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# Anaerobic digestion of solid agroindustrial waste in semi-continuous mode: Evaluation of mono-digestion and co-digestion systems

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#### ABSTRACT

The present study aimed at investigating the anaerobic digestion of four agroindustrial waste, namely cotton gin waste, winery waste, olive pomace and juice industry waste, in semi-continuous mode, conducting mono-digestion and co-digestion assays, using an artificial organic fraction sample as co-substrate. These assays were divided into two groups, in which different conditions were applied. Group I investigated the variation in two operational parameters, i.e. the organic loading rate (OLR) and the hydraulic retention time (HRT), while in Group II, the assays were fed with different substrates in a sequential order. Results showed more elevated specific methane yields for co-digestion assays compared with mono-digestion assays. Maximum yields were achieved at an OLR of 1.0 gVS/(L·d) and a HRT reduced to half of the initial. Further reduction of the HRT coupled to an increase of the OLR generally caused a significant decrease of specific methane yields, as well as one case of severe overloading, i.e. the mono-digestion of juice industry waste, which resulted in instability and ultimately system failure. Sequential feeding with different substrates led to a more equilibrated operation, especially for co-digestion systems, with higher specific methane yields being observed during the phases corresponding to winery waste and juice industry waste substrates. Overall more positive results were obtained in the cases in which the latter substrates were fed to the reactors at process startup.

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#### 1. Introduction

Agroindustrial activities represent one of the leading sectors of the economy, especially for countries such as those situated in the Mediterranean region, which are characterized by high production rates of agricultural commodities (Fountoulakis et al., 2008). Processing of agricultural commodities for the production of various food products leads to the generation of large amounts of waste materials. Among the most produced agroindustrial waste in Mediterranean countries are those originating from wine and olive oil production, as well as from citrus fruits (mainly orange) and cotton processing (Pellera and Gidarakos, 2016). The principal solid waste generated during wine production is grape marc, which is produced during the crushing, pressing, and draining stages. It is a lignocellulosic material rich in polyphenols and it is mostly composed of stalks, skins and seeds (Pala et al., 2014; Vatai et al., 2009). As far as olive oil extraction is concerned, waste generation depends on the type of system being used. Three-phase systems generate a type of solid waste, which is known as olive pomace

http://dx.doi.org/10.1016/j.wasman.2017.06.026 0956-053X/© 2017 Elsevier Ltd. All rights reserved. and is composed of olive pulp, skin, pieces of kernels and oil (Carlini et al., 2015; Kalderis and Diamadopoulos, 2010). Orange juice manufacturing generates large amounts of solid waste, which are mainly composed of peels (60–65%), while they also contain seeds and membrane residues (Martín et al., 2010; Negro et al., 2016). Cotton processing waste are produced during the ginning process and are usually comprised of burs, stalks, leaves, immature cottonseed and other plant materials (Hamawand et al., 2016; Placido and Capareda, 2013). Agroindustrial waste such as those described above, require appropriate management, in order to avoid potential environmental problems related to their disposal. To this regard, the use of these materials for energy production would seem as a suitable solution (Aboudi et al., 2015, 2016; Anjum et al., 2016).

Anaerobic digestion (AD) has been recognized as an effective waste management technology and has been used for treating various waste types, including agricultural and agroindustrial waste, the organic fraction of municipal solid waste, livestock effluents, sludge, etc. (Da Ros et al., 2016). AD involves the biological conversion of the organic matter present in waste materials, through the action of a microbial consortium under anaerobic conditions (Ward et al., 2008). The main product of AD is a methane-rich biogas. Methane obtained through AD is considered as a renewable energy

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Nomenclature			
AOF AW CGW FAN HRT JW OLR OP sCOD SMY	artificial organic fraction agroindustrial waste cotton gin waste free ammonia nitrogen (mg/L) hydraulic retention time (d) juice industry waste organic loading rate (gVS/(L·d)) olive pomace soluble chemical oxygen demand (mgO <sub>2</sub> /L) specific methane yield (mLCH <sub>4,STP</sub> /gVS <sub>added</sub> )	STP TA TAN TPH TS VA VS WW	standard temperature and pressure total alkalinity (mgCaCO <sub>3</sub> /L) total ammoniacal nitrogen (mg/L) total phenols (mgGAE/L) total solids (%, wet basis) volatile acids (mg/L) volatile solids (%, wet basis) winery waste

source and an excellent alternative to fossil fuels for heat and electricity generation. Apart from biogas, anaerobic digestion also results in the production of digestate, which usually has a nutrient-rich composition and, depending on its characteristics, may be composted or utilized as a fertilizer or soil conditioner (Barrantes Leiva et al., 2014; Fitamo et al., 2016; Kim and Oh, 2011; Ward et al., 2008). The efficient performance of anaerobic digestion systems depends on parameters such as temperature, pH, nutrients content, carbon to nitrogen ratio (C/N) and presence of inhibitory compounds. Often, during the digestion of certain substrates, some of these parameters may be encountered more or less distant from the optimal conditions. In those cases, the performance of the whole process may be compromised, eventually leading to failure (Esposito et al., 2012). Such phenomena depend on the composition of the substrates and are mostly related to nutrient balance and organic load issues (Mata-Alvarez et al., 2014). Therefore, care should be taken, in order to provide anaerobic digestion systems with feedstocks of suitable composition. To this purpose, co-digestion is frequently implemented (Fitamo et al., 2016).

Anaerobic co-digestion consists in the combined anaerobic treatment of two or more substrates, which have been mixed together (Astals et al., 2014; Ganesh et al., 2013). The most important parameter for obtaining a suitable feedstock composition is the appropriate selection of co-substrates and mixing ratios. In fact, co-substrates are usually chosen in order to have complementary characteristics. This favors the development of synergistic effects, which lead to an improved nutrient balance, often manifested by C/N within the optimal range. Additional benefits of codigestion include dilution of eventual potentially toxic compounds, adjustment of the moisture content, pH and buffer capacity of the mixture, increased content of biodegradable matter, as well as widening of the range of microbes participating to the process. All the above, ultimately result in improved methane production and digestate stability. Nevertheless, a univocal determination of optimal substrate mixing conditions is not possible, due to the wide variety of potential feedstock materials. Therefore, a targeted investigation should be carried out in each case (Astals et al., 2014; Esposito et al., 2012; Fitamo et al., 2016).

Apart from chemical parameters, operational parameters are also important for anaerobic digestion systems performance, with two of the most important being organic loading rate (OLR) and hydraulic retention time (HRT) (Aboudi et al., 2015; Gou et al., 2014; Ganesh et al., 2013; Ziganshin et al., 2016). In fact, finding the right balance between these two parameters is decisive for optimizing process efficiency (Aslanzadeh et al., 2014). Evidently, the optimum operational conditions are usually related to the characteristics of the feedstock materials and thus, should be determined for each separate case, either with single-substrates in mono-digestion systems, or mixed-substrates in co-digestion systems. Therefore, continuous research around this subject is necessary, especially when considering the high variability in the type and composition of possible substrates, within each geographical area.

In this study the anaerobic digestion of four agroindustrial waste, namely cotton gin waste (CGW), winery waste (WW), olive pomace (OP) and juice industry waste (JW), under semicontinuous operation was investigated. Both mono-digestion and co-digestion assays were conducted, with the latter including the use of an artificial organic fraction (AOF) sample as a cosubstrate. Currently, there is lack of studies regarding mono- and co-digestion of CGW, WW, OP and JW, under the conditions evaluated in the present study. In addition, feeding of these substrates in a sequential order, based on their seasonality, in both mono- and co-digestion modes, constitutes a major novelty, since it had not been studied before. The evaluation of such a feeding mode is important, considering that it would contribute to the controlled management and utilization of a variety of regional waste materials, by taking advantage of the seasonal variations in their availability. In addition, this strategy would also allow the operation and exploitation of such an anaerobic digestion system during longer periods of time, or even continuously.

#### 2. Materials and methods

#### 2.1. Substrates and inocula

The substrates used in this study included four agroindustrial waste (AW) samples and one artificial organic fraction (AOF) sample, which was prepared in the laboratory. The use of such an artificial sample aimed at obtaining a more controlled environment, which would allow the easier identification of the causes of different behaviors during the assays. The AW constitute the waste materials originating from four of the most important agroindustrial activities encountered in the Mediterranean region, namely cotton processing (cotton gin waste, CGW, i.e. cotton fiber, stalks, burs and leaves), wine production (winery waste, WW, i.e. grape skins, seeds and stalks), olive oil production (olive mill solid waste, i.e. olive pomace, OP) and citrus juice production (juice industry waste, JW, i.e. orange peels). After their collection, these samples were stored in zip-lock bags at -20 °C, while prior to their use each one of them was adequately prepared. Specifically, WW and JW were comminuted without drying using a food processor, CGW was dried at 60 °C and then comminuted to a particle size less than 500 µm, using a universal cutting mill, while OP was kept as received. The AOF material was an artificial sample, which intended to resemble the organic fraction of municipal solid waste (OFMSW). To this purpose, several types of household kitchen waste, including cooked pasta (2.98%) and rice (2.89%), bread (3.59%), cheese (0.87%), vegetables (36.8%, including lettuce,

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