



Article Estimation of Energy Recovery Potential from Primary Residues of Four Municipal Wastewater Treatment Plants

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Abstract: Wastewater treatment plants have been traditionally developed for the aerobic degradation of effluent organic matter, and are associated with high energy consumption. The adoption of sustainable development targets favors the utilization of every available energy source, and the current work aims at the identification of biomethane potential from non-conventional sources derived from municipal wastewater treatment processes. Byproducts derived from the primary treatment process stage were collected from four sewage treatment plants in Greece with great variation in design capacity and servicing areas with wide human activities, affecting the quality of the influents and the corresponding primary wastes. The samples were characterized for the determination of their solids and fats content, as well as the concentration of leached organic matter and nutrients, and were subjected to anaerobic digestion treatment for the measurement of their biomethane production potential according to standardized procedures. All samples exhibited potential for biogas utilization, with screenings collected from a treatment plant receiving wastewater from an area with combined rural and agro-industrial activities presenting the highest potential. Nevertheless, these samples had a methanogens doubling time of around 1.3 days, while screenings from a high-capacity unit proved to have a methanogens doubling time of less than 1 day. On the other hand, floatings from grit chambers presented the smallest potential for energy utilization. Nevertheless, these wastes can be utilized for energy production, potentially in secondary sludge co-digestion units, converting a treatment plant from an energy demanding to a zero energy or even a power production process.

Keywords: screenings; fats; biogas potential; wastewater treatment plant; energy utilization; anaerobic digestion

1. Introduction

The activated sludge process has been identified as an efficient method for the treatment of a wide range of wastewaters for a long time. The process is based on the aerobic degradation of organics, while pretreatment is required for the upstream removal of suspended solids contained in raw wastewaters. The design of a wastewater treatment plant is mainly carried out in order to satisfy the corresponding effluent quality regulations while less efforts are taken towards energy requirements. Effluent quality is achieved at the cost of significant energy consumption. Aerobic processes are highly energy-demanding techniques with an average energy input reported to range from 0.30 to 1.89 kWh/m³ depending on the treatment method used and the influent properties [1]. Secondary aeration accounts for about 50% of the total electricity demand and 25–40% of the plant operating costs [1–3]. Wastewater treatment plants are considered as the highest municipal



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). energy consumers [4]. This issue is becoming crucial since the overall energy demand by wastewater treatment plants in European countries is estimated at 27 TWh/yr [5].

The increase of electricity costs in combination with the awareness of climate change has given rise to efforts aiming at the development of energy conservation policies and the implementation of energy-efficient measures that can contribute to the international sustainability development goals. Anaerobic digestion of excess sewage sludge represents a common practice in activated sludge processes, developed rather as a method of stabilization of wasted sludge than focusing on its energy valorization. Dedicated methodologies have been applied including energy surveillance, benchmarking, and auditing tools adapted to wastewater treatment plants, resulting in 20–40% savings in energy consumption [6]. However, these methods ignore the inherent energy content of raw wastewaters.

The energy content of raw wastewater has been measured by bomb calorimetry at about 14.7 kJ/g COD, exceeding almost nine times the electricity demand for its aerobic treatment [7]. Taking into account a daily production of about 100 g COD/person, the energy content of wastewaters for a city with 1 million inhabitants is estimated at around 1470 MJ/d; these estimations are carried out without considering the energy content of industrial wastewaters, while recent calculations report an energy content chemically bound to influents exceeding 153 KWh/person equivalent-year [8,9]. Nevertheless, the benefits of energy utilization of the contents of raw influents have not been given much attention so far, due to the traditional concept of wastewater considered as a waste requiring energy for its safe disposal and not as a valuable resource. Energy harvesting from raw influents might support efforts towards self-energy efficiency of wastewater treatment plants, shifting them from energy consumers to zero-energy plants or even to power producers.

Traditionally, anaerobic digestion of excess sludge is the primary energy harvesting method often applied in wastewater treatment plants. However, an alternative potential energy source is represented by the primary screenings including papers, wood, plastics, stones, metal pieces, rag, etc., and byproducts such as fats, oils, and grease (FOG). These materials can be directly valorized since they are separated during the primary treatment stages in existing units, without the need for the installation of additional facilities. Screenings are currently disposed in sanitary landfills, representing an additional operation cost while contributing to GHG emissions during their biological degradation in landfills [10].

The energy utilization of these byproducts has received less interest mainly due to their small amount compared to the much larger amounts of excess sludge [11]. For example, about 1.5 kg/person-equivalent of screenings have been estimated for 2007 in Germany [12], while in another study in France, average production was estimated to range from 0.53 to 3.49 kg/person [11]. However, the amount of screenings and FOGs is expected to increase due to intense urbanization, the enforcement of more strict EU regulations related to primary treatment yields, and the application of measures to alleviate the pressure due to increased influent loadings bringing a wastewater treatment plant close to the design capacity [13]. Moreover, the utilization of every available energy resource has been recently re-examined for various reasons. These include the development of advanced technological innovations for fine screening (enhancing screening removal), which is a pre-requirement in advanced membrane bioreactor units, as well as the adoption of sustainability principles by EU countries, which are associated with less waste diverted to landfills and the utilization of non-conventional wastes for energy and resource recovery [14,15].

Anaerobic digestion represents a challenging method for the energy valorization of non-conventional wastes from wastewater treatment plants, which is favored since it is a process conventionally applied for excess sludge treatment. However, very few studies have been reported in the literature dealing with the examination of biogas production potential of screenings and FOGs, mainly due to the non-homogeneous character of these wastes and the utilization of FOGs for biodiesel production [11,16]. The concentration of volatiles solids in screenings may exceed 90% of total solids [13,17], resulting in biogas production as high as 0.62 L/kg vs. [18]. The application of fine screenings in a municipal wastewater treatment plant and the following anaerobic digestion demonstrated a 40%

reduction of energy demand in the Netherlands [19]. On the other hand, the addition of FOGs collected from restaurants in a municipal sludge digester resulted in a 30 to 80% increase in biogas production [20].

Nevertheless, the biomethane production rate is greatly affected by the composition of screenings and FOGs. The characteristics of these wastes are related to a great number of factors such as the presence of a combined or separated sewer system, the number and the type of the sewer system pumping stations upstream to the treatment plant, the catchment area, and the primary stage installations. In addition, the properties of the wastes can be varied due to specific climatic conditions, consumers behavior, and time frameworks, including seasonal, weekly, or daily dimensions [11].

The objectives of this work include the examination of energy valorization of nonconventional wastes produced from municipal wastewater treatment plants, the determination of the corresponding biomethane potential during anaerobic digestion, and the identification of the relation to their composition and the type of primary treatment stage configuration. The primary target of the work is the justification of the beneficial role of screenings and FOGs towards energy utilization, as an alternative to currently used management methods of these side streams.

2. Materials and Methods

2.1. Samples Collection

Samples were collected from different areas of the primary treatment stage of four wastewater treatment plants (WWTP) located in Northern Greece, with various design capacities and servicing areas including a wide range of human activities. Samples identification and information on their origin are provided in Table 1.

Table 1. Identification of samples collected from the primary treatment stage of four wastewater treatment plants and their characteristics.

No	Wastewater Treatment Plant Identification		Substanta Origin	C11
	Design Capacity, m ³ /Day	Area	- Substrate Origin	Symbol
1	8400	Urban, semi-rural, piscatorial	Screening from mechanical bar screen, 6 mm spacing Floatings from aerated grit chamber	ASC AFL
2	11,400	Urban, rural	Screening from mechanical bar screen, 3 mm spacing Floatings from aerated grit chamber	KSC KFL
3	23,400	Urban, rural, agro-industrial	Screening from mechanical bar screen, 50 mm spacing Screening from mechanical bar screen, 15 mm spacing Screenings from mechanical bar screen, 50 mm spacing	LSCC LSCF LSCST
			Floatings from aerated grit chamber	LFL
4	155,150	Urban, industrial	Screenings from mechanical bar screen, 10 mm spacing Floatings from aerated grit chamber	TSC TFL
			Screenings from 5 mm bar screen of sludge from primary sedimentation	TPS
			Sludge anaerobic digester used for inoculation	1

About 1 kg of each sample was collected, placed in plastic bottles, and transferred to the laboratory for further analysis. Samples were stored at 4 °C in a constant temperature refrigerator until the time of analysis. Prior to storage, samples were subjected to milling using a knife mill in order to receive particles of similar size of about 0.1 mm.

2.2. Chemical Analysis

The analysis of the samples took place for the determination of total and volatile solids, and fats using standard methods, i.e., drying at 105 °C for total solids; thermal treatment at 550 °C for volatile solids; and Soxhlet extraction for total fats [21]. Water-leached organic matter and nutrients concentrations were determined by the addition of 0.3 g of dry

solids in 20 mL of deionized water and stirring for 20 min, followed by centrifugation and determination of COD, N-NH₄, and P-PO₄ in the aqueous phase. Parameters were measured using a HACH-Dr Lange DR3900 spectrophotometer and the corresponding standard cuvette test kits, i.e., the LCK714 COD kit, the LCK303 ammonium kit, and the LCK049 orthophosphates test kit, respectively.

2.3. Biochemical Methane Potential

The biochemical methane potential (BMP) was determined by anaerobic digestion treatment performed in batch reactors. Glass reactors with a total volume of 322 mL and a working volume of 120 mL were used. In each reactor an amount of substrate containing 1 g of vs. was added followed by the inoculum to reach a working volume of 120 mL and achieve 202 mL headspace. Inoculum was collected from the anaerobic digester of the municipal wastewater treatment plant No. 4, with the highest design capacity operating for a long period. The glass reactors were sealed using rubber stoppers and aluminum caps suitable for retrieving gas samples. In addition, a blank sample was prepared, containing 120 mL of pure inoculum. The treatment for each of the 12 substrates along with the blank were carried out in triplicates. All reactors were initially flushed with pure nitrogen gas (N₂), in order to ensure anaerobic conditions. The batch reactors were incubated at a temperature of 37 ± 1 °C for 80 days.

The production of methane (CH₄) was determined by injecting gas samples from the reactors into a gas chromatographer (GC-2010plusAT, SHIMADZU, Kyoto, Japan) equipped with an appropriate detector and columns [22]. For each sample, 150 μ L of gas were acquired from the headspace of the reactor with a gas-tight syringe outfitted with a pressure lock. During the first 10 days the reactors gas composition was monitored daily and after that on a bi-daily schedule. A standard gas mixture (60% CH₄, 40% CO₂) was utilized to determine the % concentration of CH₄ of each gas sample. The obtained peak area was compared to that of a standard gas mixture (60% CH₄, 40% CO₂) injected at atmospheric pressure in the chromatographer. The calculation of the volume of the produced CH₄ was carried out by the multiplication of the headspace volume of each reactor with the % concentration of CH₄ of each gas sample [23].

For each substrate, the maximum specific growth rate of the methanogens (μ_{max}) was derived from a graph of the natural logarithm of methane (CH₄) production as a function of time, and calculation of the maximum value from the tangent's angle was conducted [24]. The methanogens' maximum doubling time (T_{doublemax}) was calculated as the quotient of the natural logarithm of 2 (ln2) divided by the methanogens μ_{max} [25].

3. Results

The content of primary samples in total and volatile solids and fats is shown in Figure 1. Efforts were taken to collect all samples at the same time period, in order to exclude potential seasonal variations. As shown, samples presented high total solids content, while volatile solids ranged from as low as 20% up to 90% of the total solids.

In addition to solid and fat content, a significant role in the energy valorization of samples through anaerobic fermentation is played by the composition of leachable organic matter, as well as the concentration of nutrients, i.e., nitrogen and phosphorous; these data are provided in Figure 2 for the various samples.

Biomethane production potential (BMP) represents a crucial parameter for the assessment of energy valorization of samples by anaerobic fermentation. BMP is illustrated in Figure 3 in mL of methane produced per g of vs. for the various samples as a function of fermentation time, while total net methane production in L/kg vs. is given in Table 2, excluding the corresponding methane produced by the inoculum, i.e., the blank sample.



Figure 1. Total solids, volatile solids, and fats content in primary treatment samples from four WWTPs.



Figure 2. Leached COD, nitrogen, and phosphorous content in primary treatment samples from four WWTPs.



Figure 3. Biomethane production potential for the various samples collected from the four WWTPs of the study.

Sample	Total Net CH ₄ (L/kg VS)	Standard Deviation
ASC	522.2810	38.9924
AFL	95.0346	59.6485
KSC	207.4496	57.2870
KFL	207.4093	9.4035
LSCC	544.3497	27.5496
LSCF	741.9625	65.2636
LSCST	364.8005	44.7425
LFL	89.9468	14.4040
TSC	255.7938	54.7417
TFL	200.3265	32.0558
TPS	472.7396	48.0594
Ι	179.0224	3.8975

Table 2. Total net methane production in L/kg vs. for each sample.

A crucial parameter in the efforts for the energy utilization of WWTPs residues is the time required to deliver an adequate amount of biogas, in relation to existing anaerobic fermenters of secondary sludge corresponding to sludge retention times of about 30 days. The kinetics of methane production for the various samples are shown in Figure 4, while the calculated maximum specific growth rate μ_{max} and methanogens doubling times are given in Figure 5.









80

60

time, d







Figure 4. Time required for the production of 20, 40, 60, and 80% percentage vol. of total methane for the samples collected from WWTP No 1 (**a**), 2 (**b**), 3 (**c**), 4 (**d**).



Figure 5. Estimated μ_{max} values and average methanogens doubling time as deduced from BMP curves of various samples.

4. Discussion

The aim of the study is the determination of the valorization potential of byproducts produced during the primary treatment of municipal wastewaters by the utilization of their energy content through anaerobic digestion. The BMP potential of the individual byproducts was utilized as an indication of their capacity for production of biogas; this parameter is commonly used for the estimation of the theoretical maximum potential of various wastes [26]. In addition, BMP represents a required preliminary step for the examination of the feasibility of anaerobic digestion of a wide range of wastes and products, and it has been applied in this study to elucidate the assessment of the properties of these unconventional energy sources. Such an approach will in addition reveal potential operation problems during the addition of screenings in sludge digestion, such as inhibition of the anaerobic biota resulting in negligible biogas production. On the other hand, successful results will enhance byproducts utilization, and expected potential benefits include the reduction of side streams amounts conventionally disposed in sanitary landfills, as well as suppression of aeration demands in the following activated sludge process in a wastewater treatment plant. Nevertheless, the amount of these byproducts favors their co-digestion in existing sewage sludge anaerobic digestion units rather than their treatment in individual reactors. Their co-digestion is expected to result in raising the biogas production rate compared to sludge treatment solely, accounting for about 300-400 mL/g vs. [27]. Additional effects on digestate quality should be encountered, as soon as the feasibility of the process will be justified through the estimation of BMP potential. Nevertheless, a study has been reported on the quality of digestate from anaerobically treated screenings which was similar to the corresponding one from sewage sludge, towards its valorization through phosphorous recovery [17]. The overall estimation of potential economic and energy revenues due to the introduction of these side streams in existing treatment plants requires an integrated assessment of all potential benefits expected, including energy earnings from their utilization, reduction of landfilling costs, environmental costs revenues associated with fewer total GHG emissions, etc.

According to the information provided in Table 1, samples were collected from four WWTPs, with a wide range of design capacity, and servicing areas of different human activities. Selected WWTPs represented processes with a wide range of wastewater flowrates, ranging from as low as $8400 \text{ m}^3/\text{d}$ to values exceeding $155,000 \text{ m}^3/\text{d}$. In addition, these units receive wastewaters from septic tanks reflecting, therefore, an additional contribution to screenings production. On the other hand, the composition of screenings was expected to vary due to the different origin of influents.

The lowest values of volatile solids were observed for the screenings collected during delivery of effluents from septic tanks transferred to one treatment plant by trucks and for the sewage screenings and the primary sludge screenings of the same WWTP. It seems that the volatile content of the samples can be attributed to their origin and the treatment stage: wastewater in septic tanks remains for a long time, subjected to anaerobic degradation of organic matter. Therefore, volatile solids in these effluents are expected to be lower than the corresponding solids removed in sewage treatment systems. On the other hand, the low volatile content of the screenings from the treatment plant with the highest capacity might be associated with the long sewage network system of the certain effluents, and their large transfer time from point of source to the treatment plant which might result in less organics content. Nevertheless, the fat content in the samples presented values reaching up to 30% of the total dry content, with great variation between the samples collected even from the same treatment plant. Similar values of volatile solids content have been reported in the literature ranging from 77 up to 95% of dry solids, depending on the screen size and the wastewater origin, with raw samples exhibiting considerably great variations in the moisture and the total solids content [11,17].

In addition to solids content, according to data given in Figure 2, high leached phosphorous and ammonia nitrogen contents were measured for the samples collected from the WWTP receiving effluents from an area with combined human activities, including extended agricultural areas and farming lands. Nevertheless, low leached organic matter values were observed in these samples, while the highest values of COD, up to 1000 mg/L, were measured for the other samples, without, however, an indication of potential trends due to the origin or wastewater characteristics. Limited studies related to the content of leached compounds from screenings have been reported, citing rather high COD values, ranging from 0.8 up to 1.6 g COD/g VS, close to theoretically estimated ones [28], while most studies include the elemental analysis of carbon, nitrogen, and phosphorous in screenings [13]. It must be underlined that biogas production potential is favored under certain ratios of organic matter: nitrogen:phosphorous and the presence of appropriate amounts of both carbon and nutrients sources is required for the efficient operation of anaerobic biocommunities. Although elemental nitrogen and phosphorous concentrations were not measured in this work, the corresponding leached contents of the nutrients were determined, as an indication of their presence in appropriate amounts and their availability towards the efficient operation of the anaerobes.

BMP results are given in Figure 3 as a function of reaction time, while the corresponding total net methane production is provided in Table 2. The methane potential is defined as the maximum produced methane of a specific substrate [29]; however, a methane production curve often presents an initial lag phase, and, therefore, the methane potential measurement for 80 days, as used in the study, provides valuable information about the requirement of a pretreatment step to speed up the whole process. As shown in Table 2, all samples exhibited methane production ranging from low values of around 90 L/kg vs. up to 740 L/kg vs. observed for fine screenings. Surprisingly, floatings from grit chambers had the lowest BMP of all samples from each wastewater treatment plant, although they were expected to deliver high biogas production potential due to their high content in fats (Figure 1) and the corresponding process stage where these samples are collected. As can be seen in Figure 1, fats represent a small fraction of the organic matter, and it is assumed that organic compounds other than fats in these samples were not easily assimilated by the anaerobes towards biogas production. In addition, it has been reported that the complicated composition of floatings containing low density materials such as cellulosic fibers can greatly affect the corresponding energy utilization available for anaerobic digestion [19], while methanogens can convert only a fraction of organics to

biogas ranging from 50 to 60% [10,30]. On the other hand, methane production has been reported to be inhibited due to the formation of long-chain fatty acids during fats anaerobic degradation [31]. Nevertheless, the low BMP potential of floatings from the other two treatment plants is in line with their low content of fats.

Screenings from the plants located in urban areas were similar or slightly higher than the corresponding BMP potential of floatings, while large differences were observed in BMP values of screenings collected from treatment plants receiving effluents from areas with combined activities. The highest methane potential values were measured for the fine and the coarse screening samples from the plant with agro-industrial activities. This may be attributed to various reasons, such as the short distance of the sewage network and the corresponding short time of effluents travelling to the treatment plant, which is allowing less washing and degradation of the compounds, or the presence of potential effluents from agricultural and farming lands in the municipal effluents with a high methane production capacity. Moreover, samples originating from the primary sludge screen presented a rather high methane potential, the highest of the samples obtained from the certain treatment plant. This is a valuable indication of the high energy content of that sludge which is currently disposed together with the secondary anaerobically treated sludge in composting units, with loss of the contained energy content. Efforts were taken to identify potential relations of BMP values shown in Figure 3 to the corresponding chemical composition of the samples in Figures 1 and 2, but no significant trends were observed.

It should be mentioned that substrates obtained from wastes and residues of municipal wastewater treatment plants may have considerable methane potential, depending on their origin and the wastewater treatment stage: anaerobic digestion of FOGs resulted in the production of 271 to 344 and from 325 to 681 mL of methane per g of vs. added, corresponding to samples with volatile solid content up to 30% and 60%, respectively [20]. Similar results regarding methane production capacity were observed by screenings fed to a continuous anaerobic bioreactor ranging from 197–512 mL of CH_4/g vs. with 41.5% total and 29.4% volatile solids content [32]. Nevertheless, wastewaters from livestock production represent more efficient substrates, as they contain higher amounts of total solids and fatty compounds per m³ of raw material than municipal wastewater: The theoretical biomethane potential from fats is estimated to be about 990 mL/g, while that from proteins is about 640 mL/g vs. and from hydrocarbons is about 415 mL/g vs. [33]. Although residues from wastewater treatment plants may have lower biogas potential than wastes of livestock production, the benefits from the utilization of the energy content is profound, considering the current methods of handling, including either disposal in sanitary landfill or incineration in municipal facilities.

Data in Figure 4 indicate the time required to receive a certain volume percentage of the total methane production, i.e., 20, 40, 60, and 80%, for each sample. The wider a column, the longer the time required to produce the corresponding methane volume, while shorter columns and less wide bars represent samples producing methane in short times. Similar observations can be drawn from the results shown in Figure 5, where μ_{max} and methanogens doubling time are reverse parameters, the latter corresponding to the time required for doubling the cells of the anaerobic micro-organisms. The lower the methanogens doubling time, the higher the maximum growth rate μ_{max} .

As shown in Figure 4, more than 50% of gas volume is produced at short reaction times, while production of up to 80% of total gas required longer time, depending on the particular sample. Samples collected from the WWTPs with low design capacities required rather long times to reach up to 80% volume of methane, exceeding 70 days, while the shortest times were observed for the residues from the WWTP in the agricultural area, varying between 15 and 30 days (although floatings may require as long as 70 days). Moreover, 60% of total gas volume was already produced in a period of less than 10 days for these samples, and a similar behavior was observed for the corresponding samples from the high-capacity treatment plant. However, slow kinetics were obtained for the residues from the other plants.

These results are in line with the corresponding data on average methanogens doubling time given in Figure 5, where short times were calculated for the high design capacity unit, ranging from 0.8 to 1.13 days, followed by the agricultural area treatment plant with slightly higher methanogens doubling times from 1.34 to 1.53 days. The highest methanogens doubling times were measured for the low-capacity plant with semiindustrial characteristics, reaching up to 2.38 days, almost three times higher than the lowest value. The above observations are becoming important for the energy recovery of these substrates during co-digestion with secondary sludge in these units. The low methanogens doubling time is associated with short retention times for the treatment of these substrates, which could strongly affect the design of anaerobic reactors in a new WWTP by requiring smaller reactor volumes. In addition, the range of the μ_{max} values and consequently of the doubling time is within that reported for a wide range of samples [22,34], and, therefore, the methanogens' growth rate was not differentiated due to the particular substrates used in this study.

Taking into account the BMP results presented in Table 2 and Figure 3 and the corresponding kinetic data in Figures 4 and 5, the beneficial role of the corresponding samples as potential energy sources could be identified. In general, all samples represent promising materials for energy recovery and the enhancement of energy production processes in the corresponding wastewater treatment plants. Nevertheless, efforts to identify the samples with the highest potential for biogas production should take into consideration both biogas cumulative volume and biota doubling time: under that framework, the fine and coarse screenings from the wastewater treatment plant receiving effluents from areas with mixed activities represent the best candidates for energy recovery through anaerobic digestion, followed by the primary sedimentation sludge screenings and the coarse screenings of the high flowrate municipal treatment plant. Both plants have already under operation an anaerobic digestion plant of secondary sludge, and the proposed energy utilization of their primary residues might represent an easy-to-apply process aiming at their energy self-efficiency.

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