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Anaerobic digestion of cheese whey: Energetic and nutritional potential for the dairy sector in developing countries

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ABSTRACT

Cheese whey (CW) is the main waste generated in the cheesemaking process and has high organic matter content and acidity. Therefore, CW disposal is a challenge for small to medium enterprises (SMEs) in the dairy industry that do not have any type of treatment plant. Anaerobic digestion (AD) is an attractive process for solving this problem. The aim of this research was to determine the biomethane and struvite precipitation potentials of CW from four dairy SMEs. First, changes in CW properties (organic matter and pH) were evaluated. Second, biomethane and struvite potentials were assessed using cattle slurry as inoculum. The organic matter in CW varied from 40 to 65 g VS/kg, 65 to 140 g COD/L, and 2 to 10 g/L for VFAs depending on the sampling time and type of sample. The pH of the CW samples ranged from 3 to 6.5. In the anaerobic biodegradability analysis, methane yields reached 0.51 to 0.60 L CH₄/g VS_{added}, which represented electrical and caloric potentials of 54 and 108 kW h/m³ for CW, respectively. Organic matter removal in all experiments was above 83%. Moreover, anaerobic digestates presented NH₄⁺/PO₄³⁻ molar ratios between 2.6 and 4.0, which are adequate for struvite precipitation with potential production of 8.5–10.4 g struvite/L CW. Finally, the use of biogas as energetic supplement and struvite as soil fertilizer, represents economic savings of US\$ 6.91/m³ CW and US\$ 5.75/m³ CW in terms of electricity and fertilizer use, respectively. The energetic, agricultural and economic potentials, evidence that AD process is a feasible alternative for cheese whey treatment.

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1. Introduction

The dairy industry plays an economically important part in the agricultural sector in most industrialized and many developing countries. Dairying improves food security and represents a source of employment and income to millions of smallholder families. More than 80% of the produced milk in developing countries comes from small producers (production of milk under 500 L/d) (Bennet et al., 2005). In Colombia, dairy companies use, on average, 1.35 × 10⁶ L of milk per year for cheesemaking, and 95% of these companies are small to medium enterprises (SMEs) (USP, 2015). This dairy chain generates residual liquid fraction well-known as CW, which represents approximately 90% of the milk employed. CW has an elevated organic load that varies in the range of 45–65 g/kg for volatile solids (VS) and 68–94 g/L for chemical oxygen demand (COD) (Dareioti and Kornaros, 2015; Gelegenis et al., 2007; Jasko et al., 2011; Riggio et al., 2015; Saddoud et al., 2007). In terms of particulate organic matter composition, CW contains

mainly carbohydrates (4–5%, of which lactose has the highest concentration), proteins (0.6–0.8%), and lipids (0.4–0.5%) (González Siso, 1996). Because of the high organic content of CW, alternative treatments have been developed, including a) land application as fertilizer, b) valorisation via biological treatments, and c) physicochemical treatments to produce and recover valuable compounds such as proteins and lactose. Physicochemical treatments have been successful for dairy companies with high processing volumes and enough capital to invest in their implementation. On the contrary, for SMEs, CW disposal is a challenge because they do not have the economic resources required for the proper treatment and valorisation. Therefore, these companies prefer to give away the residue for farm animal feeding or discharge it into the municipal sewage system, which can cause serious environmental hazards (Prazeres et al., 2012).

Anaerobic digestion (AD) can be a triple action process for CW treatment: pollution discharge reduction, energy obtainment, and nutrient recovery (Kataki, 2016). The application of AD to treat CW depends on the a) physicochemical composition of CW (organic matter, reduced alkalinity, and rapid acidification tendency), b) inoculum source (high buffer capacity), and c) reactor

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configuration (effluent recirculation) (Escalante et al., 2017). Biochemical methane potential (BMP) is the ultimate specific methane production of a substrate, for an indefinite degradation time. In other words, BMP allows the produced volume of methane generated by the substrate (in terms of VS or COD added) to be determined (Angelidaki et al., 2009). In addition, BMP is an indicator used to evaluate the viability of the anaerobic process and to assess the digester design based on operational parameters, such as methane production (B_0) and disintegration rate constant (k_{dis}) (Astals et al., 2013).

Previous research has reported BMP values for CW between 0.32 and 0.85 L CH₄/g VS (Ergüder et al., 2001; Labatut et al., 2011; Dreschke et al., 2015). Even though CW has high organic matter content, methane yield is limited owing to the production of volatile fatty acids (VFAs) during lactose fermentation. For this reason, acid accumulation may lead to a decrease in pH, growth of acetogenic bacteria, and inhibition of methanogenic activity (Yang et al., 2003).

AD is known for its organic matter stabilisation and removal. Nevertheless, during this process, most of the nutrients remain in the digestate in N/P ratios between 2 and 4 (Song et al., 2011). Although this digestate has good fertilizing properties, its direct application to crops has disadvantages, such as ammonium emission during irrigation (Nkoa, 2014) and introduction of pathogens to fields (Appels et al., 2008). To address these problems, practical solutions have been proposed for recovering nutrients from the digestate. One of these alternatives is to obtain fertilizers through the precipitation of magnesium ammonium phosphate hexahydrate (MgNH₄PO₄·6H₂O), also known as struvite (Stolzenburg et al., 2015). Struvite is formed as crystals, which are precipitated naturally when the molar ratio of Mg:NH₄:PO₄ is above 1:1:1 (Uludag-Demirer et al., 2005). Struvite has a lower water solubility in comparison with commercial fertilizers, which improves its yield and inhibits the uncontrolled dispersion of nutrients (Karak, et al., 2015).

In this context, for AD and struvite precipitation to be feasible, it is necessary that the substrate possesses two characteristics: availability and high nutrient content. Previous studies report the utilisation of different kinds of substrates to obtain struvite. Among these substrates, the uses of swine manure (Lin, 2012), wastewater digested liquors (Gonzalez et al., 2009), and cattle slurry (Tao et al., 2016) are remarkable. However, CW and its digestate have not been widely investigated for the application of this technology.

Therefore, the aim of this research was to identify opportunities to recover both energy (as BMP) and nutrient (as struvite precipitation potential (SPP)) resources by means of anaerobic degradability of CW from four dairy SMEs. In this sense, anaerobic potential (BMP and SPP) was the indicator for studying the feasibility of a synergic integration between dairy companies and dairy farms as an alternative CW treatment.

2. Materials and methods

This study was developed in three stages: a) evaluation of organic matter changes in CW from four dairy SMEs, b) BMP determination of CW, and c) SPP determination of biogas digestate from CW.

2.1. Evaluation of organic matter changes in CW from four dairy SMEs

CW from four dairy SMEs (C1 to C4) located in Santander, Colombia was studied. C1 recycles the generated CW (2300 L/d) for its own cheesemaking process. C2 gives their CW (2500 L/d) away for farm animal feeding. C3 produces 160 kg of cheese per day and generates 130 L of CW/d, and C4 is the smallest of the

companies and uses a homemade process, which generates 9 L of CW/d. See additional information about each company in Table S1 (Supplementary data).

For five weeks, samples of CW from each company were collected twice per week. Samples were carried to the biotechnology laboratory on ice and then stored at 4 °C in the laboratory.

Organic matter content, (VS) (SM 2450E) and (COD) (SM 5220D), and pH (SM 4500B) were evaluated according to protocols of the APHA (2005). Soluble organic matter, in terms of VFAs, was quantified using titration (Jobling et al., 2014). Data were analyzed by means of multiple sample comparison (LSD Fisher) using software Statgraphics Centurion XV for the significant difference between CW companies. The differences were considered significant for $p < 0.005$.

2.2. Determination of biochemical methane potential and energy considerations

2.2.1. Substrate and inoculum

The substrate for BMP tests was CW from the four SMEs. For each company, new samples were collected during five days (1 per day) and homogenized at the end of that period. The resultant sample was characterized in terms of VS and pH according to the APHA (2005), and VFAs were quantified using gas chromatography (Raposo et al., 2015).

Cattle slurry was employed as inoculum for the BMP tests. Its physicochemical characterization included 11.3 g VS/kg, pH of 7.79 ± 0.01 , and specific methanogenic activity (SMA) of 0.08 g CH₄ COD/g VS·d with an inoculum to acetate ratio of 5 g VS_{inoculum}/g acetate.

2.2.2. BMP test

BMP tests were carried out in 100 mL glass bottles (operational volume of 60 mL), in triplicate, according to the protocol suggested by Angelidaki et al. (2009). The samples were flushed with N₂/CO₂ (80/20% v/v), sealed with a thick butyl rubber stopper and an aluminum crimp, and stored at 37 ± 2 °C. The inoculum to substrate ratio, on VS basis, was kept at 1.5 for all tests. A positive control assay was performed using lactose. A blank (inoculum alone) was used to determine the background methane yield of the inoculum. Methane produced during the BMP assay was quantified via volumetric displacement of an alkaline solution (0.5 N). The displaced volume of methane was normalized and expressed in terms of specific methane production (SMP) as m³ CH₄/kg VS_{added}. Initial and final concentrations of VS and VFAs and the pH levels were quantified (APHA, 2005; Jobling et al., 2014).

The cumulative CH₄ production was adjusted to the first-order kinetic model according to Eq. (1):

$$B(t) = B_0 * (1 - e^{-k_{dis} * t}) \quad (1)$$

where k_{dis} is the degradation constant rate (d⁻¹), t is time, and B_0 is the BMP (Astals et al., 2013).

B_0 and k_{dis} were determined using the curve-fitting tool (cftool) of Matlab® software. Additionally, parameters were analyzed with Minitab 17 software, and media analysis was used to verify statistical differences between the kinetic parameters.

2.2.3. Energy considerations

The energetic contribution of CW volume was determined for each sample as follows:

$$P_{EE} = \rho_{CW} * VS_{CW} * BMP * \alpha_{EE} \quad (2)$$

$$P_{CE} = \rho_{CW} * VS_{CW} * BMP * \alpha_{CE} \quad (3)$$

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