



Energy recovery from agro-industrial wastewaters through biohydrogen production: Kinetic evaluation and technological feasibility

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ABSTRACT

Biohydrogen production from cassava, dairy and citrus processing wastewaters (WWs) without nutritional supplementation was evaluated in anaerobic single-batch reactors at 37 °C for 70 h. Hydrogen production from cassava, dairy and citrus WW was 31.41, 37.25 and 28.95 mL g⁻¹ of chemical oxygen demand (COD). The kinetic parameters indicated that H₂ production rates for cassava processing WW (0.32 mL h⁻¹) and dairy WW (0.31 mL h⁻¹) were similar, whereas citrus processing WW exhibited the highest value (0.59 mL h⁻¹). The carbohydrate degradation rate (k_1^{APP}) was highest for dairy WW (0.045 h⁻¹), but the most efficient overall conversion of organic matter to H₂ (k_2^{APP}) was observed with cassava WW (0.014 h⁻¹). The rate of conversion of the organic matter of the cassava WW together with its ready availability resulted in a recovery of 0.59 10⁹ MJ year⁻¹, the highest in this study. Cassava WW showed the highest hydrogen production potential (97.9 mL), when compared with dairy (76.1 mL) and citrus WW (66.6 mL). The economic estimation based on the gasoline energetic equivalent indicated that a single process of H₂ production allowed the maximum economic yield of US\$ 0.009 cents L⁻¹ WW. Alternatively, a sequential hydrogen and methane process could increase energy recoveries and economic yields to values near 10.48 kJ g⁻¹ COD and US\$ 0.61 cents L⁻¹ WW (US\$ 6.10 m⁻³ WW).

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1. Introduction

Currently, worldwide energy demands are primarily supplied by fossil fuels, which are directly linked to greenhouse gas emissions. As main consequence, the search for renewable energy sources has become critical. Hydrogen gas is an interesting energetic alternative because its utilization does not involve the emission of greenhouse gases, and organic wastes can be used as feedstock in its production. Among the methods employed for H₂ production, dark fermentation using organic wastes as raw materials could be a

strategy to overcome some economic issues involved in the production process [1].

Some wastes could show some nutritional deficiencies to be used directly as raw materials in a hydrogen production process [2] and [3]. Thus, various important ratios of nitrogen and phosphorous have been reported in the literature to compensate the possible lack of such nutrients. The most common ratios for H₂ production are C:N = 47:1 [4], C:N:P = 100:0.5:0.1 [5], C/N = 100:2.2 [6] and C:N:P = 100:0.8:0.3 [7]. Hawkes et al. [8] stated that the COD:N ratios in H₂ production studies varied between 11:1 and 200:1 and found that the COD:P ratios presented an even greater variation (73:1 to 970:1). Micronutrients and trace metals are essential for cell maintenance and are required for metabolism and growth, transport processes, enzyme cofactors and dehydrogenases. These requirements were inferred from the elemental composition of anaerobic bacteria, which includes Cu, Zn, Mo, Ni and V [9]. Iron has been reported as essential for H₂ production [10] because is the element that forms ferredoxin and

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Abbreviations

COD	chemical oxygen demand (oxidizable organic matter), in milligrams per liter	Co	cobalt
TS	total solids, in milligrams per liter	Se	selenium
VSS	volatile suspended solids, in milligrams per liter	S	sulfur
FS	fixed solids, in milligrams per liter	V	vanadium
WW	wastewater	C _C	carbohydrate concentration, in milligrams per liter
C	carbon	C _{COD}	concentration of oxidable organic matter, in milligrams per liter
N	nitrogen	S	limiting substrate concentration, in milligrams per liter
P	phosphorus	X	biomass
Ca	calcium	μ	specific growth rate of the microorganisms
Mg	magnesium	r_s	rate of substrate utilization
Mo	molybdenum	C _x	biomass concentration
Na	sodium	Y _{X/S}	yield of substrate to biomass
K	potassium	$u_{\max}$	maximum specific growth rate of the microorganisms
Ni	nickel	K _s	half-velocity constant
Cu	copper	(K ₁ ^{App})	apparent kinetic constant for carbohydrate degradation
Fe	iron	(K ₂ ^{App})	apparent kinetic constant for COD reduction
Zn	zinc	SMP	soluble metabolites production

hydrogenase enzymes. Other nutrient requirements include concentrations of Ca (100–200 mg L⁻¹), Mg (75–150 mg L⁻¹), Na (100–200 mg L⁻¹), Fe (20–100 mg L⁻¹) and K (200–400 mg L⁻¹) [11] and [12]. Some studies have employed raw wastes as substrates for H₂ production [7,13,14]. In most cases, the carbon sources were supplemented with complex nutritional additives [5,15–19] to improve process hydrogen yielding, thereby decreasing its economic feasibility.

Food processing WW could be interesting raw materials sources for dark fermentation targeting hydrogen production because of their high carbohydrate and nutrients content [20]. These WW could require less or none nutrient supplementation to achieve a feasible hydrogen production process. Cassava starch processing WW could be an ideal raw material for the dark fermentation process, due its rich composition of carbohydrates, nutrients (N, P) and minerals (Fe, Zn, Mg) [5] and [21]. High content of easily degradable carbohydrates and soluble organic substances (5–50 g L⁻¹ COD) in dairy WW can support bacterial growth [22] and [23] for hydrogen production. Citrus WW, obtained from orange peels processed for pectin production, has 17% of soluble sugars [24], being a rich source of carbohydrates.

In Brazil, the current production of cassava starch WW is 184,100,000 cubic meters per year based on data provided by FAO (Food and Agriculture Organization of the United Nations) (2008) [25] and estimates that cassava processing generates 7 cubic meters per ton of product [26]. Dairy WW generation is typically 2.5 times the volume of milk processed [27]. Data from FAO (2008) [25] indicate that 31,200,000 tons of milk were processed in 2008, resulting in 78,000,000 cubic meters of WW. In addition, in 2013, 82,072.94 cubic meters of citrus processing WW was generated [24] according to IBGE (Brazilian Institute for Geography and Statistics) reports [28].

Given the availability of the aforementioned WWs and the growing need for clean energy sources, this paper discusses the use of bench-scale batch reactors to evaluate the potential of non-supplemented substrates (cassava, dairy and citrus WW) for the production of hydrogen and the recovery of energy. In addition, apparent kinetic parameters were assessed for scaling up the process, and simple, economic estimations were performed to evaluate the process economic sustainability. Since there are no works in

hydrogen production through dark fermentation of cassava, dairy and citrus WW without nutrient supplementation, the present paper fills a gap in the discussion of technical and economic feasibility of biohydrogen production.

2. Materials and methods

2.1. Experimental procedure

Experiments were conducted in 2 L batch reactors (Duran® flasks). Headspace of the bioreactors (1 L) was flushed with nitrogen gas to maintain anaerobic conditions. All experiments were performed in shaker fermenters at 37 °C with orbital shaking at 150 rpm for 70 h. Each bioreactor was inoculated with an anaerobic biomass obtained from a UASB reactor treating poultry slaughterhouse WW (26 g VSS L⁻¹). Prior to the inoculation step, the inoculum received a heat-shock pretreatment to inhibit the H₂-consuming bacteria, according to the protocol described by Wang and Wan [29].

Cassava WW was sampled from a cassava starch processing plant (Toledo, PR, Brazil). Dairy WW was obtained from the cleaning procedure of a UHT milk processing plant (Francisco Beltrão, PR, Brazil), and citrus WW was sampled at a plant extracting pectin from citrus (Limeira, SP, Brazil). Reactors were filled with 900 mL of filtered (0.20 µm membrane) wastewater diluted with tap water to its working concentration (approximately 2 g L⁻¹ COD) and with 100 mL of inoculum. pH was adjusted to 5.5 by adding sodium bicarbonate or hydrochloric acid. Control substrate (sucrose) was complemented with the nutritional medium shown in Table 1. Gas and liquid samples were periodically obtained from the reactors for analyses. Internal pressure of each flask was measured using a pressure gauge (Desin Instruments® TPR-18, Barcelona – Spain) with detection range of 0–500 mbar.

For the estimation of economic impact of WW nutritional supplementation, a control medium containing sucrose as carbon source was utilized (Table 1). This control medium was chosen based on previous successful evaluations [30–32]. Additionally, the medium consisted of a low cost substance and contained considerably lower nutrient concentrations than those found in the assayed WWs, as shown in Table 1.

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