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Improving anaerobic digestion of pig manure by adding in the same reactor a stabilizing agent formulated with low-grade magnesium oxide



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ABSTRACT

Struvite precipitation and pig manure anaerobic digestion were coupled in the same reactor in order to mitigate the inhibitory effect of free ammonia and avoid precipitator costs. The stabilizing agent used to facilitate struvite precipitation was formulated with low-grade magnesium oxide by-product; an approach that would notably reduce struvite processing costs. The interaction between pig manure and stabilizing agent was analyzed in batch experiments, on a wide range of stabilizing agent additions from 5 to 100 kg m⁻³. The monitoring of the pH and ammonia removal during 24 h showed the high capacity of the stabilizing agent to remove ammonia; removal efficiencies above 80% were obtained from 40 kg m⁻³. However, a long-term anaerobic digester operation was required to assess the feasibility of the process and to ensure that the stabilizing agent does not introduce any harmful compound for the anaerobic biomass. In this vein, the addition of 5 and 30 kg $\mathrm{m^{-3}}$ of the stabilizing agent in a pig manure continuous digester resulted in a 25% (0.17 m³ kg⁻¹) and a 40% (0.19 $m^3 kg^{-1}$) increase in methane production per mass of volatile solid, respectively, when compared with the reference digester (0.13 m³ kg⁻¹). Moreover, the stability of the process during four hydraulic retention times guarantees that the stabilizing agent did not exert a negative effect on the consortium of microorganisms. Finally, scanning electron microscopy and X-ray diffraction analysis confirmed the presence of struvite as well as two precipitation mechanisms, struvite precipitation on the stabilizing agent surface and in the bulk solution.

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1. Introduction

Today, in most countries, intensive pig farming is concentrated in certain regions. There, the high production of pig manure linked to the insufficient amount of available land makes pig manure management and treatment important to minimize its contaminant potential [1,2]. Amongst all the treatment options, anaerobic digestion (AD) is a technology that is widely used since it: (i) avoids volatile organic compound emissions, (ii) stabilizes organic matter and (iii) recovers energy through methane production [3]. However, pig

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manure AD regularly presents low efficiencies due to the high concentration of ammonia and its low hydrolysis rate [3,4].

In AD, a wide range of inhibiting total ammonia nitrogen (TAN) concentrations have been reported, varying from 1.5 to 14 kg of nitrogen per cubic meter, where differences can be attributed to the characteristics of the substrates and the inoculum, the environmental conditions (mainly temperature and pH) and the adaptation periods [5,6]. TAN in anaerobic reactors has two forms: unionized or free ammonia (NH₃) and ionized ammonia or ammonium (NH₄). Although both forms have been reported as inhibitors of methanogenic activity NH₃ is the most toxic form. NH3 concentration depends basically on three parameters, i.e. TAN concentration, temperature and pH [7,8]. In order to mitigate the inhibitory effects of NH₃ many successful research efforts have been carried out before AD like air stripping, zeolite addition, clay addition and struvite precipitation [9-14]. However, these technologies would involve the construction and operation of another unit, which would be, in most cases, unfeasible for small and medium biogas plants.

High TAN removal efficiencies have been obtained for several waste streams by adding magnesium (Mg²⁺) and phosphate (PO_4^{3-}) to precipitate struvite (MgNH₄PO₄·6H₂0), which is a valuable slow-release fertilizer [15-17]. The addition of magnesium and phosphate is necessary to ensure the formation of struvite, since usually the amount of them in waste streams, like in pig manure, is not high enough to remove all NH₄ [12,17,18]. Nevertheless, the high price of raw materials and the large quantities of phosphate and magnesium required to achieve high ammonia removal efficiencies might cause a significant increase in processing costs, making struvite precipitation unfeasible [19,20]. To solve this problem, some researchers have used magnesium by-products as raw materials [18–21]. Another option to reduce the struvite processing cost is to couple anaerobic digestion and struvite precipitation in the same reactor. This approach has only been studied by Lee et al. [22] in a continuous food waste digester with MgCl₂ addition and by Uludag-Demirer et al. [23] in batch manure digesters with MgCl₂ and Mg(OH)₂ addition. However, to our knowledge, no references have been found evaluating the utilization of magnesium by-products within the reactor to precipitate struvite during anaerobic digestion. The introduction of inhibitory and/or toxic compounds for the anaerobic biomass is the main drawback when an industrial byproduct wants to be introduced in an anaerobic digester [1]. Therefore, in addition to the ammonium removal potential of the by-product, a long-term digester operation is required to assess the viability of the process.

The main objective of the present study is to couple anaerobic digestion and struvite precipitation in the same reactor where a stabilizing agent, formulated with a magnesium oxide by-product, is used to facilitate struvite precipitation. To achieve this goal the research sought to: (i) determine the ammonium removal efficiency when different doses of stabilizing agent are added to pig manure; (ii) compare the long-term operation performance of a pig manure anaerobic digester with and without the addition of a stabilizing agent; and (iii) identify the struvite precipitation mechanisms.

2. Materials and methods

2.1. Source of the low-grade magnesium oxide and pig manure

Low-grade magnesium oxide (LG-MgO) powder was supplied by Magnesitas Navarras, S.A. (Navarra, Spain). LG-MgO was generated during the calcination of natural magnesite in a rotary kiln at 1100 °C to obtain caustic calcined magnesia and then was collected in the fabric filters of the air pollution control system. Table 1 sets out the chemical composition of the major elements shown by X-ray fluorescence (XRF) to be the most stable corresponding oxides as well as the other physicochemical parameters.

Pig manure (PM, see Table 2 for its characterization) and digested pig manure, used as inoculum, were collected from a centralized anaerobic digestion plant. The facility digests at mesophilic conditions around 100,000 metric tons of pig manure per year utilizing the VALPUREN[®] process [2]. After collection, the pig manure was stored at 4 °C until it was used.

2.2. Formulation of the stabilizing agent

The stabilizing agent (SA) was formulated according to the procedure described in the patent [24]. Briefly, phosphoric acid was added slowly to an aqueous slurry of LG-MgO with a high solid-to-liquid ratio. The exothermic acid-base reaction generated a mixture with a high content of newberyite (MgHPO₄·3H₂O) and other magnesium phosphate compounds, such as magnesium phosphate tribasic (Mg₃(PO₄)₂) and magnesium phosphate dibasic (MgHPO₄), which coated the particles of LG-MgO that did not react on the basis of the shrinking core model [25,26]. Then, the compound was dried and crushed to a particle size of about 500 μ m.

As a result, instead of adding LG-MgO and phosphoric acid directly into the reactor, the stabilizing agent was formulated in advance obtaining a solid product in powder form, nontoxic or aggressive and easy to handle [24]. Table 1 presents the chemical composition of the major elements shown by

| Table 1 — X-ray fluorescence characterization of the LG-MgO and the stabilizing agent. | | |
|--|--------|------|
| | LG-MgO | SA |
| Oxides composition | | |
| MgO (%) | 63.7 | 31.8 |
| CaO (%) | 9.9 | 4.7 |
| SO ₃ (%) | 4.1 | 1.1 |
| Fe ₂ O ₃ (%) | 2.4 | 1.2 |
| SiO ₂ (%) | 2.0 | 0.9 |
| K ₂ O (%) | 0.3 | <0.1 |
| Al ₂ O ₃ (%) | 0.2 | <0.1 |
| V ₂ O ₅ (%) | 0.1 | 0.26 |
| MnO (%) | 0.1 | 0.1 |
| P ₂ O ₅ (%) | <0.1 | 32.8 |
| Physicochemical parameters | | |
| Loss of ignition (1100 °C) (%) | 16.7 | 27.4 |
| Density (kg m ⁻³) | 3200 | |
| Specific surface (m ² kg ⁻¹) | 10,400 | |

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