Anaerobic co-digestion of Fat Oil and Grease (FOG) with sewage sludge: Effect of thermal and thermo-alkaline pre-treatments on FOG for enhanced biogas production

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Abstract
The aim of this study was the assessment of co-digestion of sewage sludge with fat, oil and grease (FOG) and the investigation of the effects of thermal (70 °C and 100 °C) and thermo-alkaline (pH = 9, 70 and 100 °C) pre-treatments on biogas production. Co-digestion was carried out in lab-scale batch and semi-continuous digesters under mesophilic conditions (35 °C). Batch experiments of different substrate ratios showed an improvement in biogas production related to FOG loading. Biogas production and COD removal efficiency in batch tests showed that thermo-alkaline (pH = 9, 100 °C) pre-treatment on FOG was the most effective method (enhanced biogas production by 14.3 % over the control). According to the results from three semi-continuous digesters, the highest biogas production was observed in the digester which was fed with pre-treated FOG at 100 °C, whilst the percentage of COD and TS reduction reached 52.01 % and 46.96 %, respectively. Finally, the potential application of these findings in WWTPs is discussed, in terms of energy saving and economic profit.

Keywords
anaerobic co-digestion, FOG, thermal pre-treatment, biogas production, thermo-alkaline pretreatment

1. Introduction
Cyprus, being a Member State of the European Union, is obliged to conform to the European policies for waste management. The environmental legislation has not been fully implemented by the island, mainly due to the problematic areas of industrial waste and livestock treatment and management. Landfilling has so far been the
preferred method for waste disposal in the island. However, the investigation of sustainable and environmentally friendly alternatives such as biomass technologies employed in the production of biogas for heat and power generation, is of utmost importance [1].

For decades, anaerobic digestion has been used in Wastewater Treatment Plants (WWTPs) for biogas production. A recent advancement is the anaerobic digestion of sewage sludge for green energy production is the utilization of co-substrates. Co-digestion of sludge with various organic residues has proven to be an effective and low-cost improvement for obtaining increased methane yields [2, 3]. Anaerobic co-digestion provides significant advantages compared to the conventional alternative, such as co-substrate valorization, pH buffering, improved nutrient ratios, diluted inhibitory compounds etc [4, 5].

Lipid-rich wastes, called fat, oil and grease (FOG) are of particular interest in anaerobic digestion processes and have been employed in the co-digestion of sewage sludge [6]. Several industries produce FOG in large quantities, but it mainly originates from the food service industry (restaurants, food-processing factories etc.) Research is ongoing and many studies have demonstrated the importance of the usage of FOG as a co-substrate, due to its high theoretical methane potential compared to other substrates, i.e. 0.9-1.4 Lg⁻¹ at 65-70% CH₄ [7, 8]. Despite its capacity to increase biogas production, FOG can cause severe operational problems in WWTPs as it tends to clog pipes and drains, leads to biomass washout due to flotation, gives out unpleasant odors and attracts flies [9, 10]. Another significant problem associated with the anaerobic digestion of FOG, is the inhibition caused by long chain fatty acids (LCFAs) produced in the digester during FOG degradation. LCFAs have toxic effects on methanogenic bacteria and lead to digester failure as a result. The mechanism behind the toxicity is not fully known yet, but adsorption onto the cell membrane and cell lysis is suspected [11, 12]. Recent studies however, have shown that the negative impact of LCFAs on methanogens could be reversed and emphasize the use of acclimation as a key factor to avoiding inhibitory and toxic effects [13, 14].

Many studies have been conducted in the field of anaerobic co-digestion of sewage sludge with FOG and most of them report a significant increase in methane yield. Kambouris et al. (2009b) [15] have investigated the co-digestion of sewage sludge with grease trap sludge collected from restaurants (48% GS content on VS basis) and achieved 2.95 times higher methane yield. Luostarinen et al. (2009) [4] report in their findings an increased methane yield by 60%, resulting from co-digesting sewage sludge with grease from a meat processing plant. Other authors have demonstrated an increase in methane production exceeding 100% when adding grease sludge to digester [16, 17]. Finally, Davidsson et al. (2008) [18] have tested the co-digestion of sewage sludge and grease trap sludge in batch and semi-continuous processes. While batch processing resulted in high methane yield (845-928 Nm³ CH₄ / tVSadded), semi-continuous co-digestion was not feasible when excessive lipid content was present in the feed mixture.

Additionally, various pre-treatment methods can be used to assist in the production of higher methane yields. The pre-treatment methods described in the literature most often concern sewage sludge and they involve thermal hydrolysis, thermal-chemical treatment, enzymatic hydrolysis and ultrasonic treatment [19, 20]. A few studies have reported the effect of pre-treatment methods such as acid treatment and saponification on lipid-rich
wastes (FOG). Saponification leads to the conversion of insoluble lipids to soluble soaps, through hydrolysis and neutralization, which have surfactant properties and improve contact between microorganisms and substrates. FOG saponification has been investigated under various experimental conditions such as 80 °C with an excess of KOH [21], 80 °C with KOH at pH = 9 [22] and finally 60–120 °C and 150 °C with NaOH 0.3 % w/w [23].

The aim of the present study is to assess the feasibility of co-digesting FOG collected from the grease trap of a WWTP along with sewage sludge. Particularly, this article investigates the effects of thermal and thermo-alkaline pre-treatments of FOG on biogas production and presents an economical assessment on FOG anaerobic digestion.

2. Materials and Methods

2.1 Inoculum and substrates

All test substrates used for the experiments in this study were obtained from Amathus WWTP located in Moni area, Limassol, Cyprus. Anaerobic digester sludge (ADS) was used as the inoculum for initiating the digestion reactions in each test. Sewage sludge consisted of primary sludge (PS) from primary clarifier and waste-activated sludge (WAS). FOG was collected from the grease trap of the WWTP and was used as a co-substrate in the experimental units. All substrates were transported to the laboratory twice a week and were stored at 4 °C upon delivery. The characteristics of the inoculum and FOG utilized in this study are presented in Table 1.

2.2 Analytical methods

All quality parameters including pH, total solids (TS), volatile solids (VS), chemical oxygen demand (COD) and soluble chemical oxygen demand (SCOD) were analyzed according to Standard Methods for the Examination of Water and Wastewater [24]. Measurement of pH was performed using the potentiometric method with a digital pH/mV meter (Orion Digital pH/mV Meter, Model 611). The measurement of Chemical Oxygen Demand (COD) was based on the Standard Closed Reflux Colorimetric Method described in section 5220-D. Samples of 1 ml (original or diluted) were added to a Hach reflux tube, followed by 0.6 ml of digestion solution and 1.4 ml of sulfuric acid reagent. The tubes were tightly sealed and inverted three times to mix properly. The mixtures were then refluxed in a Hach COD reflux reactor (Model 45600) at 150 °C for 2 hours. After cooling, the samples were analyzed against de-ionized water using a Shimadzu UV/VIS scanning spectrophotometer (Model UV-2101/3101 PC) at a wavelength of 600 nm. The SCOD was measured using the supernatant of samples after centrifugation (15,000 rpm for 3 min) and filtration through a membrane filter (0.45 μm pore size).

2.3 Biodegradability batch assays

In this work, series of BMP tests based on the principles described by Owen et al. (1979) [25] were conducted to assess biogas production from the anaerobic co-digestion using FOG under different ratios as co-
substrate with sewage sludge. Anaerobic co-digestion was carried out in 125 mL serum bottles with a working volume of 100 mL (Table 2). All BMP bottles were flushed with a mixture N\textsubscript{2} / CO\textsubscript{2} (80/20 % as volume) gas, sealed with rubber stoppers and then incubated at 35 °C and 100 rpm. The volume of biogas production was measured by releasing the gas pressure in the serum bottles using a glass syringe (50 mL) allowing it to equilibrate with the atmospheric pressure. BMP tests lasted more than 30 days and every batch experiment was triplicated.

2.4 Thermal and thermo-alkaline batch pretreatments of FOG

The effect of thermal (70 °C and 100 °C) and thermo-alkaline pretreatments on FOG substrate was studied in batch tests. During thermal pre-treatment sample of FOG substrate was heated at 70 °C and 100 °C for duration of 30 minutes. For the thermo-alkaline pre-treatment samples of FOG were first adjusted to pH = 9 with NaOH 3M solution in a 250 mL beaker covered with plastic wrap and agitated with a magnetic stirrer for 15 min and then heated at 70 °C and 100 °C as described above. After cooling at room temperature, 50 mL of pretreated samples were collected and placed in 125 mL serum bottles and supplemented with ADS (25 mL), PS (12,5 mL) and WAS (12,5 mL). All serum bottles were flushed with a mixture N\textsubscript{2} / CO\textsubscript{2} (80/20 % as volume) gas and incubated under mesophilic conditions (35 °C). Prior to flushing the bottles and at the end of the procedure (post-digestion) samples were collected for COD determination.

2.5 Lab-scale semi-continuous anaerobic co-digestion

The digestion experiments were carried out in three lab-scale digesters (Duran bottles 2L) with 1L liquid volume and constant hydraulic retention time (20 d). Temperature was maintained at 35 °C (mesophilic) using a water bath. For the start-up period all digesters were inoculated with 2L ADS from WWTP. Digesters were run on a semi-continuous basis and they were manually fed with 100 mL (flow rate) of mixed substrates. Digesters were fed and withdrawn once a day. One digester (A) was fed with a mixture of PS and WAS and served as the control digester, while the other two digesters were fed with FOG (B) and pre-treated FOG (C) (Table 3). The daily biogas volume was measured by displacement of an acidified brine solution (10% NaCl w/v, 2% H\textsubscript{2}SO\textsubscript{4} v/v) in a graduated plastic cylinder.

3. Results and discussion

3.1 Effect of FOG co-substrate on biogas production

As seen in the (Fig. 1), adding FOG to the digester increases the biogas production potential. The highest biogas production was observed for the reactor containing 40% FOG. For the first 12 days of digestion, an accelerated rate of biogas production was observed whereas for the following days the production rate decreased. The digesters containing 20% (D3) and 40% (D4) FOG show similar results in the total production of biogas for the 36 days of digestion. The biogas volumes produced by the two digesters was 393 mL and 414 mL, respectively. Interestingly, the biogas production rate for the first 13 days is higher for the digester containing 20% FOG and not
the one containing 50% FOG. This suggests that the methanogenic microorganisms acclimatized more easily to the lower concentration of FOG.

3.2 Impact of thermal and thermo-alkaline pretreatments on batch anaerobic co-digestion

The effect of thermal pre-treatment on FOG at 70 °C and 100 °C for 30 minutes increases the potential for biogas production (Fig. 2). Thermal pre-treatment at 100 °C presents a slightly higher increase in biogas production compared to 70 °C. The total biogas production was 523 mL at 100 °C and 510 mL at 70 °C. Thermal pre-treatment accelerates the hydrolysis rate of FOG, thus leading to the cleavage of long chain biomolecules. The thermo-alkaline pre-treatment of FOG at pH=9 and 100 °C proved to be the most efficient pre-treatment method with total biogas production 529 mL. Biogas production rates were determined for the 14th and 26th day of co-digestion. The highest production rate for day 14 was observed for the thermo-alkaline pre-treatment at pH=9 and 100 °C, at 23.28 mL/d. For day 26 the biogas production rate for the thermal pre-treatment at 100 °C and the thermo-alkaline pre-treatment at pH= 9 and 100°C were 18.38 mL/d and 17.76 mL/d, respectively.

Fig. 3 depicts the COD removal efficiency for each pre-treatment method. As shown, the most efficient method is the thermo-alkaline pre-treatment at pH=9 and 100 °C, with a COD reduction percentage at 52.96 %. The thermal pre-treatment at 100 °C has also proved to be a potent method for COD removal reduction at 45.27 %. The results in Fig. 3 correspond to the total biogas production observed for each pre-treatment method, so it is evident that the highest COD reduction percentage corresponds to the highest biogas production.

3.3. Semi-continuous anaerobic digestion

Three laboratory digesters were operated for 57 days under different daily feed mixtures. The performance of the three digesters was evaluated based on biogas production and reduction of TS and COD, while the stability of their operation was monitored by means of temperature and pH control. The temperature was maintained between 35-37 °C throughout the experiments, thus allowing for a smooth operation. On the other hand, pH gradually decreased to 5.7 during the first days of operation for Digesters B (untreated FOG) and C (pre-treated FOG at 100 °C) and was then restored to 7.3 -7.5 with the addition of 1.5 g NaHCO₃. The decrease in pH is directly related to the addition of FOG in the digesters, because of high accumulation of (LCFAs).

Fig. 4 presents the daily biogas production for the experimental digesters. It is obvious that biogas production increased for the two digesters fed with untreated and pre-treated FOG at 100 °C, compared to the control digester (A). The daily average biogas production for digester (C) and digester (B) was 496, 7 ± 16, 9 mL/d and 420, 68 ± 26, 6 mL/d respectively, which correspond to a 26, 6 % and 13, 35 % higher biogas yield than that of digester (A). As shown by Fig. 4 η by the results obtained from the digesters, thermally pre-treated FOG at 100 °C (digester C) led to a higher biogas production compared to untreated FOG (digester B), both added in the feed mixture at the same ratio. This can be attributed to the fact that thermal pre-treatment on FOG at 100 °C leads to the improved disintegration of cell membranes, thus resulting in a greater solubilization of organic compounds [26]. COD removal efficiency was found to be 49, 01 % and 52, 01% for digesters (B) and (C) respectively (Fig. 5).
Moreover, the addition of FOG in the feed mixtures of the experimental units led to a greater reduction in TS, which was equal to 42, 35 % and 46, 96 % for digesters (B) and (C) respectively (Fig. 6). SCOD determination was also performed and the highest concentration was observed for digester (C), at 1655 mg / L. The results indicate an increase in soluble organics by the use of thermal pre-treatment on FOG. SCOD for digesters A and B was 1109 mg / L and 965 mg /L respectively and other studies have shown similar results. Luostarinen et al., [4] investigated the co-digestion of a mixture of animal by-products (ABP) from meat-processing industry and of sewage sludge found SCOD values equal to 620 mg / L and 760 mg / L, while the values obtained by Luste et al., [27], were between 1100 and 1200 mg / L. grease trap sludge from a meat-processing plant and sewage sludge

3.4. Economic assessment of co-digestion of FOG with sewage sludge

The results obtained from the co-digestion of FOG with sewage sludge showed an increase in biogas production under different ratios of FOG substrate. However, it is important to assess the economic feasibility of the results and their potential implementation in the existing digestion system at the Amathus WWTP in Moni area (Limassol, Cyprus). The WWTP has a treatment capacity of 272 000 population equivalent (PE) with a design wastewater flow equal to 40 000 m³/d. The design organic load is 16,320 kg/d and 31,920 kg/d for BOD and COD respectively. Treatment of wastewater consists of mechanical, biological and tertiary treatment of the effluent and anaerobic digestion of primary (PS) and waste activated sludge (WAS). Average daily flow rates to the anaerobic digesters are 184 m³/d of primary sludge (2, 5% TS) and 123 m³/d of waste activated sludge (5% TS), with an average biogas production of 2000 m³/d. The produced biogas is used in a combined heat and power energy recovery system (CHP) consisting of internal combustion engines, for generating electrical power and thermal energy with efficiencies equal to 36, 2 % and 49,1 %, respectively. The daily amount of FOG collected by grease trap at the Amathus WWTP is 2 m³/d.

Based on the experimental results, the maximum recommendable amount of FOG to be loaded to the digestion system is 5 % or 10 % of the incoming primary sludge (PS) and waste activated sludge (WAS). The biogas production efficiency of anaerobic digestion loaded with 5 % and 10 % FOG was 20, 7 % and 16, 2 % higher than the one without added FOG. Furthermore, results indicate that the necessary investment for the co-digestion of FOG is an economically feasible solution with significant future incomes for the WWTP (Table 4). The price for the delivery of electricity to the Public Electricity Authority of Cyprus (EAC) is € 0, 11 per kWh. The annual financial profit on electricity sales, by loading the existing digester with 5 % and 10 % FOG will reach 17, 15 % and 13, 9 %. According to the daily flow rates of primary and waste activated sludge into the anaerobic digester, the daily requirement of FOG is calculated at 16.15 m³/d for 5 % and 34 m³/d for 10 % addition. In 2014 about 2040 m³ of FOG and scum was disposed of from the WWTP. Likewise, FOG from hotels, restaurants and other enterprises equipped with grease traps are also disposed of. In both cases, FOG could be collected and processed at the WWTP as a co-substrate with sewage sludge for enhanced biogas production.
4. Conclusion

Anaerobic co-digestion of FOG and sewage sludge successfully increases the biogas production and allows for the beneficial use usage of FOG. Moreover, different pre-treatment processes on FOG, such as thermal and thermo-alkaline, may be enhanced the biogas potential to an even greater extent. Co-digesting FOG with sewage sludge it is an environmentally friendly alternative to landfilling and thus provides a significant environmental advantage. Finally, the feasibility study on the utilization of FOG in biogas production at the Limassol – Amathus WWTP, may provide greater economic incentives for the use of excess biogas to generate electricity and thermal energy, due to the expected rise in annual profit.

Acknowledgements

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References

Fig. 1 Biogas production from the co-digestion of FOG and sewage sludge under different ratios.

![Graph showing biogas production from co-digestion of FOG and sewage sludge under different ratios.](image)

- D1 (5 % v/v)
- D2 (10 % v/v)
- D3 (20 % v/v)
- D4 (40 % v/v)
- D5 (control)

Fig. 2 Biogas production by applying thermal and thermo-alkaline pre-treatments of FOG in batch mode.

![Graph showing biogas production by applying thermal and thermo-alkaline pre-treatments of FOG in batch mode.](image)

- Thermo-alkaline (pH=9, 70 °C)
- Thermo-alkaline (pH=9, 100 °C)
- Thermal (100 °C)
- Thermal (70 °C)
- Control
Fig. 4 Daily biogas production of semi-continuous digesters

Fig. 3 COD reduction percentage for each pre-treatment method
**Fig. 5** COD reduction of each digester

![COD Reduction](image_url)

**Fig. 6** TS reduction of each digester

![TS Reduction](image_url)
### Table 1 Characteristics of the inoculum and FOG

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<thead>
<tr>
<th>Parameter</th>
<th>ADS</th>
<th>FOG</th>
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<tr>
<td>pH</td>
<td>7.3-7.6</td>
<td>4.1-4.8</td>
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<tr>
<td>TS (g/L)(^a)</td>
<td>42.66</td>
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<td>TVS (g/L)(^a)</td>
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<tr>
<td>COD (g/L)(^a)</td>
<td>68.3</td>
<td>29.9</td>
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\(^a\): average values

### Table 2 Composition of batch digesters

<table>
<thead>
<tr>
<th>Digester</th>
<th>ADS (mL)</th>
<th>PS (mL)</th>
<th>WAS (mL)</th>
<th>FOG (mL)</th>
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<tr>
<td>D1 (5 %) (^a)</td>
<td>50</td>
<td>22.5</td>
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<td>D2 (10 %)</td>
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<td>20</td>
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<td>D5 (control)</td>
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\(^a\): Percentages are given by volume (v/v).

### Table 3 Daily feed of semi-continuous digesters

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<tr>
<th>Digester</th>
<th>PS (mL)</th>
<th>WAS (mL)</th>
<th>FOG (mL)</th>
<th>pre-treated FOG at 100 °C (mL)</th>
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<tr>
<td>A</td>
<td>50</td>
<td>50</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>B</td>
<td>25</td>
<td>25</td>
<td>50</td>
<td>0</td>
</tr>
<tr>
<td>C</td>
<td>25</td>
<td>25</td>
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### Table 4 Profit estimation loading FOG on anaerobic digestion

<table>
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<tr>
<th></th>
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<th>Digester with 5 % FOG</th>
<th>Digester with 10 % FOG</th>
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<tbody>
<tr>
<td>Biogas production</td>
<td>2000 Nm³/d</td>
<td>2414 Nm³/d</td>
<td>2324 Nm³/d</td>
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<tr>
<td>Electricity production</td>
<td>4686 kWh/d</td>
<td>5656 kWh/d</td>
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<tr>
<td>Heat power production</td>
<td>264.8 kWh</td>
<td>319.6 kWh</td>
<td>307.7 kWh</td>
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<td>€ 622, 16</td>
<td>€ 598, 95</td>
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<td>Incomes from sale electricity per day</td>
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<td>€ 227.088</td>
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