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Anaerobic digestion of grape pomace: Effect of the hydraulic retention time on process performance and fibers degradability

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To optimize the anaerobic digestion of grape pomace under mesophilic conditions, continuous digesters were operated at different hydraulic retention times (HRT) (30, 20, 15 and 10 days) equivalent to organic loading rates (OLR) of 2.5, 3.7, 5.7 and 7.3 kg COD m^{-3} d^{-1}, respectively. At HRTs of 30 and 20 days, steady state conditions were observed with methane yields of 0.984 ± 0.013 NL d^{-1} and 1.362 ± 0.018 NL d^{-1}, respectively. The HRT of 15 days was found critical because of acids accumulation through the experiments. When the OLR of 5.7 kg COD m^{-3} d^{-1} was reached, methane production was found to be instable. Finally, at HRT of 10 days, a failure of the system was observed due to the washing of the methanogenic microorganisms. Regarding the degradability of the lignocellulosic fractions, the maximum reduction yields for hemicellulose and cellulose were noted for HRTs of 30 and 20 days, while lignin was not degraded throughout the different experiments. For an optimization of the process, HRT of 20 days can therefore be recommended for productive use in large-scale applications.

1. Introduction

Anaerobic digestion (AD) experiments in continuous mode are long and complex, so that the great majority of studies are based on the results of batch tests (Barbanti et al., 2014; Kafle and Chen, 2016; Pellera et al., 2016). The latter, although valuable for determining the maximal methane production of a substrate under specific conditions, may not accurately predict the performance of large-scale anaerobic digesters, due to their dependence on the inoculum type, the substrate to inoculum ratio and the experimental method used (Holliger et al., 2016). Moreover, it is complicated to screen the physicochemical parameters affecting process stability and determine the lignocellulosic fractions biodegradability. Consequently, continuous digesters are required to evaluate and quantify the effect of a specific substrate and selected operating conditions on AD to adequately optimize of the process (Carrère et al., 2010; Pokój et al., 2015).

A series of operating parameters including pH, temperature, digester configuration, Organic Loading Rate (OLR) and Hydraulic Retention Time (HRT) have been investigated due to their effect on digesters performance and methane production (Dareioti and Kornaros, 2014; Mariakakis et al., 2011; Nasir et al., 2012; Rincón et al., 2008; Torkian et al., 2003). Among them, HRT has been reported as one of the most important parameters significantly affecting microbial ecology in continuous digesters and thus must be optimized.

HRT is the average time an input organic matter stays inside the digester before it comes out. Based on different literature studies, it has been shown that HRT of 30 days is usually used to evaluate the AD of many agro-industrial wastes in continuous mode (Kafle and Kim, 2013; Ruffino et al., 2015). The longer a substrate is kept in the digester, the more complete its degradation will be. The disadvantage of a longer HRT is that a large digester size is needed for a given amount of substrate. Although a shorter retention time is essential for reducing the digester volume, a compromise must be considered to maximize methane production and to prevent system inhibition or failure. This inhibition may be due to the accumulation of acids or the washout of the microorganisms.

OLR is the amount of digestible material that is fed to the digester, in kg COD per m^3 digester and day. A high load enables high microorganism growth and high biogas production, but it also puts pressure on the microorganisms and can lead to process collapse due to acid accumulation, during the acidogenesis stage. To maximize the production of biogas while maintaining stability in the

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digester, a study suggested that the organic load must be a wide operating range which can vary between 0.25 and 3 kg COD m^{-3} d^{-1} (Rajeshwari et al., 2000). However, recent literature study has shown that it is possible to operate CSTR digester with higher applied OLR such as 7.15 kg COD m^{-3} d^{-1} (Dareioti and Kornaros, 2015).

To our best knowledge, research papers evaluating the AD of grape pomace (GP) are mostly based on the results of batch tests (Caramiello et al., 2013; Carchesio et al., 2014; Dinuccio et al., 2010; Fabbri et al., 2015). In a recent study, methane production from winery residues were determined by batch trials under thermophilic conditions. GP and wine lees appeared to be the most promising substrates with an estimated potential of 0.34 and 0.37 Nm^3CH_4 $kgVS_{fed}$, respectively. To evaluate the feasibility of a continuous AD process, a lab-scale semi-continuous reactor was constructed. A specific biogas production of 0.29 Nm^3 $kgVS_{fed}$ from GP was observed when working under HRT of 40 days (Da Ros et al., 2016). In a previous study, we have determined the methane potential of GP in a Continuous Stirred Tank Reactor (CSTR) with an OLR of 2.5 kg $COD{\cdot}m^3{\cdot}d^{-1}$ and a HRT of 30 days (El Achkar et al., 2016). In addition, we have studied the effects of several chemical and physical pretreatments on the methane production of this by-product (El Achkar et al., 2018). Here, we investigate the optimization of the long-term operation of mesophilic and continuous anaerobic digesters treating GP. Our main purpose is to check whether a residence time of less than 30 days is adequate or not for a sufficient digestion of GP, optimizing the process. Thus, continuous digesters were operated at different HRTs (30, 20, 15 and 10 days) equivalent to final OLRs of 2.5, 3.7 and 5.7 and 7.3 kg COD m^{-3} d^{-1}, respectively. In addition, at the end of the experiments, the effects of HRT on the biodegradability of the main lignocellulosic components (hemicellulose, cellulose and lignin) were assessed.

2. Materials and methods

2.1. Materials

The GP variety "Chenin blanc", was obtained from a wine company (Domaine Des Acacias, Layon, France, 47° 12' 25.949" N 0° 25' 51.392" W). The grapes were carefully harvested at maturity during the 2015 vintage and fresh samples of GP were collected immediately after the pressing operation and transported to the laboratory. On arrival, the stalks fragments were removed, and the raw material was stored at −20 °C until utilization. An active inoculum was collected from the digester of the waste-water treatment plant of Saint-Brieuc city in France. Particulate matter (>500 mm) was removed from the inoculum by passing through sieve.

2.2. Analysis

Total Solids (TS) content were determined by dry weight in oven at 105 °C until constant weight. Afterwards, Volatile Solids (VS) were determined in a muffle furnace after 4 h at 550 °C. Total Chemical Oxygen Demand (COD) was measured using Merck COD Spectroquant® test, range 500–10,000 $mg{\cdot}L^{-1}$, and by a spectrophotometer NOVA 60 (Merck, Germany). After 2 h at 150 °C, it was considered that all organic matter is oxidized by the hot sulfuric solution of potassium dichromate.

2.3. Van-Soest fractionation

The structural polysaccharides and lignin fractions were determined using the method of Van-Soest (Mertens, 2002; Van Soest et al., 1991). The total soluble compounds were extracted with a neutral detergent solution, acting on the sample for 1 h at 100 °C. This solution consists of Na_2HPO_4, sodium tetraborate, α-amylase, sodium EDTA, sodium lauryl sulfate and sodium sulfite. The extracted fraction was separated from the insoluble neutral detergent fibers by filtration. Then, hemicellulose was extracted by an acid detergent solution (cetyltrimethylammonium bromide 20 kg m^{-3}, 98 kg m^{-3} of H_2SO_4) acting for 1 h at 100 °C. This fraction was separated from the insoluble acid detergent fiber by filtration. Cellulose was extracted after a treatment with 1317 kg m^{-3} of H_2SO_4 for 3 h at ambient temperature (20 ± 1 °C). The filtration residue corresponds to the lignin associated with the inorganic material. Lignin fraction is thus determined in a muffle furnace after 4 h at 550 °C.

The reduction yields of hemicellulose, cellulose and lignin in the digesters were calculated according to the following equation (Bayr and Rintala, 2012):

$$\text{Reduction yield}(\%) = \frac{\text{Amount in the feed} - \text{Amount in the digestate}}{\text{Amount in the feed}} \times 100$$

2.4. Methane production in continuous mode

The system for measuring methane production in continuous mode consists of three main units (Fig. 1). The incubation unit is a thermostatic water bath (37 °C) which contains eight digesters with a working volume of 2 L (Bioprocess Control, Lund, Sweden). Each glass digester is equipped with two sealable tubes; the first one for drawing off the digestate and the second for supplying the GP. Digesters were stirred manually for one minute, twice a day, to ensure adequate mixing. The biogas produced passes through a tube connecting the incubation unit to the CO_2 fixing unit (6 M NaOH solution), which allows CH_4 to pass through while

Fig. 1. Continuous digesters system: incubation unit (a), CO_2 fixation unit (b) and CH_4 volume measurement unit (c).

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