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Alkalinity and high total solids affecting H_2 production from organic solid waste by anaerobic consortia

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ABSTRACT

The optimization of total solids in the feed (%TS) and alkalinity ratio (γ) for H_2 production from organic solid wastes under thermophilic regime was carried out using response surface methodology based on a central composite design. The total solids levels were 20.9, 23.0, 28.0, 33.0 and 35.1% whereas the levels of alkalinity ratio (defined as g phosphate alkalinity/g dry substrate) were 0.15, 0.20, 0.30, 0.41 and 0.45. High levels of TS and γ affected in a negative way the H_2 productivity and yield; both response variables significantly increased upon decreasing the TS content and alkalinity ratio. The highest H_2 productivity and yield were 463.7 N mL/kg-d and 54.8 N mL/g VS_{rem}, respectively, predicted at 20.9% TS and alkalinity ratio 0.25 (0.11 g $CaCO_3$/g dry substrate). The alkalinity requirements for hydrogenogenic processes were lower than those reported for methanogenic processes (0.11 vs. 0.30 g $CaCO_3$/g COD). Adequate alkalinity ratio was necessary to maintain optimal biological activity for hydrogen production; however, excessive alkalinity negatively affected process performance probably due to an increase of osmotic pressure. Interestingly, reactor pH depended only on the alkalinity ratio, thus the buffer capacity was able to maintain a constant pH independently of TS levels. At $\gamma = 0.15$–0.30 the pHs were in the range 5.56–5.95, which corresponded to the highest hydrogen productivities and yields. Finally, the highest metabolite accumulation corresponded with the highest removal efficiencies but not with high H_2 productivities and yields. Therefore, it seems that organic matter removal was channeled toward solvent generation instead of hydrogen production at high TS and γ levels. This is the first study that shows the requirements of alkalinity in solid substrate fermentation conditions for H_2 production processes and their interaction with the content of total solids in the feed.

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1. Introduction

Hydrogen is a promising alternative to fossil fuels. As a clean fuel it can be directly used to produce electricity in fuel cells and/or burnt with minimal pollutants emissions [1–3]. Recently, the interest in fermentative biohydrogen production has increased because a wide range of organic compounds can be used as substrates, and also it can be coupled to the "biorefinery" concept for further reclaiming of fermentation by-products [4,5]. There are reports where the organic fraction of municipal solid waste has been used as substrate for hydrogen production [6–10]. Yet, the total solids (TS) content

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of the feed has been very low (<10%) which hinders and elevates the cost for eventual disposal and/or post-treatment of digestates solids. The feasibility of hydrogen production from fermentation at high TS (>20%) has been demonstrated and slightly explored [11–13] and as a result the concept of acidogenic solid substrate anaerobic digestion A-SSAD has been demonstrated and coined. However, inadequate TS levels (either low and particularly too high) during the A-SSAD would decrease the product generation due mainly to poor energy/mass transfer process [14,15] and excessive organic metabolite accumulation which could lead to process inhibition [16]. In this regard, the acidogenic conditions developed in the A-SSAD have been utilized for inhibiting H_2-consuming methanogens [17] and propitiating hydrogen accumulation. Yet, alkalinity and TS levels have to be optimized in order to provide an adequate buffer capacity that could keep a favorable and stable pH in the digester for optimal biological activity [18,12]. In this way, adequate pH could vary in a relatively narrow range dictated by several factors. On the one hand, pH should be sufficiently low in order to inhibit the methanogens as it was mentioned above. On the other hand acidogenic pH has to be higher than the pK_a of most of the organic acids generated (acetate, propionate and butyrate) since the cell is far more sensitive to the undissociated acid than the corresponding anions [19,20]. Moreover, pH should be above 5.0 to preclude the shift from hydrogenogenic to solventogenic fermentation.

In the literature, alkalinity requirements for methanogenic systems are well known [18,21], however, the studies of requirements for hydrogenogenic systems are still scarce [22]. In particular for A-SSAD, it is desirable to operate systems at high TS with the minimum buffer requirements in order to reduce costs by alkalinity addition and disposal/treatment of digestates solids. So far, to the best of our knowledge, there are no studies available in the open literature that reported the effect of alkalinity and TS content on the anaerobic hydrogen production from organic solid waste at high TS levels (>20%). Thus, the objective of this work was to optimize levels of TS and alkalinity in A-SSAD for hydrogen production from organic solids wastes in thermophilic regime by applying response surface methodology based on central composite design (CCD). The response variables were hydrogen productivity (NmL H_2/(kg d)), hydrogen yield (NmL H_2/g VS_{rem}), removal efficiency (% VS), pH, organic acids and solvents accumulation (mg COD/$kg_{wet\ mass}$). They were determined in a TS range of 20–35% and alkalinity levels between 0.15 and 0.45 g phosphate alkalinity/g dry substrate, according to previous experience in the group [12].

2. Materials and methods

2.1. Inocula and substrate

The A-SSAD were seeded with digestates from methanogenic solid substrate anaerobic digesters degrading a mixture of organic solid wastes [23]. Paper (40%) and food (60%) wastes were blended, milled, dried and stored at 4 °C until being used as substrate. The main characteristics of feedstock were: VS (% TS) 60.8%; total Kjeldahl nitrogen (% TS) 1.9%; cellulose (% TS) 26.2%, lignin (% TS) 19.4% and 1.09 g COD/g TS. Before feeding the reactors, the substrate moisture was adjusted with different buffer solutions to give the TS levels required by the experimental design. The phosphate buffer solutions (pH 7.21) that consisted of K_2HPO_4 and KH_2PO_4 were made according to the Henderson–Hasselbalch equation [24] maintaining the $[HPO_4^{=}]/[H_2PO_4^{-}]$ ratio equal to 1.0 and increasing the total salt concentration (Table 1).

2.2. Reactors and experimental procedure

The reactors consisted of 1.0 L glass jars fitted with rubber stoppers and an outlet for biogas. The mass of the reactors was 0.5 kg. The reactors were fed with the wet substrate twice a week in a draw-and-fill mode in an anaerobic glove chamber with a mass retention time of 21 days. The reactors were kept in an insulated wood chamber at 55 ± 1 °C.

2.3. Experimental design

Alkalinity and TS levels were optimized for maximum hydrogen production using one of the response surface methodologies, the central composite design CCD [25]. For a CCD with two factors, including four center points, a set of 12 experiments was carried out. Two factors were taken at five different coded levels: (−1.414), (−1.00), (0.00), (+1.00) and (+1.414).

The alkalinity ratio (γ) was defined as:

$$\gamma = \frac{A}{S_{dry}} \quad (1)$$

where A is alkalinity expressed as g of phosphate salts (44% KH_2PO_4 + 56% K_2HPO_4) and S_{dry} is the dry substrate (g). The levels studied for γ were: 0.15, 0.20, 0.30, 0.41 and 0.45 (which corresponded to 0.073, 0.097, 0.145, 0.198 and 0.218 g $CaCO_3$/g

Table 1 – Experimental design and levels of independent variables in terms of coded and natural units.

No. reactor	Range of levels				Buffer concentration[c] (g/L)
	Coded		Actual		
	x_1	x_2	Total solids[a] (%)	Alkalinity ratio[b] (γ)	
1	−1.00	−1.00	23.00	0.20	47.42
2	1.00	−1.00	33.00	0.20	75.86
3	−1.00	1.00	23.00	0.41	79.92
4	1.00	1.00	33.00	0.41	125.28
5	−1.41	0.00	20.93	0.30	57.57
6	1.41	0.00	35.07	0.30	110.83
7	0.00	−1.41	28.00	0.15	48.28
8	0.00	1.41	28.00	0.45	107.69
9	0.00	0.00	28.00	0.30	82.35
10	0.00	0.00	28.00	0.30	82.35
11	0.00	0.00	28.00	0.30	82.35
12	0.00	0.00	28.00	0.30	82.35

a Defined as the sum of TS coming from organic solid wastes (dry substrate) and phosphate alkalinity.
b Defined as g phosphate alkalinity/g dry substrate
c $[HPO_4^{=}]/[H_2PO_4^{-}] = 1.0$.

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