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From sewage sludge and agri-food waste to VFA: Individual acid production potential and up-scaling

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ABSTRACT

Volatile fatty acid (VFA) production through anaerobic fermentation may constitute an innovative solution for organic waste management within the context of circular economy. In the present study, the evolution of individual VFA during laboratory-scale fermentation of sewage sludge (SS), winery wastewater (W_{ww}) and meat and bone meal (MBM) was assessed, focusing on the effect of pH (5.5 and 10) and temperature (35 and 55 °C). Up-scaling of the fermentation process was evaluated in batch operation. The latter showed that specific VFA could be produced, giving similar individual evolution to lab-scale testing. To be precise, acetic acid percentage ranged within 30–65% and increased up to 5900 mg O_2 L^{-1} during SS fermentation at 55 °C and pH 9. In addition, 60% butyric acid was reached during W_{ww} acid fermentation at 55 °C, which corresponded to 6670 mg O_2 L^{-1} concentration in the fermentation broth. Regarding valeric acid, over 20% proportion and 2700 mg O_2 L^{-1} were reached in MBM acid fermentation at 35 °C. Finally, iso-valeric maximum level ranged within 15–17% in SS alkaline fermentation at 55 °C, which represented a concentration close to 2000 mg O_2 L^{-1}. Interestingly, co-fermentation of agri-food waste and SS at thermophilic temperature and alkaline pH, boosted the VFA concentration 1.7–2 fold, which suggests that anaerobic co-fermentation of substrates from different nature could give promising outcomes in full-scale operation.

1. Introduction

In the last decades, modifications in environmental legislation in the European Union have promoted sewage sludge valorisation in urban wastewater treatment plants (WWTP). Consequently, anaerobic digestion (AD) has been widely implemented as an on-site management solution for sludge. Nonetheless, it is still quite common the final disposal of sludge in landfills or land application to occur without stabilization, without hygienization or without recovering energy. The latter contradicts the recently published "Closing the Loop – an EU action plan of the Circular Economy" which goes a step forward the previous waste legislation by including specific measures to address waste management and to promote the reuse of materials (Com (2015), 614). In the context of new waste policies and an unstable biogas market, which can be drastically affected by new legislation such as the Spanish law RD 900/2015, it can be foreseen that adaptation of waste management strategies in the framework of circular economy is likely to be undertaken by employing innovative solutions. A feasible strategy towards the implementation of circular economy could be transforming widely implemented AD for biogas production into volatile fatty acid (VFA) fermentation.

VFA are added value molecules that can be used for many applications such as (i) a carbon source for biopolymer production (i.e. PHA) or biological nutrient removal in WWTP, (ii) precursors for biodiesel and other fuels, and (iii) chemical building blocks for the production of pharmaceuticals, and solvents. Regarding market size for the main VFA (i.e. 2–5 carbon atoms), the share for acetic acid is the biggest one, followed by propionic acid, and then butyric acid, presenting values up to 3,500,000 t/year, 180,000 t/year and 30,000 t/year, respectively (Zacharof and Lovitt, 2013). In contrast, the market price is the highest for butyric acid, followed by propionic acid; acetic acid is the product with the lowest market price compared to other VFA. Interestingly, the world market for bio-products is estimated to increase, for 2020, more than 5 times the market size in 2010, reaching 515 billion € (Jankowska et al., 2017). Therefore, despite the differences in market size, the exploitation of all VFA could be profitable if efficient production solutions are implemented. In this respect, it is remarkable that although the main synthesis route for VFA worldwide is currently

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based on the chemical transformation of petrochemicals, their biological synthesis has recently gained interest due to the above-mentioned policy framework and to the urgent need to diminish dependency on non-renewable fossil fuels. VFA can be synthesized from organic waste streams in mixed culture engineered fermentation processes, and thus they may constitute one of the main products of novel biorefineries, which will most likely be oriented towards the production of biofuels and valuable chemicals by transforming biomass-based feedstock (Satinder et al., 2017).

According to Zhou et al. (2017), metabolic routes in anaerobic fermentation conditions may greatly influence the final VFA distribution in the fermentation broth, i.e. acetate/ethanol fermentation, propionate fermentation, butyrate fermentation, mixed acid type fermentation, lactate type fermentation and homoacetogenic fermentation. Moreover, the anaerobic metabolism in mixed culture media is inherently linked to the experimental conditions, namely pH, temperature, organic loading rate (OLR), inoculum source, retention time, and operating mode. Sewage sludge anaerobic fermentation for VFA production has been addressed in the literature by several authors in the last decade (Lee et al., 2014; Maspolim et al., 2015; Ma et al., 2016; Wu et al., 2017). The influence of pH, temperature and retention time in the acidogenic fermentation of sewage sludge has been a major subject of study. On the one hand, it has been reported that the pH values for VFA production lies within the range 5.25–11, although it may be changed depending on the kind of substrate to be fermented (Jankowska et al., 2017). On the other hand, according to recent studies, alkaline fermentation can be considered a promising technology for VFA recovery (Liu et al., 2011, Jankowska et al., 2015, Fang et al., 2017). Regarding the effect of temperature, Hao and Wang (2015) stated that thermophilic conditions lead to higher VFA accumulation. VFA production yield and degree of acidification degree have been addressed in the afore-mentioned studies, while individual VFA production has been analysed to a lesser extent. The influence of pH, substrate complexity and retention time on VFA production potential and individual VFA of various organic streams has been recently discussed (Fang et al., 2017; Jankowska et al., 2017).

To the author's knowledge, little literature is available dealing with up-scaling and pilot-scale VFA production by means of anaerobic fermentation. Longo et al. (2015) studied the alkaline fermentation of sewage sludge in a reactor of 500 L followed by a filtration step. Additionally, den Boer et al. (2016) obtained promising results for VFA production optimization, employing a co-culture of microorganisms with kitchen biowaste and potato peels in order to produce short and medium chain fatty acids, under microaerobic conditions. Nonetheless, further studies are required for VFA production optimization prior to full-scale implementation.

The main objective of the present study was to evaluate the anaerobic fermentation of sewage sludge and agri-food waste for the production of a VFA-rich by-product. Firstly, the study aimed to assess and compare the individual VFA production potential from urban sewage sludge (SS) and two primary organic streams from the Spanish agri-food industry, namely winery wastewater (W_{ww}) and meat and bone meal (MBM). For that purpose, individual VFA production profile was assessed in batch stage laboratory assays under acid pH and alkaline pH (i.e., 5.5 and 10) and different temperature conditions (i.e., 35 °C and 55 °C). Secondly, the present research aimed to evaluate the effect of up-scaling on the fermentation products of the afore-mentioned organic waste streams, comprising both mono-fermentation assays and two co-fermentation scenarios, namely SS + W_{ww} and SS + MBM.

2. Materials and methods

2.1. Organic waste streams

In this study the waste streams analysed consisted of (i) SS collected from the thickener of a municipal WWTP in San Sebastian (Spain) which performs a high-load biological treatment, and two biowaste streams, namely (ii) W_{ww} collected from an industrial plant which obtains value added products (i.e. alcohol, tartrate, grape seed oil, and enocianin) from the raw waste generated in wine production (mainly marks, lees, scrape and wine) and (iii) MBM from a meat waste valorisation plant which processes meat waste from the slaughterhouse industry. The latter were collected from industrial plants located in the Northern Spain. Both W_{ww} and MBM were regarded as interesting waste streams for VFA recovery, due to the importance of the wine and meat sector in the Spanish agri-food industry (Cesce, 2016). In addition, to the author's knowledge there is a lack of literature available regarding their anaerobic fermentation. The main characteristics of the substrates used in the present study are shown in Table 1.

Digested sludge from the anaerobic digester of the above mentioned municipal WWTP was used as inoculum and as a source of active acidogenic biomass. All waste samples and inoculum were stored at 5 °C prior to use (see Table 2).

2.2. Analytical methods

Acetic acid, propionic acid, isobutyric acid, butyric acid, isovaleric acid and valeric acid were determined on the filtered fraction with an Agilent GC–6890 gas chromatograph equipped with a FID (Flame Ionization Detector) and a capillary column (DB-FFAP, 30 m × 0.25 mm i.d., 0.25 μm film, Agilent J&W: ref. 122-3232E). The filtered fraction was obtained by means of centrifugation (4000 rpm, 5–10 min) followed by vacuum filtration (Whatman 1.5 μm) and it was processed by liquid–liquid extraction with an organic solvent (Ter-Butil-Metyl-Eter) in order to obtain the sample for VFA determination. Pivalic acid was used as an internal standard solution.

The VFA results were expressed as g COD g^{-1} acid, with the following conversion rates: 1.07 g COD g^{-1} acetate, 1.51 g COD g^{-1}

Table 1
Characteristics of the organic waste streams and the inoculum used in the present study.

	SS	W_{ww}	MBM	Inoculum
pH	6.2 ± 0.1	5.3 ± 0.2	NA	7.45 ± 0.2
TS (%)	5.3 ± 0.9	9.1 ± 0.46	99.2	3.2 ± 0.3
VS (%)	4.1 ± 0.6	6.5 ± 0.61	68.1	2.0 ± 0.2
tCOD (mg O_2 L^{-1})	56,457 ± 10,697	97,170 ± 5864	1016[a]	27,205 ± 3254
sCOD (mg O_2 L^{-1})	2776 ± 697	38,860 ± 7457	n.a.	1882 ± 684
TAN (mg N L^{-1})	152 ± 33	224 ± 97	n.a.	1922 ± 296
TKN (mg N L^{-1})	4262 ± 1314	3,108 ± 436	86[b]	n.a.

[a] mg O_2 g^{-1} TS.
[b] mg N g^{-1} TS.