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Effect of feedstock composition and organic loading rate during the mesophilic co-digestion of olive mill wastewater and swine manure

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ABSTRACT

In the present study, the optimisation of the mesophilic anaerobic co-digestion process of olive mill wastewaters (OMW) together with swine manure (SM) was investigated. Batch and continuous mode experiments were performed in order to define the most efficient mixing ratio and to determine the performance of the reactors under different organic loading rates (OLR). In batch experiment, the most efficient mixing ratio consisted of 40% OMW and 60% SM, since it presented the highest methane production equal to 277 mL CH_4/g COD, which corresponded to 79% of the theoretical yield. It was found that the effectiveness of this mixing ratio was not affected in the continuous operation of the reactors. The stepwise increase of the OLR did not affect negatively biomethanation, although the concentration of the inhibitory compounds of the OMW was higher. Under OLR of 4.4 g volatile solids/(L-feed·day) the methane yield of the reactors fed with 40% OMW reached 373 mL CH_4/gVS (78% of the theoretical yield). The findings of the present study proved that the co-digestion of OMW together with SM is a sustainable solution, capable to efficiently treat simultaneously these residual residues.

1. Introduction

Olive mills generate large quantities of solid and liquid wastes during the process of olive oil production. In the Mediterranean countries, the annual amount of olive mill wastewaters (OMW) is estimated to be up to 30 million m^3 [1]. OMW is a dark coloured, thick, aqueous and acidic waste (pH between 3 and 5.9), that possesses high pollutant load (50–150 g/L COD) [2,3]. Therefore, the disposal of OMW to water or ground receptors without a successful treatment process will lead to severe environmental problems.

In the cited literature, several technologies to treat OMW have been proposed, including physicochemical methods (e.g. dilution, evaporation, sedimentation) and biological methods (e.g. aerobic pretreatment, anaerobic digestion) [4]. Among these, anaerobic digestion has been reported to be one of the most promising methods in terms of effectiveness, environmental and economic impact [5].

Anaerobic digestion is a widely applied method to treat organic wastes, as it can greatly reduce the amount of organic matter, while in the same time provides energy in the form of biogas. During the recent years, anaerobic digestion process has been emerging spectacularly with an annual growth rate of 25% [6]. The increase in the growth rate is mainly However, the anaerobic digestion of OMW has been previously described as problematic, mainly because this substrate contains polyphenols and long chain fatty acid compounds that may inhibit the biological process [7]. Furthermore, the low amount of nitrogen and the low alkalinity of OMW have a negative impact on methanogenesis [8]. Another drawback during the anaerobic digestion of OMW is that this waste is seasonable and therefore it can be utilised in full scale biogas plants only during the months between November and February.

In order to overcome the above problems, many researchers have proposed the co-digestion of OMW together with other wastes. The general principle of the anaerobic co-digestion is to treat simultaneously different types of wastes in a reactor, in order to maximise the methane yield. As the OMW contains high fraction of organic matter but has low nitrogen content, the ideal substrate to be used in a co-digestion process would be one that possesses the opposite characteristics of OMW. For this reason, swine manure (SM) which is a rich in ammonium nitrogen waste could be a good complementary effluent for the co-digestion of the OMW [9]. Moreover, an additional motivation for the utilisation of swine

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manure in anaerobic digestion systems is that natural degradation of manure results in uncontrolled emission of CH_4 during storage, which is undesirable because of its global warming effects [6]. By mixing these two types of wastes the C/N ratio will be improved and also the ammonia toxicity, that could inhibit methanogenesis if SM was digested alone, will be avoided. Moreover, SM has high buffer capacity and will maintain the pH value of the mixed substrate within the optimum range for biomethanation. Marques [10] who investigated the co-digestion of OMW and piggery effluents in an up-flow anaerobic filter type reactor concluded that the process converted 70–80% of the influent COD. In another research, Angelidaki et al. [11] reported that the combined anaerobic digestion of olive mill effluents with SM using up-flow anaerobic sludge blanket (UASB) reactors is a potential and economic feasible method to treat olive mill effluents.

The aim of this study was to optimise the mesophilic anaerobic co-digestion of OMW and SM. Different mixing ratios of the two substrates were evaluated, in order to determine the most effective one, in terms of minimising the organic content and achieving the highest methane yield. Furthermore, the impact of the organic loading rate (OLR) on the co-digestion process was investigated, in order to define the tolerance of the process by a stepwise increase of the organic content of the mixed substrate. Finally, in the highest OLR, the effect of feedstock composition was tested again in order to determine, the most efficient mixing ratio under continuous operation of the reactors.

2. Materials and methods

2.1. Characterisation of the used substrates and preparation of the feedstock

OMW was produced by a three-phase centrifugation system for olive oil extraction located in Municipality of Thermaikos, Greece. OMW was diluted with distilled water in order to have the same volatile solids concentration with swine manure. Raw swine manure was obtained from a pig farm located in Municipality of Thermi, Greece. After arrival to the laboratory, the swine manure was shredded and sieved to separate large particles. Both substrates were stored at −20 °C and thawed at 4 °C for 2–3 days before use. The composition of the diluted OMW and raw swine manure are presented in Table 1.

2.2. Inoculum

The inoculum used in all experiments of the present study was well digested sludge derived from a mesophilic anaerobic reactor that was fed with OMW and SM at a ratio of 50:50 v/v. The total and working volume of the reactor were 2 L and 1.5 L respectively. The inoculum had a pH of 7.32, VS content equal to 6.6 g/L and the VS/TS ratio was 0.46.

Table 1
Characteristics of the used substrates.

Parameter	Unit	Swine manure (SM)	Olive mill wastewater (OMW)
		Values	
pH	–	7.38 ± 0.01	4.93 ± 0.01
Total solids (TS)	g/L	76.0 ± 0.2	57.5 ± 0.3
Volatile solids (VS)	g/L	55.8 ± 0.1	55.7 ± 0.2
Chemical oxygen demand (COD)	g/L	60.58 ± 0.02	142.94 ± 0.04
Total Kjeldahl nitrogen (TKN)	g/L	6.47 ± 0.02	1.78 ± 0.01
Ammonium nitrogen ($N-NH_4^+$)	g/L	2.82 ± 0.05	0.77 ± 0.02
Total volatile fatty acids (VFA)	g/L	2.2 ± 0.2	4.1 ± 0.3

2.3. Biochemical methane potential (BMP) test

A batch assay was carried out to determine the methane potential of different mixing ratios of OMW and SM. The experimental setup of the BMP test was consisted of 1 L glass bottles. The working volume of each glass bottle was 900 mL, while the volume of the headspace was 100 mL. Eight different SM:OMW mixing ratios were examined; 100:0, 90:10, 80:20, 70:30, 60:40, 50:50, 40:60 and 30:70. The examined mixing ratios were selected according to our preliminary exclusively batch experimental design, where four mixing ratios were applied 100:0, 80:20, 60:40, 30:70 [8]. However, in the present BMP test a distinctly wider range of mixing ratios was examined in order to define and to compare the effect of the co-digestion more extensively. Each mixing ratio was tested in twice in triplicate glass bottles in order to demonstrate reproducibility of the obtained results and to perform statistical analysis. Additionally, control reactors containing only inoculum were also used in order to estimate the background methane production due to organic matter content of the inoculum itself. The inoculum:substrate ratio in all bottles was 10:90 v/v. After inoculation the glass bottles were flushed with nitrogen gas to ensure anaerobic conditions, were sealed with rubber stoppers and finally were placed in an incubator that was operating at 35 ± 1 °C. Each glass bottle was equipped with a magnetic stirrer to keep the substrate in suspension. The composition of the methane in the biogas was measured in a daily basis until maximum total production of biogas was achieved.

2.4. Continuous mode experiments

Continuous mode experiments were carried out in two continuous stirred tank reactors (CSTR), denoted as R1 and R2. Each reactor had a total and a working volume of 3.9 L and 3 L, respectively and was continuously stirred with the assistance of a magnetic stirring device. The reactors were placed in an incubator in order to maintain the operating temperature stable at 35 ± 1 °C. The hydraulic retention time (HRT) of the reactors was kept constant at 30 days. The substrate was automatically provided to the reactors twice a day with the use of a double-head peristaltic pump. During the first experiment, the effect of OLR on the stability of the process was examined. Five OLR were tested; 1.2, 2.0, 2.8, 3.6 and 4.4 gVS/(L-feed·day). The stepwise increase of the OLR was achieved by diluting the mixed substrate with less quantities of distilled water. Both reactors were fed with the same substrate containing SM and OMW at the best mixing ratio found from the BMP test. During the second experiment, the OLR of the reactors was maintained steady at 4.4 gVS/(L-feed·day), while the mixing ratio of the SM and OMW in the substrate was changed. R1 was fed with substrate at a ratio that corresponded to 10% lower OMW concentration in the mixture, while R2 was fed with substrate at a ratio that corresponded to 10% higher OMW concentration in the mixture compared with the best mixing ratio found from the BMP test. In both experiments the biogas production was recorded daily, while methane content in biogas, pH and volatile fatty acids (VFA) concentration were measured once or twice per week.

2.5. Analytical methods

Total solids (TS), volatile solids (VS), pH, total Kjeldahl nitrogen (TKN), ammonium nitrogen and chemical oxygen demand (COD) were determined according to the Standard Methods for the