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Screening of biological sulfate reduction conditions for sulfidogenesis promotion using a methanogenic granular sludge

M. Mora, J. Lafuente, D. Gabriel*

GENOCOV Research Group, Department of Chemical, Biological and Environmental Engineering, Escola d'Enginyeria, Universitat Autònoma de Barcelona, 08193 Bellaterra, Spain

HIGHLIGHTS

- Crude glycerol was selected as the most adequate C source for sulfate reduction (SR).
- Optimal conditions for S^{2-} promotion were 35 °C, pH = 8.5, and COD/S over 7.0 g O_2/g S.
- A maximum SR rate of 5.06 ± 0.58 mg S L^{-1} h^{-1} was obtained under optimal conditions.
- Methanogenic granular sludge became mainly sulfidogenic after the screening tests.
- Sulfidogenic activity in the UASB reactor was promoted in a startup of 15 days.

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ABSTRACT

Effluents containing great amounts of oxidized sulfur compounds, such as sulfate or sulfite, can be valorized as elemental sulfur from a sequential reduction-oxidation biological process. However, the most important, challenging step to be optimized is the reduction of sulfate. The present study aimed at seeking out the optimal conditions to promote sulfidogenesis instead of methanogenesis using waste carbon sources and a methanogenic granular sludge. Crude glycerol showed better results in terms of the consumed COD/S-Sulfate ratio compared with acetate, cheese whey, pig slurry, and vinasse. Then, the screening of several conditions (T, pH, and COD/S-Sulfate ratio) and the effects of air presence and dissolved sulfide inhibition on sulfate reduction was carried out. Sulfidogenesis was promoted at 35 °C, pH = 8.5, COD/S-Sulfate ratio above 7.0 g O_2 g^{-1} S, microaerophilic conditions, and dissolved sulfide concentrations below 250 mg S^{2-} L^{-1}. These conditions were tested for nearly 3 months in the startup and operation of a 2 L UASB reactor. An inlet sulfate concentration of 220 mg S L^{-1} and an HRT of 2 h were set. Removal efficiencies of approximately 90% were obtained with less than 20% of organic matter destined for biogas production.

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1. Introduction

Exhaust flue gases obtained from fossil fuel combustion or other industrial combustion processes can contain high amounts of SO_x, which must be removed before their emission to the atmosphere because they adversely affect human health, livestock, plants, and historic heritage among others (Srivastava and Jozewicz, 2001; Klimont et al., 2013). Physical and chemical treatments of flue gases have been mostly used in flue gas desulfurization (FGD) while recovering sulfur as a precipitate or as sulfuric acid (Srivastava et al., 2001). FGD technologies are efficient but generate waste streams that must be further treated (Philip and Deshusses, 2003). Presently, research focused on the energy optimization and valorization of waste effluents is becoming relevant because many wastes can be transformed into renewable energy and/or valuable products (Puyol et al., 2017). This is the case of the well-known SANI® process that focuses on the use of sulfate and sulfite contained in S laden effluents (such as liquid wastewater from the wet FGD process) as electron shuttles to treat chemical oxygen demand (COD) and nitrogen (Qian et al. 2015a, 2015b). The implementation of such a valorization-based process enables treating wastewater much more efficiently in terms of energy consumption. The transition

* Corresponding author.
E-mail address: David.Gabriel@uab.cat (D. Gabriel).

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toward a circular economy is a priority in the EU and has been applied to promote the conversion of wastes to added-value products to enhance the efficiency of resource utilization. In the actual bio-economy era, the establishment of circular economy would expand and diversify the market outlets of bio-based products as bio-based chemicals, biopolymers, fuels, and bio-energy (Maina et al., 2017).

The development of a biotechnological process to valorize S-rich effluents as elemental sulfur in an economical, robust, and environmentally friendly way is challenging because it could be applied not only to valorize SO_2 from flue gases but also sulfate- or sulfite-rich effluents generated in other industrial activities such as in pulp and paper factories, seafood processing, and tannery industry (Pol et al., 1998). Elemental sulfur is also a valuable product that is at present obtained from non-renewable energy resources and is used to produce sulfuric acid, fertilizers, and pigments among others (Lucheta and Lambais, 2012). To biologically generate elemental sulfur from sulfate or sulfite, a two-step process is required that consists of (1) sulfate or sulfite reduction to dissolved sulfide and (2) partial oxidation of total dissolved sulfide (TDS) to sulfur. To perform the reduction of sulfate or sulfite to TDS, a carbon source or an inorganic electron donor, such as H_2, is required (Muyzer and Stams, 2008). In this sense, many industrial processes generate waste liquid effluents with a high concentration of organic compounds, which could be used to obtain dissolved sulfide. The major problem of using organic matter to reduce sulfate to TDS as the first step toward elemental sulfur recovery is the competence of fermentative, acidogenic, and methanogenic microorganisms for the carbon source. Many authors who studied the competence between sulfate reducers and methanogens reported that the most relevant conditions that affect the competence for the organic electron donor and methanogenesis are the organic matter source; temperature; pH; COD/S-Sulfate ratio; and the inhibition by ammonia, O_2, and S^{2-} (Omil et al., 1997, 1998; Chen et al., 2008; Dar et al., 2008; Hu et al., 2015). In general, the literature focuses on the enhancement of methane production avoiding sulfidogenesis to reduce the presence of hydrogen sulfide in the biogas. On the contrary, there is a gap in the literature related to sulfidogenesis promotion against methane production using organic wastes under anaerobic conditions. Furthermore, several waste organic effluents have been used in research to produce biogas or to enhance the production of biogas in anaerobic digesters (such as vinasses, molasses, pig slurry, dairy industry wastewater, food industry wastewater, and olive mill waste effluents), but few compare such C sources (Filidei et al., 2003; Astals et al., 2013; Baba et al., 2013). Even less works studied the competition between sulfate reduction and methane production with different organic wastes (Jing et al., 2013).

The production of TDS under anaerobic conditions using many organic waste effluents was studied and promoted against methane production in this work. The operational feasibility of the two-step biological process mentioned above to valorize S-rich effluents relies on the selective utilization of organic waste effluents to reduce sulfate to TDS instead of producing methane. Moreover, in a sulfidogenic reactor, the production of methane would imply dissolved sulfide stripping and, as a consequence, the generation of an extremely corrosive biogas, which is difficult to manage. The use of an adequate organic waste effluent is also essential to have an economically feasible process because the cost of the electron donor should be minimal. Therefore, the main goal of our research was the obtainment of the optimal conditions to promote sulfidogenesis minimizing methanogenesis through a screening study. Afterwards, the suitability of the established conditions was evaluated through the startup and operation of an upflow anaerobic sludge blanket (UASB) reactor for sulfate reduction.

2. Materials and methods

2.1. Selection of a waste organic source for biological sulfate reduction

The sulfate reduction study was performed with a methanogenic granular sludge obtained from an industrial anaerobic digester treating wastewater from a paper recycling process. The first step of the study toward sulfidogenesis promotion was the screening of different waste organic sources (obtained as byproducts or waste effluents from industrial processes) because sulfate reduction was performed heterotrophically. The aim of this first screening was to select the C source supporting a high sulfidogenic rate with low methane production to optimize the use of COD in sulfate reduction.

Acetate, pig slurry, cheese whey, vinasse, and crude glycerol were selected as the C sources to perform sulfate reduction. The assessment of the sulfidogenic capacity of the granular sludge with each C source was performed by activity tests in sealed bottles. However, an acclimation period of the granular sludge to each C source was previously applied for 3 weeks to clearly evaluate the adequacy of the wastes for sulfidogenesis promotion with acclimated biomass. Then, three sealed bottles of 250 mL were prepared for each C source tested, which contained 5 g of settled granular sludge, mineral medium, and initial COD and sulfate concentrations of around 450 mg O_2 L^{-1} and 80 mg SO_4^{2-}-S L^{-1}, respectively. The composition of the mineral medium was as follows (g L^{-1}): NH_4Cl (0.5), K_2HPO_4 (3.5), 800 mL of distilled water, and 200 mL of tap water, adjusted to pH = 8.5 with NaOH (1 M). The total volume of each sealed bottle was 250 mL (150 mL of liquid phase and 100 mL of gas phase). The bottles were incubated in an orbital shaker at 150 rpm and 30 °C. Every three days (1 acclimation cycle), the supernatant was renewed and nitrogen was diffused in the headspace of the bottles after their sealing to ensure anaerobic conditions. Sulfate, COD, pH, and gas composition were analyzed at the beginning and at the end of the last cycle of the acclimation period to compare methanogenic and sulfidogenic capacities obtained with each C source.

The conditions set during the acclimation period and the activity tests were similar (initial granular sludge and sulfate concentrations of around 30 g L^{-1} and 80 mg SO_4^{2-}-S L^{-1}, respectively), although activity tests were performed for 24 h. Sulfidogenic and methanogenic activities were evaluated according to a methodology adapted from Dapena-Mora et al. (2007). The procedure followed before starting the activity tests consisted of a first wash of the acclimated granular sludge with a mineral medium free of sulfate and C source. The granular sludge was not centrifuged during the wash-up but settled, and the supernatant was decanted to avoid granule damaging. Once the supernatant was clean, the granular biomass acclimated to each C source was mixed and redistributed in three clean serum bottles as replicates to obtain similar biomass concentrations and comparable activities. The bottles were sealed and deoxygenated, and pulses of the C source and sulfate were added to each bottle to begin the activity tests. An initial concentration of around 700 mg COD L^{-1} was set to avoid organic carbon limitation. During activity tests, the bottles were continuously sampled and monitored. From two of the bottles prepared for each C source tested, six liquid samples were taken in 24 h and analyzed to obtain sulfate and COD concentrations. The third bottle was used to monitor biogas production by monitoring the pressure increase in the headspace (Centrepoint Electronics). The initial and final samples from the liquid phase of the third bottle were also analyzed to verify that similar sulfate reduction and COD degradation capacities were obtained in the three bottles incubated under the same conditions.

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