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Research paper

Optimization of solid-state anaerobic digestion through the percolate recirculation

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ABSTRACT

Anaerobic digestion is an environmentally sustainable way to manage organic waste, and it is able to enhance the recovery of organic carbon and nutrients in agricultural soils and to produce renewable energy. Solid-state anaerobic digestion (S-SAD) is a technology that permits the treatment of different type of residues, but is characterized by inhibition phenomena, resulting in a low operational stability. An experimental apparatus, equipped with a recirculation system for the digestate liquid fraction (percolate), was used to optimize the S-SAD system. Different frequencies of recirculation, one, two or four per day, were carried out to investigate how recirculation might affect the quality of the liquid fraction as well as the possible effects on biogas production and on the obtained solid digestate quality. Biogas production was positively affected by percolate spreading, especially when recirculation was performed 4 times per day. As shown by percolate chemical analyses, recirculation avoided the accumulation of volatile fatty acids in the liquid fraction, resulting in a better process stability. In addition, recirculation induced a large consumption of readily available compounds in the percolate, as shown by the depletion of water extractable organic C and total reducing sugars. The quality of the digested solid fraction was also improved by percolate recirculation in terms of the C/N ratio and organic N parameters. These findings showed that daily repeated recirculation of the liquid fraction is suitable to avoid inhibition phenomena during S-SAD and to improve the quality of the digestate solid fraction.

1. Introduction

Anaerobic digestion (AD) is a sustainable solution combining recycling of organic materials with the production of renewable energy (biogas) [1–5]. Animal residues-AD is a process that is used very successfully in a large number of countries because of its contribution to the reduction of greenhouse gas emissions into the atmosphere [6,7]. In fact, compared to raw materials, the use of the biomass obtained after AD resulted in a stable and partially hygienized organic product, characterized by the presence of stable organic matter [8,9].

The most common AD is based on wet technology, operating with a total solids (TS) concentration of <15% (w/w) [10,11]. This type of process is characterized by some significant technical drawbacks, i.e., the need for pre-treatments, large use of water, and consequent production of sludge that needs to be disposed of [12,13]. For all of these reasons, solid-state anaerobic digestion (S-SAD) is becoming more common [5] and consists of treating biomass and residues that maintain their shape when managed in an open pile. This condition is usually achieved with a TS concentration of >25% (w/w) [14]. The use of S-SAD allows many types of residues to be treated, with different qualities and rates of biodegradability [15,16]. Despite these positive aspects, S-SAD is characterized by inhibition phenomena, resulting in a low efficiency of biogas production. It is well known that during the anaerobic process, large amounts of volatile fatty acids (VFAs) are produced, resulting in a decreased pH (acidogenic phase). In particular, this first stage of AD is driven by acidogenic microbes, which are faster than methanogenic microorganisms, often causing the accumulation of VFAs [16,17]. Hence, S-SAD is exposed to inhibition

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phenomena caused by VFA accumulation, resulting in a low operational stability and an alteration of organic material degradation, which affects the final digestate quality [11,18]. It is also true that the use of solid inoculum might guarantee optimal conditions for methanogenic species, avoiding the inhibition phenomena [13,16] due to VFA accumulation. The use of solid inoculum causes a loss in the volume capacity of the anaerobic reactor or biocell. This issue may be solved by spreading the liquid fraction of the digestate, i.e., the percolate, on the material being treated (approximately 10% by weight of treated waste), improving the process stability and digestate quality [11,14].

Therefore, the use of percolate spreading has been proposed as a method to avoid inhibition phenomena [11,18]; but, it is also important to understand how the chemical characteristics of the percolate affect the S-SAD behaviour. Characterization of water extractable organic matter (WEOM) was widely studied to evaluate the composting process, showing that the quality of WEOM is a function of organic matter stability [19,20]. Even during S-SAD, it might be interesting to investigate how the quality of percolates changes during spreading in terms of WEOM and its influence on biogas production. To optimize S-SAD by percolate recirculation, the aim of the present study was to investigate how the frequency of recirculation might affect the quality of the liquid fraction as well as possible effects on biogas production and on the obtained solid digestate quality. Specifically, the hypotheses are as follows: i) the chemical characteristics of percolate, i.e., WEOM, may change with the frequency of recirculation, affecting the stability of S-SAD; ii) the recirculation of percolate induces the removal of inhibitor factors during S-SAD, improving the quality of the final solid digestate.

2. Materials and methods

2.1. Characteristics of the starting mixture

The initial mixture used for each trial consisted of pig slurry with straw added at a ratio of 3:1 (w/w); the inoculum, produced from previous S-SAD, was added at the same amount of pig slurry to the initial mixture. Prior to the start of the experiment, 2 L of demineralized water were added to the bottom part of the reactor, and the obtained mixture was analysed for its main chemical characteristics (Table 1).

2.2. Experimental apparatus

AD was carried out by means of laboratory reactors equipped with a recycling system for the liquid fraction (percolate) and a hydraulic gasometer to measure biogas production (Fig. 1).

Three polyethylene reactors with a 15 L capacity were used for each test. In particular, the percolate was collected through a tapped hole at the bottom of the reactor, while on the top, an output that allowed the produced biogas to pass was present. To create a separation between the percolate and solid fraction, a polyethylene filter with a porosity of 3 mm was fitted to allow passage of the liquid fraction and to prevent any solid fragments from occluding the recirculation system. The gasometer consisted of a water tank that was sealed with a hermetic cap and connected to a second tank by a plastic tube (internal diameter 4 mm). The biogas leaving the reactor generates pressure on the water present in the former tank, causing a transfer of the liquid to the second tank. The cumulative volume of water in the latter tank was measured daily, and the biogas production was calculated. This parameter was measured for 50 days, and the results were expressed as Nm^3 of biogas/t of VS. Moreover, the biogas composition and concentration (CH_4, CO_2 and O_2) were evaluated at 20 days for each trial by using an infrared portable gas detector (ETG-MCA 100, ETG Risorse e Tecnologia, Montiglio, Italy). The trials were performed in a climatic chamber under mesophilic conditions (35 ± 2 °C), where the temperature was maintained constant throughout the entire experimental period. During the experiment, we compared two different AD technologies: with and without percolate recirculation, the latter of which was used as the control. In particular, the effect of a different frequency of recirculation was investigated: once per day, twice per day (every 12 h), and four times per day (every 6 h), namely, as 1 S-SAD, 2 S-SAD, and 4 S-SAD, respectively. The duration of each recirculation was 45 min. The percolates were collected from each reactor at specific sampling times and then analysed for their chemical characteristics.

Table 1
Main chemical characteristics of the starting mixture .[a]

Parameter	Starting mixture
Dry matter (%)	20.7 ± 1.3
VS (g/kg)	877 ± 5.4
pH	8.22 ± 0.18
EC (dS/m)	5.54 ± 0.89
TOC (g/kg)	491.0 ± 5.7
TKN (g/kg)	11.9 ± 1.1
TAN (g/kg)	8.8 ± 0.7
Organic N (g/kg)	3.1
C/N	41.3

[a] All data are expressed on a dry weight basis.

2.3. Chemical analysis of the starting mixture and the final digestates

The starting mixture and final digestates resulting from the different treatments were analysed for their main chemical characteristics. The moisture content and total volatile solids (VS) were determined by weight loss upon drying at 105 °C in an oven for 24 h and ashing at 550 °C in a muffle furnace for 24 h, respectively. Electrical conductivity (EC) and pH were determined for the fresh samples at a 1:10 (w/v) solid/water suspension ratio. The total organic carbon (TOC) content was determined using the Springer-Klee wet dichromate oxidation method [21]. Fresh samples were used to determine the total Kjeldahl-N (TKN) and total ammonia nitrogen (TAN) by means of macro and micro-Kjeldahl distillation methods, respectively [22]. Total organic N was calculated by the difference between TKN and TAN. Total P was measured spectrophotometrically after digesting the dried samples with concentrated $H_2SO_4/HClO_4$ [22]. Total Cu and Zn were analysed by flame atomic absorption spectroscopy (AA 6800, Shimadzu Corp., Tokyo, Japan) after digesting the dried samples with concentrated $HNO_3/HClO_4$ [22]. All of the analyses were carried out in triplicate.

2.4. Percolates characterization

Samples of percolates were collected at 4, 8, 11, 15, 21, 28, 35, 42, and 50 days of AD from each laboratory reactor; the percolates were then analysed for the following parameters: process stability by means of the FOS/TAC (Flüchtige Organische Säuren/Totales Anorganisches Carbonat) ratio, water extractable organic C (WEOC), total phenolic compounds (TPC) and total reducing sugars (TRS).

The FOS/TAC parameter was used as an indicator of process stability and was evaluated as a ratio between the total VFAs (expressed as mg/L of CH_3COOH equivalent) and alkalinity (expressed as mg/L of $CaCO_3$) [23,24]. The FOS/TAC of percolates was determined in 20 mL of sample by means of an automatic titration device (Hach Lange TIM 840, Hach Lange Italia, Lainate,