EXPERIMENTAL ANALYSIS OF OBTAINING BIOGAS FROM WASTEWATER TREATMENT PLANT SLUDGE

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This article presents the results regarding experimental research activities of obtaining biogas from wastewater treatment plants sludge. In this research, three experiments have been done, in which the temperature varied between 34-50 °C to analyze both the mesophilic and thermophilic regime. The scope was obtaining methane and hydrogen from the sludge collected from WWTP on the Romanian coast. The maximum methane value was 2.8%, for the temperature of 35°C. The hydrogen was obtained in high quantities only in the thermophilic regime at temperatures close to 50°C.

Keywords: sludge, wastewater treatment plant, anaerobic fermentation process, biogas

1. Introduction

A sewage treatment plant is a specialized facility that treats and cleans wastewater from households, industry, or other sources, to release it back into the environment in a less harmful state [1]. This uses various chemical, biological, and physical processes to remove pollutants, bacteria, and other impurities from wastewater so that they do not affect the water quality of the natural ecosystems into which they are discharged [2]. Their role is crucial in keeping public health and environmental quality. Wastewater treatment plant sludge is a solid or semi-solid waste resulting from the wastewater treatment process. This can include various elements such as organic and inorganic substances, bacteria, sediment particles, and other waste materials that are removed from wastewater during the treatment

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process [3, 4]. Sludge may also contain nitrogen, phosphorus, and other nutrients, depending on the origin and composition of the treated wastewater [5].

According to 2022 research, the average annual sewage sludge production across different economic sectors was generally 9, 0.36, 2.2, 17.8, and 17.5 million metric tons in Australia, Japan, Europe, China, and the USA, respectively. Nevertheless, the amount of sewage sludge produced globally is never-ending. For example, the amount of sewage sludge produced in the European Union (UE) increased by more than 38% between 2005 and 2020 [3]. Sludge must be treated as recyclable waste and disposed of from the environment according to applicable laws. Sludge management, recycling, treatment, and disposal are essential to sustainable waste management, especially in wastewater treatment plants and industries**.** These steps contribute to the environmental protection and the recovery of sludge resources, thus reducing the impact on landfills and promoting the circular economy. [6].

Sludge may be subjected to various treatments, including dehydration, incineration, composting, or anaerobic digestion. These processes have been carried out to reduce the volume and remove bacteria and harmful substances. After treatment, the sludge can be used in agriculture for fertilization or biogas production. The method used must be made according to the cost of the process, the origin of the sludge, and the impact it could have on the environment [7].

Among all the mentioned solutions, we can say that anaerobic digestion is one of the most frequently used in the wastewater treatment plants. Following the research done by A.S. Giwa et al. [8] anaerobic digestion can produce biogas by converting about 30÷60% of organic matter, though it can take a longer start-up period of 10÷30 days [9]. This type of digestion is conducted by anaerobic microorganisms, which break down organic substances in oxygen-free conditions. Also, the anaerobic digestion process consists of four main stages: hydrolysis, acidogenesis, acetogenesis, and methanogenesis. The stages of the anaerobic digestion process are presented in Fig.1.

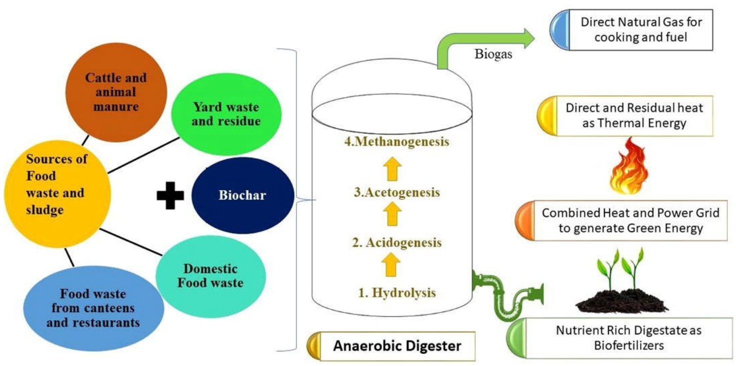

Fig 1. The stages of the anaerobic digestion process [12]

The critical step in this process is methanogenesis, which is strongly influenced by several factors: feedstock composition, organic matter ration (BOD_5), temperature and pH. The excessive presence of oxygen in the methanogenesis step can lead to the inhibition of the whole process [10], [11].

Anaerobic digestion allows the production of flammable gas called biogas. This gas contributes to the production of renewable energy, which can meet the needs of the plant [12]. The successful operation of an anaerobic reactor depends on maintaining the environmental factors close to the comfort range of the microorganisms. For example, temperature variations or changes in the substrates or the substrate concentration can lead to shutdown of biogas production. Important parameters in this process are temperature, pH, alkalinity, redox potential, organic loading rate, solid concentration, and nutrients. However not all these parameters can be measured in real time, and the cost of the final solution should ensure acceptable market access.

The temperature is the main factor influencing the biogas composition and quantity [13,14]. Depending on it, the concentration of anaerobic microorganisms can vary between 10÷60% [15]. To achieve the anaerobic digestion process, the temperature must be kept constant between 30÷37ºC with maximum variations of ±2 °C corresponding to the thermophilic environment [16]. It is also necessary to consider the continuous mixing of the sludge with the help of mixers [17,18].

In the anaerobic digestion experiment conducted by Aline Tathyana et al. [19] in Brazil, 5.4% CH_4, 1.9% CO_2, and 11 ppm H_2S were obtained on the sixth day of analysis. The total biogas production was 0.050 kg biogas/kg organic matter, resulting in 89.48 kW. S. Kalloum [20] obtained, in his analysis of the sludge, 280.31 NmL biogas with a yield of 30 NmL of biogas/mg of COD removed. According to M. B. Miranzadeh et al. [21] under optimal conditions, a specific amount of methane of 0.15÷0.34 L CH_4/gVS was produced from the primary sludge digestion.

On the other hand, Malik and Bharti [22] obtained, during summer, a biogas volume of 84952.34 m^3, while during winter, a biogas volume of 76252.81 m^3 was recorded. An important parameter that contributed to these biogas volumes was the pH, for which a value of 7.06 was identified in summer and a value of 6.89 in winter. The pH values were optimal for the development of methanogenic bacteria (Methanobacterium, Methanoccus, and Methanosarcino barkeri) which contributed to the methane production. In the summer, the experiment was carried out under thermophilic conditions (25÷45°C), while in the winter, it was carried out under mesophilic conditions (25÷35°C). In addition, other physical parameters such as total solids content, volatile solids (VS) content, and alkalinity showed much higher values in summer than in winter.

Another parameter that influences biogas production is the mixture of sewage sludge with other types of sludge. Luostarinen et al. studied the feasibility of co-digestion of sewage sludge and grease trap sludge from a meat processing plant at a temperature of 35°C. The grease trap sludge had a high methane production potential (918 m^3), but the production started slowly. Thus, by mixing with sewage sludge, methane production started quickly, and the potential increased with the addition of trap sludge (46% of the feed). The retention time was 16 days, and the organic load was 3.46 $kgVS/m^3*d$. When more than 46% trap sludge is added, the process can be inhibited [23].

In this paper, the results of experimental research in the laboratory for the anaerobic digestion process of urban wastewater treatment plant sludge were presented. The relationship between temperature, the amount of methane, and hydrogen was analyzed. For this study, three experiments were carried out in which the amount of organic matter ranged between 220÷285 $mgBOD_5/L$ and the temperature ranged between 34÷50°C.

2. Materials and methods

2.1. Experimental device

The experiment on anaerobic digestion was carried out at POLITEHNICA University of Bucharest - Laboratory for Analysis, Control, and Remediation of Contaminated Sites (The Center for Advanced Research on New Materials, Products, and Innovative Processes). Fig. 2 shows the laboratory installation, namely the bioreactor BIO-4/EDF-5.4, which has a capacity of 5.4 L.

The entire experimental set-up consisted of an autoclavable vessel, equipped with a top cover with inputs/outputs (ports), magnetically driven stirrer shaft and BIO-4 bioprocess controller, equipped with a peristaltic pump system, control unit, rotameters and manometers. The obtained gas was introduced into the gas analysis system through a hose connected to one of the openings in the lid of the autoclavable vessel. The installation was connected to the water supply as water was used to keep the constant temperature of the process [24]. The control parameters were temperature, pH, foam that formed, level inside the vessel, and mixing system. Before starting the process, the pH sensor and the peristaltic pumps were calibrated.

The next step was to introduce the sludge into the glass vessel, make the connections, set the optimal temperature for the process, and set the desired velocity. The content inside the glass vessel was kept at the desired temperature by means of a mantle found in the lower lid of the vessel. The sample was stirred regularly. The amount of biogas obtained was determined by reading the values displayed on the screen of the process biocontroller. The pH of sludge was measured using a Seven Excellence pH-meter model pH-meter S400, and the rest of the parameters TSS, COD, and BOD_5 were measured in the accredited laboratory from the wastewater treatment plant.

Fig. 2. Experimental set-up
1 – axial agitator drive system, 2 – upper lid with inputs/outputs, 3 – autoclavable vessel, 4 – condenser, 5 - gas analysis unit, 6 – peristaltic pumps, 7 – biocontroller, 8 – rotameters, 9 - manometers

2.2. Experimental method

In this study, the sludge samples were collected from a sewage treatment plant on the Romanian coast (thickened sludge) and brought to the laboratory to analyze their potential for biogas production. The samples were taken in 5 L bottles and transported in optimal and safe conditions. The interval between sampling and introduction into the installation was approximately 4 hours. By conducting the anaerobic fermentation process, the efficiency of the anaerobic fermentation process was evaluated, and the biogas production potential was determined according to the sludge properties.

Experimental research was conducted for 3 sludge samples. The sludge was introduced into the installation using a graduated glass flask. In the first experiment, 2.5 L of sludge was introduced, in the second, 4.5 L of sludge was used, and in the third experiment, 2 L of sludge was used.

The sludge parameters (TSS, COD, BOD_5) were measured in the wastewater treatment plant laboratory according to the current standards. The other parameters pH, temperature, and velocity of the agitator shaft were measured with the sensors from the experimental installation. The values of these parameters were presented in Table 1. The time parameter in the table represents the number of days the experiment was carried out.

Table 1

Characteristics of the sludge from the wastewater treatment plant

Parameters \ Concentration	Experiment 1	Experiment 2	Experiment 3

pH	7,4	6,64	6,8
TSS [mg/L]	327	348	307
COD [mg/L]	447	465	446
BOD_5 [mg/L]	233	285	220
t [ºC]	35-55	35	35
Velocity of agitator axle [rpm]	100	50	50
Time[d]	21	25	23

The duration of the experiments depended on the amount of biogas released by fermentation. Once no more gas was produced, the experiment was stopped.

3. Results and discussions

The methane results obtained for the three experiments were presented comparatively in Table 2. The amount of BOD_5 greatly influences the obtaining of CH_4. The higher the amount, the more CH_4 was obtained. In the case of obtaining H_2, the optimal fermentation temperature was 50°C. At a BOD_5 of 230 mg/L, hydrogen started to occur on day 3, and the maximum value was reached on day 6.

In the first experiment, temperatures were set, and significant amounts of hydrogen were obtained compared to the amount of sludge introduced into the installation. The highest amount of hydrogen was obtained on day 4, for a temperature of 49.8°C, and the lowest amount of hydrogen was obtained on day 3, for a temperature of 42°C. H_2S and CO_2 were also obtained during the experiment, but the focus was on the amount of hydrogen because it can be used as an energy source.

Table 2

Interpretation of the results in the case of CH_4

BOD_5 [mg/L]	220	230	285
t [°C] optimal	35	50	35
The day when CH_4 starts appearing	Day 2 (0,2% CH_4)	Day 3 (0,1% CH_4)	Day 8 (0,1% CH_4)
The day when CH_4 was maximum	Day 8 (0,9% CH_4)	Day 6 (1,8% CH_4)	Day 12 (2,8% CH_4)

In the second part of the research, experiment II, temperatures around 35°C were maintained and reasonable amounts of methane were obtained in relation to the amount of sludge introduced into the plant. Compared to the first experiment, the amount of organic matter was 62 mg/L higher, but not enough to obtain significant amounts of methane. The highest amount of methane was obtained on day 12, a methane amount of 2.8% at a temperature of 35.1°C. In the 25 days of anaerobic digestion analysis, methane started to appear on day 8. During experiment II, no significant values were recorded for H_2 and H_2S, which proves that only at higher temperatures significant amounts of these gases can be obtained.

In experiment III, a sludge sample collected from the same wastewater treatment plant was analyzed, but the amount of BOD_5 was much lower than in the previous cases. In the case of obtaining CH_4, the optimal fermentation temperature was 35°C. With the increase of BOD_5, the day of obtaining the maximum amount of methane also increases as follows: at a BOD_5 of 220 mg/L, the maximum amount was obtained on day 8; at a BOD_5 of 285 mg/L, the maximum amount was obtained on day 12.

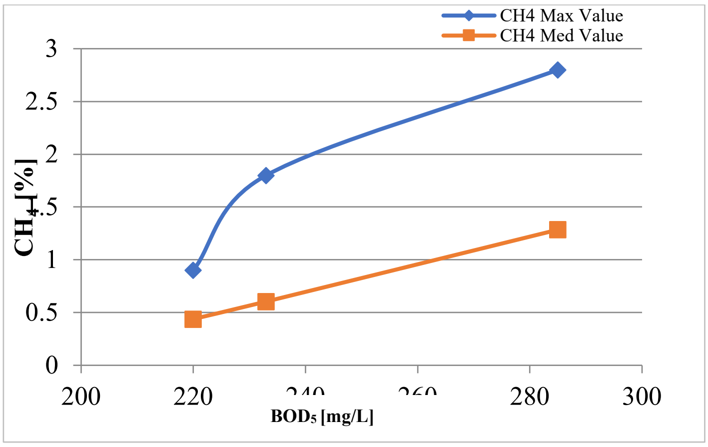

Fig. 3. Maximum and average CH_4 values as a function of BOD_5

Fig. 3 shows the variation of the maximum and average values of the methane concentrations obtained for the 3 experiments. The results show that the amount of biogas produced was directly proportional to the amount of organic matter available in the sludge introduced. Also, the maximum values are 2.5÷3 times higher than the average values.

In this experiment, it was proved that the percentage of organic matter can influence the efficiency of the anaerobic digestion process. The results obtained in this study provide essential information regarding the relationship between temperature, the amount of methane, and hydrogen obtained in the anaerobic digestion process.

4. Conclusions

The quality and quantity of available organic matter in the sludge are critical factors for the anaerobic digestion process. In this article, experimental research has shown the relationship between methane and hydrogen production and the sewage sludge temperature the production of methane and hydrogen, and the temperature of sewage sludge.

The experimental tests carried out within this paper reveal the following conclusions from the first experiment: it was concluded that hydrogen was obtained in higher amounts only in the thermophilic regime, at temperatures close to 50°C. The highest amount of hydrogen was obtained at 1013 ppm at 49.8°C. At a BOD_5 of 230 mg/L hydrogen started to appear on day 3, and the maximum value was reached on day 6. In the second part of the research, the highest amount of methane was 2.8% at a temperature of 35.1°C. In the third experiment, the maximum amount of methane was 0.9% at a temperature of 35.5°C. In the case of obtaining CH_4, the optimal fermentation temperature was 35°C. With the increase of BOD_5, the day of obtaining the maximum amount of methane also increases as follows: at a BOD_5 of 220 mg/L, the maximum amount is obtained on day 8; at a BOD_5 of 285 mg/L, the maximum amount is obtained on day 12.

Given the extended duration of an experiment, the research will continue with the realization of a mathematical model that will help to obtain the result for more cases in less time.

The use of biogas obtained from sewage sludge has been an efficient way to manage waste and produce renewable energy. The obtained biogas can also be used to produce electricity or heat in cogeneration systems. Energy recovery of biogas from wastewater treatment plant sludge is a sustainable strategy that brings both environmental and economic benefits, contributing to the transition towards a greener and more energy-efficient society.

In conclusion, the research proved that significant amounts of BOD_5 are required to have large amounts of CH_4 and H_2, respectively. The amount of organic matter influences the biogas production.

R E F E R E N C E S

[1]. *N. Parashar, S. Hait*, Abundance, characterization, and removal of microplastics in different technology-based sewage treatment plants discharging into the middle stretch of the Ganga River, India, Science of The Total Environment, **Vol. 905**, December 2023, 167099

[2]. *A. Talukdar, P. Kundu, S. Bhattacharya and N. Dutta,* Microplastic contamination in wastewater: Sources, distribution, detection and remediation through physical and chemical-biological methods, Science of The Total Environment, **Vol. 916**, March 2024, 170254

[3]. *J. Michalska, J. Turek-Szytow, A. Dudło and J. Surmacz-Górska,* Characterization of humic substances recovered from the sewage sludge and validity of their removal from this waste, EFB Bioeconomy Journal, **Vol. 2**, November 2022, 100026

[4]. *T. Bauer, L. E. Burgman, L. Andreas and A. Lagerkvist,* Effects of the different implementation of legislation relating to sewage sludge disposal in the EU, Detritus Journal, **Vol. 10**, June 2020

[5]. *J. Cui, H. Wang, D. Hantoko, X. Wen, E. Kanchanatip and M. Yan*, Nitrogen and phosphorus recovery from sludge treatment by supercritical water gasification coupled with struvite crystallization, Journal of Water Process Engineering, **Vol. 55**, October 2023, 104070

[6]. *D. Mara, S. Cairncross,* Health guidelines for the use of wastewater in agriculture and aquaculture. Report of a WHO Scientific Group, Environmental Science, Agricultural and Food Sciences, February 1992

[7]. *D. Zmirou, P. Thoumelin,* L'incinération des déchets et la santé publique. bilan des

[8]. *A. S. Giwa, N.J. Maurice, A. L., X. Liu, Y. Yunlong, and Z. Hong,* Advances in sewage sludge application and treatment: Process integration of plasma pyrolysis and anaerobic digestion with the resource recovery, Heliyon, **Vol. 9**, September 2023, 19765

[9]. *J. Wan, L. Zhang, B. Jia, B. Yang, Z. Luo, J. Yang, P. Boguta and X. Su,* Effects of enzymes on organic matter conversion in anaerobic fermentation of sludge to produce volatile fatty acids, Bioresource Technology, **Vol. 366**, December 2022, 128227

[10]. *Mohamad Y. Mustafaa, Rajnish K. Calayb, E. Roman,* Biogas from Organic Waste - A Case Study, Procedia Engineering **Vol. 146**, 2016, pp. 310-317

[11]. Biogas: Practical Guide (2010).

[12]. *S.R. Deena, A.S. Vickram, S. Manikandan, R. Subbaiya, N. Karmegam, B. Ravindran, S. W. Chang, M. K. Awasthi*, Enhanced biogas production from food waste and activated sludge using advanced techniques – A review, Bioresource Technology, **Vol.**355, July 2022, 127234.

[13]. *W. Suksong, K. Promnuan, J. Seengenyoung, S.O-Thong,* Anaerobic Co-Digestion of Palm Oil Mill Waste Residues with Sewage Sludge for Biogas Production, Energy Procedia, **Vol. 138**, October 2017, pp. 789-794

[14] *I. Junior, R. Almeida, M. Cammarota*, A review of sludge pretreatment methods and co-digestion to boost biogas production and energy self-sufficiency in wastewater treatment plants, J. Water. Proc. Eng., **Vol. 40** (2021), 101857

[15] *A. Leitão, D. Moni, C. Maria*, Anaerobic co-digestion of food waste with sewage sludge from wastewater treatment plant of Sequele, Luanda-Angola. Env. Chall., **Vol. 9,** (2022)

[16] *Y. Hu, T. Kobayashi, W. Qi, H. Oshibe and K. Xu*, Effect of temperature and organic loading rate on siphon-driven self-agitated anaerobic digestion performance for food waste treatment, Waste Management, **Vol. 74**, pp. 150-157, April 2018.

[17] *E. Ibrahimi, I.E. Khay, A. Maakoul, M. Bakhouya*, Techno-economic and environmental assessment of anaerobic co-digestion plants under different energy scenarios: a case study in Morocco, Energ. Conv. Mnag., **Vol. 245** (2021), 114553

[18] *C. Zhang, H. Su, J. Baeyens, and T. Tan*, Reviewing the anaerobic digestion of food waste for biogas production, Renewable and Sustainable Energy Reviews, **Vol. 38**, pp. 383-392, 2014.

[19]. *A. T. A. Felca, R. M. Barros, G.L.T. Filho, I.F.S. Santos, E. M. Ribeiro*, Analysis of biogas produced by the anaerobic digestion of sludge generated at wastewater treatment plants in the South of Minas Gerais, Brazil as a potential energy source, Sustainable Cities and Society, **Vol. 41**, August 2018, pp.139-153.

[20]. *S. Kalloum H. Bouabdessalem, A. Touzi, A. Iddou, M.S. Ouali,* Biogas production from the sludge of the municipal wastewater treatment plant of Adrar city (southwest of Algeria), Biomass and Bioenergy, **Vol. 35**, July 2011, pp. 2554-2560.

[21] *M. B. Miranzadeh, M. Jafarsalehi, J. Akram, M. Ebrahimi, A. Mazaheri, M. Mashayekh*, Boosting biogas production in the wastewater treatment plants: A narrative review on co-digestion of sewage sludge with internal and external organic waste, Bioresource Technology Reports, **Vol. 26**, 2024, 101856.

[22] *D. S. Malik, U. Bharti*, Biogas production from Sludge of Sewage Treatment Plant at Haridwar (Uttarakhand), Asian Journal of Experimental Sciences, **Vol. 23**, 2009, pp. 95-98.

[23] *S. Luostarinen, S. Luste, M. Sillanpaa*, Increased biogas production at wastewater treatment plants through co-digestion of sewage sludge with grease trap sludge from a meat processing plant, Bioresource Technology, **Vol. 100**, (2009), pp. 79–85.

[24]. User manual, Laboratory bioreactor EDF-5.41, www.bioreactors.net