropollutants and biological activities from sewage sludge?

L. Gonzalez-Gila, 1, M. Papa a, b, 1, D. Ferettic, f, E. Cerettic g, G. Mazzolendid, f, N. Steinbergd, R. Pedrazzanie, f, G. Bertanzab, f, J.M. Lema a, M. Carballaa, *  

a Department of Chemical Engineering, School of Engineering, Universidad de Santiago de Compostela, Rúa Lope Gómez de Marzoa, E-15782 Santiago de Compostela, Spain  
b Department of Civil, Environmental, Architectural Engineering and Mathematics, University of Brescia, via Branze 43, I-25123 Brescia, Italy  
c Department of Medical and Surgical Specialties, Radiological Sciences and Public Health, University of Brescia, Viale Europa 11, I-25123 Brescia, Italy  
d Department of Clinical & Experimental Sciences, University of Brescia, Viale Europa 11, I-25123 Brescia, Italy  
e Department of Mechanical and Industrial Engineering, University of Brescia, via Branze 38, I-25123 Brescia, Italy  
f Brescia University Research Center “Integrated Models for Prevention and Protection in Environmental and Occupational Health” (MISTRAL), Italy

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Abstract  
The occurrence of emerging organic micropollutants (OMPs) in sewage sludge has been widely reported; nevertheless, their fate during sludge digestion remains unclear. The objective of this work was to study the fate of OMPs during mesophilic and thermophilic anaerobic digestion (AD), the most common processes used for sludge stabilization, by using raw sewage sludge without spiking OMPs. Moreover, the results of analytical chemistry were complemented with biological assays in order to verify the possible adverse effects (estrogenic and genotoxic) on the environment and human health in view of an agricultural use of digested sludge. Musk fragrances (AHTN, HHCB), ibuprofen (IBP) and triclosan (TCS) were the most abundant compounds detected in sewage sludge. In general, the efficiency of the AD process was not dependent on operational parameters but compound-specific: some OMPs were highly biotransformed (e.g. sulfamethoxazole and naproxen), while others were only slightly affected (e.g. IBP and TCS) or even unaltered (e.g. AHTN and HHCB). The MCF-7 assay evidenced that estrogenicity removal was driven by temperature. The Ames test did not show point mutation in Salmonella typhimurium while the Comet test exhibited a genotoxic effect on human leukocytes attenuated by AD. This study highlights the importance of combining chemical analysis and biological activities in order to establish appropriate operational strategies for a safer disposal of sewage sludge. Actually, it was demonstrated that temperature has an insignificant effect on the disappearance of the parent compounds while it is crucial to decrease estrogenicity.

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1. Introduction  
A great number of organic micropollutants (OMPs) enters sewage treatment plants (STPs), including pharmaceuticals, personal care products, steroid hormones, industrial chemicals, pesticides and many others (Luo et al., 2014). Sludge is the endpoint of most hydrophobic pollutants through sorption (Carballa et al., 2008), but also of an important fraction of hydrophilic OMPs not biotransformed during the wastewater treatment. The concentrations of OMPs in sewage sludge are much dependent on their physicochemical characteristics and usage rates, varying strongly among countries or even the STPs. In general, hydrophobic substances, such as triclosan (TCS) and musk fragrances, are detected at important concentrations (up to 10,000 μg/kg), while much lower levels (10–100 μg/kg) are measured for hydrophilic pharmaceuticals, as diclofenac (DCF), trimethoprim (TMP), ibuprofen (IBP), naproxen (NPX), carbamazepine (CBZ) or sulfamethoxazole (SMX) (Stasinakis, 2012).

The presence of emerging pollutants in aquatic environments has already been considered by the Water Framework Directive, which establishes a “Watch List” and a priority list of substances. In this sense, some OMPs, such as DCF, 17α-ethinylestradiol (EE2), 17β-estradiol (EE2), and...
The main aim of this work was to combine chemical and biological methods in order to evaluate the fate of OMPs and the estrogenic and genotoxic activities during mesophilic and thermophilic sludge digestion, at environmentally relevant concentrations (no OMPs spike was performed). To the best of our knowledge, this is the first study conducting an integrated assessment of the biotransformation of OMPs and these specific toxicities to evaluate the effectiveness of different AD strategies.

2. Materials and methods

2.1. Organic micropollutants

20 compounds commonly used in daily life were considered in this study: three musk fragrances, galaxolide (HHCB), toalalide (AHTN) and celestolide (ABDI); three anti-inflammatories, ibuprofen (IBP), naproxen (NPX) and diclofenac (DCF); four antibiotics, sulfamethoxazole (SMX), trimethoprim (TMP), erythromycin (ERY) and roxithromycin (ROX); four neurodrugs, fluoxetine (FLX), carbamazepine (CBZ), diazepam (DZP) and citalopram (CTL); three endocrine disrupting compounds, triclosan (TCS), 4-ocytlyphenol (NP) and 4-nonylphenol (NP); and three hormones, estrone (E1), 17β-estradiol (E2) and 17α-ethinyloestradiol (EE2).

2.2. Sewage sludge

A mixture of primary and secondary sludge (70/30, v/v), coming from the thickener and the activated sludge floater of a nearby STP in Santiago de Compostela (Spain), was used. The STP is designed for 184,000 population equivalent with an average wastewater flowrate of approximately 55,000 m³/d, which is mainly composed by domestic wastewater (hospital discharges represent 1–2% of the total flowrate). The characteristics of the mixed sewage sludge were almost stable along the experimental period (330 d). The average pH was 5.4 ± 0.3, the total and soluble chemical oxygen demands (COD) were correspondingly 34.5 ± 5.9 g/L and 2.9 ± 1.0 g/L, the average concentration of total (TS) and volatile (VS) solids were 28.8 ± 5.5 g/L and 22.3 ± 4.1 g/L respectively, and the content of volatile fatty acids (VFA) was 2.1 ± 0.9 g/L. More differences would be expected regarding the season, because of rainfalls, but it seems that the main factor affecting the sludge characteristics was the operation of the STP. The measured values are in accordance with previously reported data for sewage sludge coming from the same STP (Carballa et al., 2007).

2.3. Lab-scale anaerobic digesters and monitoring campaigns

Two continuously stirred (IKA RW20, 150 rpm) tank reactors with a total volume of 15 L (liquid volume of 13 L) were operated in parallel conditions, except for the temperature. One digester was mesophilic (MAD, 37 °C) and the other one thermophilic (TAD, 55 °C). The reactors were inoculated with biomass from a STP mesophilic anaerobic digester (sludge retention time (SRT) of 25–30 d) and operated semi-continuously by feeding the sludge mixture manually every day. The operation of the digesters can be divided into three periods: start-up (days 0–15) with an organic loading rate (OLR) of below 1 g COD/L d and a SRT of 40 d; Period I (days 15–90) was characterized by a SRT of 30 d and an average OLR of 1.1 g COD/L d; and Period II (days 90–330), with a SRT of 20 d and an OLR of around 1.8 g COD/L d. Conventional parameters of raw and digested sludge were analysed twice a week to check the performance of both reactors.

In order to evaluate the fate of OMPs and the estrogenic and the genotoxic activities during different AD conditions, two monitoring campaigns were conducted: one during Period I (days 71–77, spring 2014) and the second one during Period II (days 267–273, autumn 2014). Two samples of sewage sludge and digestates from MAD and TAD were taken in different days of each sampling campaign. The samples were immediately centrifuged at 3500 rpm.
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