



# Improvement of the biomethanization of sewage sludge by thermal pre-treatment and co-digestion with strawberry extrudate

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## ABSTRACT

The management of sewage sludge is an important issue in developed countries due to the highly polluting character of this waste. Biomethanization is a widely employed technology for this purpose, although it has several disadvantages such as low methane yield, poor biodegradability, and high sensitivity to nutrient imbalance. In this paper, a thermal pre-treatment (120 °C, 2 atm) is proposed to improve the biomethanization yield of sewage sludge and strawberry extrudate by solubilizing organic matter from the residual raw materials. Additionally, the co-digestion of sewage sludge with strawberry extrudate is evaluated as this combined treatment allows enhancing the nutrient balance and diluting inhibitors from sewage sludge. Therefore, the main aim of this study is the joint evaluation of the pre-treatment and co-digestion of this waste, which has never been described before in literature. The individual thermal pre-treatment of sewage sludge for 15 min was found to increase the soluble carbon concentration (mainly as volatile fatty acids), nitrogen, and phosphorus by 165%, 16%, and 24%, respectively. In contrast, the variation in the concentration of soluble carbon, nitrogen, and phosphorus for strawberry extrudate was as low as 10%, 32%, and 43%, respectively. Subsequent individual biomethanization tests showed a positive relationship between substrate solubilization and methane yield enhancement, with an increase of around 29% and 16% for sewage sludge and strawberry extrudate, respectively. Moreover, the co-digestion of sewage sludge and strawberry extrudate, both without pre-treatment, enhanced the stability and diluted the nitrogen concentration inside the digesters, although the methane yield was slightly lower than expected. In contrast, the most suitable combination was found to be the co-digestion of pre-treated sewage sludge and raw strawberry extrudate as it was also stable, but showed a synergy in methane production ( $237 \pm 29 \text{ mL}_{\text{STP}}/\text{g VS}$ ; STP: 0 °C, 1 atm). This combined treatment might be considered an interesting alternative for the combined treatment of both polluting wastes.

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## 1. Introduction

Vast amounts of sewage sludge are produced in wastewater treatment plants (WWTP) worldwide. The management of these wastes is an expensive and environmentally sensitive problem. The cost of treating sewage sludge can account for around 50% of the total operating costs in a WWTP. Moreover, there are several problems associated with the management of this waste, such as the presence of heavy metals, organic micropollutants, and pathogens, which require the hygienization of the sewage sludge

(Hendrickx, 2009). According to the European Union list of wastes laid down in Commission Decision 2000/532/CE, sewage sludge is a hazardous waste. Due to the environmental impacts of sewage sludge, such as odorous emissions or the generation of polluting leachate, landfill disposal is not a sustainable management solution. For this reason, the European Union has set the target to reduce final waste disposal by 35% in 2016 (compared to 2000) in all Member States (Lundin et al., 2004).

Among the currently available management methods, anaerobic digestion is a highly efficient process which allows the recovery of energy as biogas for electricity and on-site heating due to the high heating value of methane (Appels et al., 2008). Moreover, this method is one of the options set out in the Working Document on Sludge and Biowaste (2010), which is a proposal to revise Directive

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### Nomenclature

Alk	alkalinity (mg CaCO <sub>3</sub> /L)
C <sub>2</sub>	acetic acid (mg C <sub>2</sub> /kg)
COD	total chemical oxygen demand (g O <sub>2</sub> /kg)
D.L.	detection limit
FS	total fixed solids (mg/kg)
GAL	glucose, sodium acetate and lactic acid solution
N-TN	total nitrogen (mg/L; mg/kg)
P-P <sub>total</sub>	total phosphorus (mg/kg)
P <sub>soluble</sub>	soluble phosphorus (mg/kg)
STP	standard temperature and pressure conditions (0 °C, 1 atm)
sCOD	soluble chemical oxygen demand (g O <sub>2</sub> /kg)
TN <sub>soluble</sub>	soluble total nitrogen (mg/kg; mg/L)
TOC	total soluble organic carbon (mg/kg)
TS	total solids (g/kg)
VA	volatile acids (C <sub>2</sub> –C <sub>6</sub> ) (mg C <sub>2</sub> /L)
VS	total volatile solids (g/L; mg/kg; g/kg)
Y <sub>CH<sub>4</sub>/S</sub>	methane yield coefficient (mL CH <sub>4</sub> /g VS)
WWTP	wastewater treatment plant

86/278/EEC on the agricultural use of sewage sludge and sewage sludge management and whose application will be extended in coming years. Nevertheless, the anaerobic digestion of sewage sludge has several drawbacks, such as low methane production, poor biodegradability, and the presence of high concentrations of inhibitory compounds such as ammoniacal nitrogen (Bolzonella et al., 2005). It is therefore necessary to improve the biomethanization of this waste in order to ensure its viability and efficiency. The use of pre-treatments in sewage sludge management has been widely reported in the literature, including physical, biochemical, acidic or alkaline, heat-shock, freezing and thawing processes (Carrère et al., 2010; Cesaro and Belgiorno, 2014). Among the available options, thermal pre-treatments have been proposed by several authors to enhance the biomethanization of sewage sludge (Wang et al., 2010; Liu et al., 2012). Thermal pre-treatment allows the solubilization of the organic matter and facilitates the hydrolysis phase, which is the rate-limiting step when degrading solid organic waste with a high content of complex compounds (Ortega et al., 2008; Gabriel et al., 2011). However, the operational variables of the pre-treatments must be optimized to ensure the efficient use of energy. In this regard, the solubilization of organic matter (variation of the ratio between soluble and total organic matter concentration) has been used as an indicator of the process efficacy (Kim et al., 2003; Cesaro et al., 2014). Nevertheless, as the pre-treatments entail energy consumption and, occasionally, a decrease in the stability of the subsequent biomethanization process at increasing the acidity excessively, some authors have proposed the addition of a readily available co-substrate in order to enhance the methane yield coefficient, dilute the inhibitory compounds contained in the substrates, and compensate the excessive nitrogen concentration in sewage sludge (Chen et al., 2008). For this reason, strawberry extrudate might be an interesting co-substrate given its high C/N ratio and low heavy metal content. Strawberry extrudate is produced during the processing of strawberries to make marmalade, yogurt and flavorings, which employed about 21% (close to 1 million tons) of strawberry crops around the world in 2012 (FAOSTAT, 2014). Most of these products are obtained from mashed strawberries which are pressed to extract the desired compounds (flavors). The remaining waste extrudate is transformed into around 7% of the manufactured strawberry weight and must be managed adequately (Pollard et al., 2006). Previous

research into the anaerobic co-digestion of sewage sludge and strawberry extrudate has shown this technique to be effective in avoiding nitrogen inhibition in sewage sludge, although a positive synergy in methane generation was not observed. Despite this, studies on the anaerobic co-digestion of strawberry extrudate and fish waste have also shown an enhancement of the stability of the process, whereas the methane yield did not show positive synergy (Serrano et al., 2013, 2014). Furthermore, the joint improvement of the anaerobic digestion of sewage sludge by thermal pre-treatment and co-digestion with another substrate has not been described previously in the literature. To the best of our knowledge, the studies of Wang et al. (2014) and Cano et al. (2014) are the only ones that have reported the combination of thermal pre-treatment and sewage sludge co-digestion with another substrate. However, these studies only focused on the effect observed on methane production and not on the effect of the pre-treatment on the solubilization of the substrate.

The main objective of this work is to optimize the anaerobic digestion of sewage sludge through a thermal pre-treatment and/or its co-digestion with strawberry extrudate. The thermal pre-treatment was optimized to ensure the applicability of this study at industrial scale. The study could be considered of special interest for the centralized treatment of both polluting wastes, as well as to evaluate the viability of this treatment against other management methods such as landfill disposal.

## 2. Materials and methods

The experiment was carried out in accordance with the materials and methods described in this section. Sigma-Plot software (version 11.0) was used to create graphs, perform the statistical analysis (mean value and standard deviation), and fit the experimental data to the trends presented in this work.

### 2.1. Chemical analyses

The variables determined in the solid fraction of sewage sludge and strawberry extrudate before and after the thermal pre-treatment were total chemical oxygen demand (COD, g O<sub>2</sub>/kg), total solids (TS, g/kg), total fixed solids (FS, g/kg), total volatile solids (VS, g/kg), total phosphorus (P-P<sub>total</sub>, mg/kg), and total nitrogen (N-TN, mg/kg). All analyses were carried out in accordance with the test methods for the examination of composting and compost developed by the US Department of Agriculture and the US Composting Council (Thompson et al., 2001). Additionally, soluble chemical oxygen demand (sCOD, g O<sub>2</sub>/kg), total soluble organic carbon (TOC; mg/kg), total soluble nitrogen (TN<sub>soluble</sub>, mg/kg), soluble phosphorus (P<sub>soluble</sub>; g/kg), and separate volatile fatty acids were determined after extraction with distilled water (Thompson et al., 2001). TOC and TN<sub>soluble</sub> were determined using a Rosemount Analytical Dohrmann DC-190 carbon analyzer. The TOC analyzer was calibrated with a standard solution of potassium phthalate prior to the TOC analyses. Separate volatile fatty acids (acetic, propionic, butyric, isobutyric, valeric, isovaleric, and caproic acid) were also determined in both wastes. The determination was carried out using a Hewlett–Packard HP-5890 gas chromatograph equipped with a 15 m × 0.53 mm (i.d.) Nukol-silica semicapillary column and a flame ionization detector. The oven temperature was gradually increased from 100 °C to 150 °C at a rate of 4 °C/min. Helium (28.6 kPa), nitrogen (28.6 kPa), hydrogen (14.3 kPa), and air (28.6 kPa) were used as carrier gas at a flow rate of 50 mL/min.

Additionally, the following variables were determined in the effluents of the anaerobic reactors: pH, volatile fatty acids (VA, mg C<sub>2</sub>/L), alkalinity (Alk, mg CaCO<sub>3</sub>/L), and TN<sub>soluble</sub> (mg/L). The pH was measured using a Crison 2001 digital pH meter. All analyses were

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