Waste Management 72 (2018) 178–185

Contents lists available at ScienceDirect

Waste Management

journal homepage: www.elsevier.com/locate/wasman

Comparison of the acidogenic and methanogenic potential of agroindustrial residues

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ARTICLE INFO

Article history:
Received 18 May 2017
Revised 18 November 2017
Accepted 20 November 2017
Available online 24 November 2017

Keywords:
Agroindustrial residues
Acidogenic fermentation
Mixed culture
Anaerobic digestion
Volatile fatty acids
Carboxylates

ABSTRACT

The methanogenic and acidogenic potentials of six different agroindustrial residues, i.e. of fruit pulps and brewery residues, were determined. For all substrates, the methanogenic conversion yield was systematically higher than the acidogenic one in Chemical Oxygen Demand (COD) terms, ranging from 0.46 to 0.87 $g_{COD\_CH4}/g_{COD\_substrate\_fed}$ and from 0.24 to 0.56 $g_{COD\_tVFA}/g_{COD\_substrate\_fed}$, respectively. During methanogenic conversion, brewery trub exhibited the highest methane potential (304 $ml_{CH4}/g_{COD\_substrate}$). Trub also exhibited the highest total volatile fatty acids (tVFA) concentrations in the mixed liquor (ML) during acidogenic conversion (29.7 g_{COD_tVFA}/kg_{ML}). Acetic, butyric and caproic acids were the main carboxylates produced by the different substrates. Despite the lower conversion yields, the economic value of the acidogenic product (carboxylate streams) is higher than that of methanogenic conversion (methane) due to the higher value of carboxylates and their potential use in finer applications (e.g. bio-based products) compared to energy production form methane.

1. Introduction

Agroindustrial residues like fruit, vegetable or brewery waste have attracted increasing attention during the last decade owing to the potential of valorising their organic fraction for various energetic or material applications, thus bringing added value to the entire process from which they are generated (Oreopoulou and Tzia, 2007). According to Eurostat, the amount of the waste categories (i) animal & mixed food waste and (ii) vegetal waste (classification W091 and W092, respectively) generated by the economic activity "Manufacture of food products, beverages and tobacco products (activity C10–C12 according to NACE-Rev2 classification) amounted to 22.1 Mt in 2014 in the EU-28 (Eurostat-Waste Statistics, 2017). The valorisation potential of these residues is, therefore, attractive as suggested by several authors. Indicatively, for the type of waste that are of interest in this study, Ravindran and Jaiswal (2016) and Federici et al. (2009) both highlighted the potential of food processing waste and commented on the technological challenges that need to be overcome to increase product yield and decrease operating costs for their conversion. Mussatto (2009) and Mathias et al. (2014) also reviewed the biotechnological potential of the brewing industry by-products and their possible applications.

Among other options for the treatment of agroindustrial waste, anaerobic digestion has been widely implemented at industrial scale, typically, for the production of methane (biogas), a substitute of natural gas (Angelidaki et al., 2011). Anaerobic digestion is a sequence of four naturally occurring and interdependent processes i.e. hydrolysis, acidogenesis, acetogenesis and methanogenesis. Methane has been the main focus in literature, as the final product of anaerobic digestion. Nevertheless, short chain carboxylic acids deriving from the intermediate acidogenic phase like acetic, propionic, butyric and caproic acids, otherwise called volatile fatty acids (VFA) or carboxylates, are also of high industrial interest. Acidogenesis (or acidogenic fermentation) has been mostly studied in view of increasing biogas (e.g. two-stage anaerobic digestion systems) or biohydrogen (i.e. dark fermentation) yields, but it can be envisaged as a stand-alone process with a product of higher added value (Singhania et al., 2013; Alkaya and Demirer, 2011; Zacharof and Lovitt, 2013). VFA have a variety of potential end-uses, for example as monomers for polyhydroxyalkanoates (PHA) production, for bio-based solvent production, for biological nutrient removal, etc. Interest in VFA has increased during the last years in the context of the carboxylate platform, a term used in the biorefinery context highlighting the multitude of production processes and potential applications of VFA (Jang et al., 2012; Agler et al., 2011).

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https://doi.org/10.1016/j.wasman.2017.11.033
0956-053X/

Each phase in the sequence of anaerobic digestion is realised by a different group of microorganisms (i.e. acidogenic bacteria, acetogenic bacteria, methanogenic archaea) working in synergy but often also competition (Angelidaki et al., 2011). While methane production occurs naturally under anaerobic conditions, separating acidogenesis requires a more careful control of the process conditions. Acetogenesis and methanogenesis must be avoided to increase the net conversion yield of the substrate into VFA. This control is especially challenging for mixed culture fermentations, i.e. fermentation by natural microbial consortia, not pure cultures (Arslan et al., 2016; Rodríguez et al., 2006). Some advantages of using mixed cultures are: the ability to treat various types of complex substrates owing to the microbial diversity, no need for sterile conditions, lower costs of production and potential for a continuous process (Kleerebezem and van Loosdrecht, 2007). On the other hand, the lack of product specificity and the complexity of the medium are still a bottleneck in terms of product yields and recovery and prevent mixed culture fermentation from wide implementation at industrial scale.

While methane production is already established as an industrial process, acidogenic fermentation is still under research. Research is focusing mainly on (i) the influence of process parameters of batch or continuous operation (pH, substrate concentration, HRT, OLR) on the concentration and composition of VFA (Arslan et al., 2016; Lee et al., 2014; Gameiro et al., 2016; Jiang et al., 2013) and (ii) downstream processing for the concentration, recovery, separation and purification of VFA streams (Zacharof and Lovitt, 2014; López-Garzón and Straathof, 2014; Arslan et al., 2017).

Keeping in mind the difference in maturity between the two processes, the purpose of this study is to highlight the interest of mixed acidogenic fermentation as an alternative option for organic solid waste treatment. For that we determined and compared both the acidogenic and methanogenic potential of a number of organic residues of agroindustrial interest. The present paper presents the results and discusses the observed differences. The decision over the one or the other process will ultimately depend on the product yields, the economic potential deriving thereof and the investment required to harness it. To the best of our knowledge, this is the first study where these two processes are systematically examined as two separate processes for the same waste fraction.

2. Materials and methods

2.1. Substrates

Two types of agroindustrial residues were investigated: (i) fruit pulps after juice extraction and water washing, more specifically date, pear and apple pulp from a syrup producing industry (Aubel, Walloon Region, Belgium), (ii) brewery residues, more specifically hot trub, spent yeast, and spent grain from a small-scale brewery (Ath, Walloon Region, Belgium). The properties of the substrates are resumed in Table 1.

2.2. Concentrated activated sludge

Activated sludge from the municipal wastewater treatment plant in Mont-St-Guibert, Belgium was used as primary inoculum for both methanogenic and acidogenic conversions. The sludge was left to settle in the dark at 4 °C for two days and the supernatant was removed. Average properties of the concentrated sludge were: Total Solids (TS) in the fresh matter (FM) at 0.024 ± 0.005 g_{TS}/g_{FM} (8 samples from 4 experiments), Volatile Solids (VS) at 0.40 ± 0.08 g_{VS}/g_{TS} (10 samples from 4 experiments) and COD at 20.7 ± 1.2 g_{COD}/kg_{FM} (13 samples from 4 experiments. The treatment of the inoculum prior to each conversion is presented in 2.3.1 and 2.4.1, respectively.

2.3. Methanogenic conversion

2.3.1. Inoculum preparation

For the preparation of the methanogenic inoculum, the primary inoculum was maintained at 35 °C under anaerobic conditions for at least 4 weeks and fed every week with fresh activated sludge, concentrated as described in Section 2.2, at a ratio of 0.1 $g_{COD\_concentrated\_sludge}/g_{COD\_primary\_inoculum}$. The container of the primary inoculum was closed with a rubber cap. One extremity of a PVC tube was inserted through the rubber cap into the headspace of the container, while the other extremity was immersed into Erlenmeyer flask filled with water. This allowed the release of biogas while preventing air contact with the container (Angelidaki et al., 2011). Ten days before the start of the experiment, the inoculum received a last feed at a ratio of 0.3 $g_{COD\_concentrated\_sludge}/g_{COD\_primary\_inoculum}$ (Donoso-Bravo et al., 2015).

2.3.2. Bioreactor preparation

The anaerobic digestion took place in 1 l Schott Duran GL45 bottles with a lateral glass tube of 4 cm long placed at an angle of 45° upwards. A 2-way Luer polycarbonate valve (Fisher Scientific) was connected via a short PVC tube to the extremity of the glass tube. Gas sampling and pressure measurements were achieved through this valve. The bioreactor-bottle was closed with a PBT screw-cap, containing a PTFE coated silicone seal.

At the start of the experiment, the methanogenic inoculum was introduced in the reactors and the substrate was subsequently added. Experiments were conducted in triplicates. The quantities of each substrate added in the reactors are shown in Table 2. Three reactors contained the inoculum and water instead of substrate and served as a control. After adding the inoculum and the substrate, the headspace of the reactor was flushed with N_2 for one minute before closing the reactor, in order to remove oxygen and establish anaerobic conditions. The volume of each reactors' headspace was determined by comparing the weight of each reactor (i) empty, (ii) full of water, (iii) filled with the final volume of mixed liquor.

2.3.3. Monitoring of methanogenic conversion

Depending on the substrate, the anaerobic digestion lasted between 20 and 50 days. At regular intervals, the pressure of the reactor's headspace was measured with a manometer (UNIK 5000 PTX5072-TA-A3-CA-H0-PA, GE Measurement & Control Solutions) in order to determine the quantity of biogas produced, through the ideal gas law. The manometer was connected to the reactor with a 3-way valve, permitting the connection of a 50 ml polypropylene syringe to sample the gas phase. After sampling, the reactor was depressurized to atmospheric pressure that was also recorded. The gas phase sample was immediately analysed by GC-TCD (see Section 2.4) to determine its composition in CH_4, CO_2, H_2, O_2 and N_2.

The total volume of biogas produced and its composition permitted to calculate the amount of CH_4 produced. The amount of methane produced in the control reactors was subtracted from the amount of methane produced in the reactors with the substrate, in order to provide the net amount of methane produced by the substrate. The conversion yield of the substrate to methane (methanogenic potential) is determined by Eq. (1).

$$R_{CH4} = \frac{V_{CH4} * F}{Q_{substrate}}, \quad (1)$$