



Combined effects of thermal pretreatment and increasing organic loading by co-substrate addition for enhancing municipal sewage sludge anaerobic digestion and energy production

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ABSTRACT

In this work, the thermal pretreatment of municipal sewage sludge (MSS) associated to its anaerobic co-digestion with the olive processing wastewater (OPW) was investigated for increasing complex organic matter disintegration and bio-conversion into energy. The hydrolysis of MSS (47%–49%) was performed at 120 °C during 30 min of pretreatment, which was confirmed by Fourier transform infrared (FTIR) spectroscopy. Results showed that increasing total solid (TS) from 0.58% to 3.2% has no effect on the suspended volatile solid (VS) disintegration. Anaerobic co-digestion of pretreated MSS (PMSS) mixed with OPW was investigated and the effect of increasing OPW proportion on the bio-methane potentials (BMP) was examined. The anaerobic digestion of better ratios of PMSS/OPW (80%/20% and 70%/30%) was also performed in sequencing batch reactors (ASBRs). The high biodegradation yields of VS and phenols (81% and 92%, respectively) were obtained at PMSS/OPW of 70%/30% corresponding to a methane yield of 0.441 L/gVS_{inlet}. Therefore, thermal pre-treatment of MSS and OPW addition improved significantly methane yield (50%–160%) and wastes stability. Furthermore, they increased total energy production from 30.9 kWh/ton to 93.5 kWh/ton, which would provide 0.56 M€/year net benefits only from the electric power, which is considered interesting. The excess thermal energy should be used for wastes pretreatment and digester heating.

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1. Introduction

High quantities of organic MSS are produced during biological activity in the wastewater treatment plants (WWTPs). Therefore, the mass of this by-product has increased significantly in the last decades due to the expansion of population and industry (Zhang et al., 2014; Yin et al., 2016). The disposal of this waste in the land-fill is no longer feasible solution due to the land scarcity and the increasingly stringent environmental control regulations (Wong et al., 2013). Among various methods, anaerobic digestion is a promising technology for MSS valorization, which needs to be made more effective to improve methane recovery yield and digestate quality (Bougrier et al., 2006; Gebrezgabher et al., 2010; Seng et al., 2010). However, existing anaerobic digesters operated at WWTPs has shown low energy productivity with a high content of H₂S in the produced biogas. In general, they were oversized and under loaded making sludge treatment not always economically feasible

(Zhang et al., 2014). Therefore, enhancement of methane production during anaerobic digestion of MSS could improve the energy self sufficiency of the WWTPs.

In recent years, different pretreatments and/or co-substrate addition were used as solutions to accelerate the production of biogas and improve the methane potential of MSS (Anjum et al., 2016). Several reports focused on physical, chemical and biological pretreatment methods prior to anaerobic digestion of MSS (Sahinkaya, 2014; Yin et al., 2016). Thermal hydrolysis technology, which is called CAMBI, is mainly used as a pre-treatment method on an industrial scale to improve the efficiency of anaerobic digestion of MSS. In fact, hydrothermal processing uses water at elevated temperatures and pressures to deconstruct organic and inorganic components of sludge (Hii et al., 2014). Morgan-Sagastume et al. (2011) reported that pretreatment by high-pressure thermal hydrolysis was shown to improve MSS fermentation by increasing significantly the VFA production yield compared to the fermentation of raw MSS. Therefore, thermal pretreatment seems to be more efficient in terms of total solid solubilization and pathogens destruction. Various temperatures, ranging from 60 °C to 270 °C have been tested (Bougrier et al., 2008; Ariunbaatar et al., 2014).

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In fact, in our previous work, [Ennouri et al. \(2016\)](#) showed that thermal pretreatment at 120 °C has improved significantly the efficiency of MSS anaerobic digestion in terms of methane production and VS reduction. They suggested also that thermal pretreatment increased *Archaea* relative abundance from 34% to 86%.

Despite an observed efficiency, the application of physical-chemical pretreatment method alone can be insufficiently in terms of environmental footprints and energy recovery. However, the co-digestion of organic wastes in a centralized digester is a very attractive solution for improving the process viability, supporting microbial activity, and providing the potential for higher methane yield ([Lahdheb et al., 2009](#); [Toumi et al., 2015](#)). Moreover, it gives the possibility of treating wastes, which cannot be easily treated separately at low cost. The co-digestion of MSS with typical agriculture residues ([Guo et al., 2015](#)), agro-industrial by-products ([Trulli and Torretta, 2015](#); [Maragkaki et al., 2017](#)) and food wastes ([Yin et al., 2016](#)) has been investigated. The co-digestion of MSS with OPW, which have high organic carbon concentration, is a suitable solution to adjust nutrient content of MSS and could improve biogas production ([Rodriguez et al., 2006](#); [Athanasoulia et al., 2012](#)). In fact, olive oil industry represents a very important agro-industrial sector in the economy of the Mediterranean countries ([Bernardi et al., 2017](#)). It produced approximately 30 million m³ of OPW per year ([Azaizeh and Jadoum, 2010](#)), of which about 1 million m³ per year in Tunisia ([Dammak et al., 2016](#)). The OPW has a high chemical oxygen demand (COD) up to 160 g/L ([Athanasoulia et al., 2012](#); [Elmakawy et al., 2014](#)).

Little information is available regarding the effect of combining physical-chemical pretreatments of MSS and its anaerobic co-digestion with organic wastes. Therefore, in this work the anaerobic co-digestion of thermal PMSS with OPW was firstly done in batch culture at different ratios of PMSS to OPW. In a second time, the synergetic effect of PMSS and OPW co-digestion on the methane productivity and VS removal was also investigated using ASBR system. Application of the ASBR in the digestion of mixed organic wastes is of interest because of its operational flexibility and the retention of slow-growing anaerobic bacteria within the reactor ([Timur and Öztürk, 1999](#); [Shao et al., 2008](#); [Handous et al., 2017](#)).

Results of these investigations are useful for the extrapolation and the implementation of anaerobic co-digestion of PMSS and OPW in the operational WWTPs for improving methane productivity and digesters stability. In the present study an energy and economic assessment was done to set the basis for a process scale up. The WWTP of Chotrana I (Tunis), treating 50,000 m³ of wastewater a day and that has adapted anaerobic digestion technology for excess sludge treatment, was considered as a model.

2. Materials and methods

2.1. Origin and characteristics of used wastes and inoculums

The MSS was collected from sludge thickeners of the WWTP of Chotrana-I (Tunis) treating 50,000 m³ of urban wastewater a day. This plant includes four independent semi-continuously digesters having volumes of 5250 m³ each, making a total digesters volume of 21,000 m³. The digesters are fed simultaneously with a mixture of primary and secondary sludge. Seed sludge, which was used in the start up of batch and ASBR experiments, was taken from within these industrial anaerobic digesters ([Bouallagui et al., 2010](#)). The OPW was collected from local (Tunis) three-phase olive oil extraction company ([Asses et al., 2009](#)).

The physical-chemical characteristics of wastes and inoculums (anaerobic seed sludge) are shown in [Table 1](#). The used substrates are rich in organic solids with VS/TS contents of about 44.8% and 76.3% for MSS and OPW, respectively. The MSS is rich in total

Table 1

Physical-chemical characteristics of the wastes and anaerobic seeding sludge used in this work.

Parameters	OPW	Raw MSS	Inoculums (Anaerobic sludge)
TS (g/L)	57.48 ± 1.3	5.8 ± 0.21	15.58 ± 0.3
VS (g/L)	43.9 ± 0.9	3.6 ± 0.01	7.48 ± 0.05
TSS (g/L)	0.68 ± 0.03	5.2 ± 0.2	9.18 ± 0.04
VSS (g/L)	0.6 ± 0.02	3.43 ± 0.01	4.74 ± 0.02
COD total (g/L)	108 ± 2.1	5.62 ± 0.02	–
COD soluble (g/L)	93.6 ± 1.2	0.46 ± 0.01	–
C/N ratio	52.1 ± 0.7	10.4 ± 0.02	22.3 ± 0.04
pH	5.07 ± 0.05	7.08 ± 0.03	7.45 ± 0.03
Conductivity (mS/cm)	16.27 ± 0.2	3.27 ± 0.1	13.2 ± 0.1
Salts (g/L)	7.98 ± 0.1	1.62 ± 0.01	1.65 ± 0.01
Phenols (mg/L)	3920 ± 22	8.45 ± 0.1	2.13 ± 0.01

TS: Total Solids; VS: Volatile Solids; TSS: Total Suspended Solids; VSS: Volatile Suspended Solid.

suspended solids (TSS), as 89.6% of TS, which is composed of microorganisms aggregates (floc of bacteria and protozoa) in the suspension ([Zita and Hermansson, 1994](#)). However, 99% of the dry matter of OPW is represented by the soluble matter, which is more easily accessible to the anaerobic bacteria. The fresh OPW was also characterized by a high total phenols content of 3.92 g/L.

2.2. Thermal pretreatment method and operating conditions

The thermal pretreatment of MSS was investigated in an autoclave at 120 °C for 30 min ([Ennouri et al., 2016](#)). MSS samples were placed in 1 L close bottles to avoid water loss by evaporation. After a treatment cycles of 5 min, 10 min, 15 min, 20 min, 25 min and 30 min, samples were immersed into cold water to guarantee a fast cooling. The TS content of sludge has a critical influence on the disintegration efficiency during pretreatment process ([Sahinkaya, 2014](#)). For this reason the TS content has to be optimized for an effective MSS pretreatment. Therefore, thermal solubilization experiments were carried out with MSS samples containing 0.58%, 1.2% and 3.2% TS. The solubilisation of VS was chosen to evaluate the degree of sludge disintegration. The solubilisation yield (SY), which defines the percentage of transfer of particulate organic matter to soluble organic matter, was expressed by the following equation:

$$SY (\%) = \frac{VSSi - VSSp}{VSSi} \times 100$$

Where, VSSi is the initial suspended VS (g/L) and VSSp(g/L) is the pre-treated suspended VS.

2.3. Reactors design and operational conditions

2.3.1. Batch experiments: BMP tests of the PMSS/OPW

After the thermal pretreatment of MSS, the PMSS was used for the preparation of different mixtures wastes M₁, M₂, M₃, M₄, M₅ and M₆ with different proportions between its VS content and the VS content of OPW. The M₁ was composed of 100% VS coming from PMSS. The M₂, M₃, M₄, M₅ and M₆ were composed of 90%/10%, 80% / 20%, 70% / 30%, 60% /40% and 50% /50% of VS coming from PMSS and OPW, respectively. The anaerobic co-digestion of the different mixtures wastes was conducted on the basis of the BMP approach, measuring the maximum amount of methane produced per gram of volatile solids contained in the organics used as substrates ([Angelidaki et al., 2009](#)). This experimental device contains height reactors (R₁–R₈) with a total capacity of 2.5 L and operated at controlled mesophilic temperature (37 °C). Reactors R₁–R₆ were filled with substrates (M₁–M₆ respectively) and inoculums (anaerobic seed sludge), according to substrate to inoculums ratio (SIR) equal to 1 between their VS contents.

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