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Effect of moisture on pretreatment efficiency for anaerobic digestion of lignocellulosic substrates

M. Peces [a,b], S. Astals [a,c,*], J. Mata-Alvarez [a]

[a] *Department of Chemical Engineering, University of Barcelona, C/Martí i Franquès, No. 1, 6th Floor, 08028 Barcelona, Spain*
[b] *Centre for Solid Waste Bioprocessing, Schools of Civil and Chemical Engineering, The University of Queensland, St. Lucia Campus, 4072 QLD, Australia*
[c] *Advanced Water Management Centre, The University of Queensland, St. Lucia Campus, 4072 QLD, Australia*

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ABSTRACT

The present study evaluates the effect of moisture in low-temperature and ultrasound pretreatment on lignocellulosic substrates anaerobic biodegradability, where brewer's spent grain was used as model substrate. Besides moisture content, low-temperature pretreatment was also evaluated in terms of temperature (60–80 °C) and exposure time (12–72 h). Likewise, ultrasonication was also evaluated in terms of specific energy (1000–50,000 kJ $kgTS^{-1}$). In addition, the effect of substrate particle size reduction by milling pretreatment was also considered. The results clearly demonstrated that substrate moisture (total solid concentration) is a significant parameter for pretreatment performance, although it has been rarely considered in pretreatment optimisation. Specifically, moisture optimisation increased the methane yield of brewer's spent grain by 6% for low-temperature pretreatment (60 °C), and by 14% for ultrasound pretreatment (1000 kJ $kgTS^{-1}$) towards the control (without pretreatment). In both pretreatments, the experimental optimum total solid concentration was 100 gTS kg^{-1}. Thus, lowering substrate moisture, a strategy suggested attaining energetic pretreatment feasibility, needs to be analysed as another pretreatment variable since it might have limited correlation. Finally, a preliminary energetic balance of the pretreatments under study showed that the extra methane production could not cover the energetic pretreatment expenses.

1. Introduction

Anaerobic digestion (AD) is a biological process that converts organic matter into biogas through the action of different groups of microbes in the absence of oxygen (Batstone et al., 2002). AD is considered a feasible and mature technology. However, the AD of single substrates presents some drawbacks linked to substrate properties that can lead to poor biogas yields (Mata-Alvarez et al., 2014). Several of these problems can be solved by the addition of a co-substrate, a process known as anaerobic co-digestion (Mata-Alvarez et al., 2014). Nonetheless, in some regions, easily biodegradable co-substrates are already being used, or they are just not available (Astals et al., 2015; Mata-Alvarez et al., 2014). The scarcity of these co-substrates have drawn the attention to more complex agro-industrial wastes and to grow onsite energy crops (including algae cultivation), which are characterised by a high lignocellulose content. However, lignocellulosic biomass (LB) is hardly biodegradable by anaerobic microorganisms because the biodegradable organic matter is trapped in the lignocellulosic structure (Mosier et al., 2005). These facts explain the large interest on LB pretreatments over the past years, where several pretreatments (biological, thermal, mechanical, chemical or a combination of them) have been considered to break down the lignocellulosic walls with the aim of improving LB biodegradability (Hendriks and Zeeman, 2009; Mosier et al., 2005). Nonetheless, pretreatments are not exempt from disadvantages. In general, highly invasive pretreatments can lead to the generation of recalcitrant compounds (e.g. Maillard compounds), and inhibitors (e.g. furfurals and phenolic compound) for the AD process (Hendriks and Zeeman, 2009). Likewise, high expenses in the form of chemicals, energy and investment costs must be expected.

In order to improve the pretreatment efficiency, the length of pretreatment time, the energy supplied and the particle size are among the most studied parameters. To improve pretreatments energetic feasibility, some authors suggested increasing the total solid (TS) concentration of the pretreatment influent (reciprocal to lower the moisture content), therefore waste volumes are decreased, and consequently, pretreatment costs (Adl et al., 2012; Passos et al., 2014a; Perez-Elvira et al., 2009). However, these calculations assume that the effectiveness of the

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* Corresponding author at: Advanced Water Management Centre, Gehrmann Building (60), Level 4, The University of Queensland, St. Lucia, 4072 QLD, Australia.
E-mail address: s.astals@awmc.uq.edu.au (S. Astals).

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pretreatment is not affected by the TS concentration. To date, few investigations can be found evaluating the impact of TS concentration, or by subtraction moisture content, over pretreatment since TS concentration has been considered an intrinsic characteristic of the substrate. However, the limited studies where TS has been contemplated as a variable, suggest that TS concentration can influence the pretreatment efficiency. Show et al. (2007) found that diluting sewage sludge to 20–30 gTS L^{-1} increased sludge solubilisation in ultrasound pretreatment. However, higher solubilisation rates may not be directly proportional to higher methane yields (Kim et al., 2013; Ruiz-Hernando et al., 2014). Rabelo et al. (2011) found that the highest methane production from sugarcane bagasse was obtained at 40 gTS kg^{-1} for both alkaline hydrogen peroxide and lime pretreatment. These results indicate that pretreatment effectiveness might not be assumed independent of TS concentration. Nevertheless, to our knowledge, no systematic study has been performed quantifying the influence of TS concentration over pretreatment efficiency.

The main goal of this study was to evaluate the effects of TS concentration in ultrasound (physical) and low-temperature (thermal-biological) pretreatment efficiency for improved LB anaerobic biodegradability. Brewer's spent grain (BSG) was chosen as model lignocellulosic substrate since its homogeneity could allow correlating the results differences to the pretreatment conditions rather to intrinsic variability due to feedstock heterogeneity. Additionally, other variables like temperature, specific energy, and exposure time were assessed for the pretreatments above. The effect of particle size through milling pretreatment was also performed and used as a reference.

2. Materials and methods

2.1. Substrate and inoculum origin

Brewer's spent grain (BSG) was obtained from a microbrewery located in L'Hospitalet de Llobregat (Barcelona, Spain). BSG is a by-product of the brewery industry, consisting of crushed husks of malted barley grains obtained after the extraction of fermentable starch and polypeptides (Mussatto, 2014). BSG was collected right after mashing step in the brewing process (i.e. after BSG saccharification), and stored at 4 °C until its utilisation. Table 1 summarises the main characteristics of the raw BSG. Average literature values for BGS chemical composition range between 130 and 260 g kg^{-1}TS of cellulose, 192–296 g kg^{-1}TS of hemicellulose, 119–278 g kg^{-1}TS of lignin, and 153–247 g kg^{-1}TS of protein (Mussatto, 2014; Robertson et al., 2010).

The inoculum used in the biochemical methane potential (BMP) tests was collected from a centralised anaerobic digestion plant that treats pig manure at mesophilic conditions (Lleida, Spain). After collection, the inoculum was stored at 4 °C. Prior to commencement of the BMP assays, the inoculum was degassed at 37 °C for one week.

Table 1
Raw BSG characterisation.

	Units	Average
TS	g kg^{-1}	223 ± 4
VS	g kg^{-1}	214 ± 4
TSS	g kg^{-1}	216 ± 3
VSS	g kg^{-1}	206 ± 2
COD	g kg^{-1}	317 ± 10
Particle size distribution (dry weight)		
5 mm < x < 2 mm	%	61
2 mm < x < 1 mm	%	34
x < 1 mm	%	5

2.2. Analytical methods

Total solids (TS), volatile solids (VS), total suspended solids (TSS), and volatile suspended solids (VSS) were determined following the standard methods 2540G procedure with minor modifications (APHA, 2012; Peces et al., 2014). Interchangeably, moisture can be calculated as the complement of TS following Eq. (1).

$$\text{Moisture }(\text{g kg}^{-1}) = 1000\ (\text{g kg}^{-1}) - \text{TS}\ (\text{g kg}^{-1}) \tag{1}$$

Total chemical oxygen demand (COD) was determined following the standard method 5220D (APHA, 2012). Biogas composition (CH_4, CO_2) was analysed by a Shimadzu GC-2010+ gas chromatograph equipped with a capillary column (Carboxen® – 1010 PLOT) and a thermal conductivity detector as described in Romero-Güiza et al. (2014).

2.3. Low-temperature pretreatment

30 g of sample (raw or diluted BSG) were added to 250 mL glass bottles. Bottles' headspace was flushed with N_2 to assure anaerobic conditions and closed with a screw-cap and rubber septum. Finally, the bottles were placed in a temperature-controlled incubator. Two sets of experiments were carried out to assess the low-temperature (LT) pretreatment on BSG. First, the LT pretreatment was carried out at 60 and 80 °C for two exposure times (24 and 48 h) to determine if the effect of the pretreatment was due to biological or thermal processes (Bonmati et al., 2001). Second, to assess the effect of TS concentration on the LT pretreatment the BSG solid content was adjusted through deionised water additions to 150 and 100 gTS kg^{-1}. Diluted and raw (220 gTS kg^{-1}) BSG samples were pretreated at 60 °C for 12, 24, 48 and 72 h. Table S-I (supplementary data) summarises the 16 pretreatment conditions studied.

2.4. Ultrasound pretreatment

15 g of different BSG samples were sonicated in a HD2070 Sonopuls Ultrasonic Homogenizer equipped with a MS 73 titanium microtip probe and working with an operating frequency of 20 kHz and a supplied power of 70 W. The ultrasonic probe was submerged until half-height of the sample and continuously stirred at 70 rpm in an orbital shaker. The temperature was not controlled during the ultrasound (US) pretreatment, where the maximum temperature reached after sonication was 35 °C. The specific energy (kJ kg^{-1}TS) supplied was adjusted by changing the pretreatment time as for Eq. (2).

$$E_s = \frac{P \cdot t}{w \cdot \text{TS}} \tag{2}$$

where E_s is the specific energy (kJ kg^{-1}TS), P is the supplied power (kW), t is the pretreatment time (s), w is the sample weight (kg), and TS is the sample total solid concentration (kgTS kg^{-1}).

Two sets of experiments were carried out to assess the impact of US on BSG. The first experiment was carried out at a fix specific energy (20,000 kJ kg^{-1}TS) and different BSG TS concentration. The BSG diluted samples were prepared by adding deionised water to the raw BSG to obtain the desired final TS concentrations of: 150, 120, 100, 80, 60 and 40 gTS kg^{-1}. Afterwards, under optimal TS concentration (100 gTS kg^{-1}), the US was evaluated at different specific energies: 1000, 2000, 5000, 10,000, 20,000, 35,000 and 50,000 kJ $kgTS^{-1}$. Table S-I (supplementary data) summarises the 14 US pretreatment conditions studied.

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