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Short Communication

Improved methane production from ultrasonically-pretreated secondary sedimentation tank sludge and new model proposal: Time series (ARIMA)

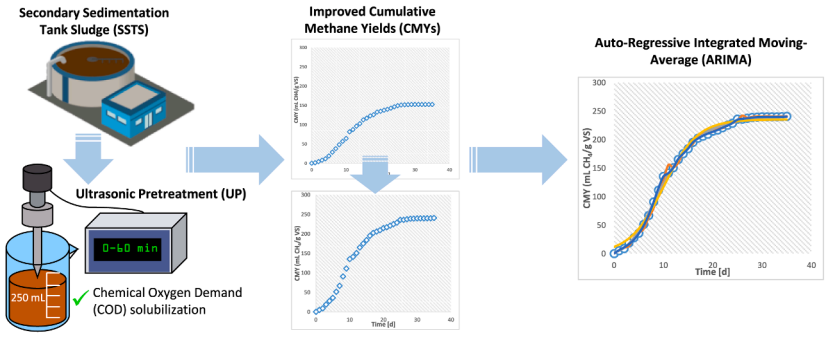
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HIGHLIGHTS

- The time series (ARIMA) is proposed as a novel model for predicting methane yield.
- The secondary sedimentation tank sludge (SSTS) was pretreated with ultrasounds.
- The highest cumulative methane yield was achieved after pretreating SSTS for 30 min.

GRAPHICAL ABSTRACT



ARTICLE INFO

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ABSTRACT

Secondary sedimentation tank sludge (SSTS) is a severe type of waste that needs to be treated to prevent human health risks. Anaerobic digestion (AD) alone can be ineffective for treating SSTS due to low substrate biodegradability. This study proposes the use of ultrasonic pretreatment (UP) on SSTS for 0 – 60 min. UP effect on SSTS was analyzed through the actual specific energy input, nominal energy input, and soluble chemical oxygen demand. The highest cumulative methane yield (CMY) of 241.56 mL/g VS obtained during the AD of the pretreated substrate (i.e., 30 min) was 58.4% higher than that of untreated SSTS. The resulting CMY curves were subsequently simulated via the modified Logistic and Gompertz, Artificial Neural Network, Auto-Regressive Integrated Moving-Average (ARIMA) models. As a novel proposal for predicting CMYs, the ARIMA model is recommended due to its simplicity and better performance than sigmoidal models (R^2 = 0.9975 – 0.9988).

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1. Introduction

The yearly production of secondary sedimentation tank sludge (SSTS) is massive in municipal wastewater treatment plants (WWTPs). For instance, in Europe, the annual output of WWTP sludge (i.e., 2007 – 2018) ranges between 7 and 8 M tonnes (Anderson et al., 2021). Taking into account that the cost for SSTS disposal is almost half the operating cost for a WWTP (Zhen et al., 2017) and that the SSTS can also contain several hazardous compounds such as heavy metals, organic pollutants, and pathogens (Li & Zhang, 2010), this organic waste has to be managed appropriately.

Among the treatments employed for treating SSTS before its disposal or reuse, anaerobic digestion (AD) is beneficial by providing SSTS stabilization and bioenergy recovery from the generated methane (Zhen et al., 2017). The hydrolysis stage is assumed to be the limiting step for the AD of SSTS due to the organic matter recalcitrance caused by a complex floc structure (e.g., extracellular polymeric substances). This would lead to the design of larger bioreactors due to the increased hydrolysis time and reduced methane yields (Brémond et al., 2018). Therefore, an adequate pretreatment method should be performed on SSTS to enhance its biodegradability.

Among the pretreatment methods (i.e., physical–chemical, biological, thermal), ultrasonic pretreatment (UP) has proven effective in disintegrating complex organic structures such as lignocellulosic materials, manure and sludge (Oliva et al., 2022a). Ultrasounds generate hydro-mechanical shear forces due to cavitation bubbles (Khanal et al., 2007) that can disrupt the SSTS biomolecules by decreasing particle dimensions, improving organic substance dissolution, and stimulating microbial and enzymatic activity during AD. Thus, the UP can result in a decrease in SSTS volume through an improvement of sludge dewaterability (Li et al., 2018).

Substrate pretreatments are assessed mostly using biochemical methane potential (BMP) tests at the lab scale. A BMP test involves a batch approach to achieve the maximum biogas yield from a raw or pretreated substrate by operating a specific inoculum, diluted background, and a prolonged incubation period. The cumulative methane production curves resulting from a BMP test are generally modeled via a first-order-kinetic equation, but a more sophisticated modeling procedure is often needed. For example, sigmoidal kinetic models such as the Modified Logistic and Gompertz Models (MLM and MGM) have been extensively employed for methane prediction (Şenol, 2021). However, a high mole of experimental data is required to correctly implement these models in a full-scale biogas plant, mainly including the properties and kinetics of the used substrates and the produced biogas. Therefore, for researchers, traditional modeling techniques are not sustainable for simulating complex and nonlinear behavior, and easier-to-apply modeling techniques are needed to understand the AD process better.

A more complex approach to predicting methane generation during AD uses data-driven models, such as Artificial Neural Networks (ANNs), which are capable of solving complex problems due to their capacity for understanding, association, adaption, and computation (Cruz et al., 2022). Although the significance of ANNs for simulating methane yield (Fig. S1), the literature for evaluating the effect of UP methods on organic substrates is limited, and no reports are available on the principle of time series analysis applied to raw and pretreated SSTS. The Auto-Regressive Integrated Moving-Average (ARIMA) modeling is one of the most reliable methods for time series analysis. This method is developed on a linear regression model, integrating the benefits of autoregressive and moving average models, thus predicting non-linear and complex data (e.g., methane production) due to linearization (Khashei & Bijari, 2011).

In this work, the effect of the UP method applied for a pretreatment time ranging between 0 and 60 min was investigated on SSTS to enhance its soluble chemical oxygen demand (SCOD) as a function of the sonolysis power. Afterwards, batch BMP assays were performed under different operating conditions (Table 1) to assess the cumulative methane yield (CMY) during the AD of raw and pretreated SSTS. Finally, the CMYs were predicted through different simulating models, i.e., MLM, MGM, ANNs, and, for the first time, the ARIMA, for all the investigated reactors.

2. Materials and method

2.1. Substrate and inoculum

For the substrate and inoculum, see Supplementary Materials (see Section S1.). The physical–chemical characteristics are summarized in Table S1.

2.2. Ultrasonic pretreatments

The UP was directly applied to 100 mL of raw SSTS with a volatile solid (VS) content of 3.19 g in a 200 mL becher using an HD 2200 Sonopuls ultrasonic homogenizer (Bandelin, Germany) equipped with a VS 70 T probe since this system was found to be particularly effective for pretreating lignocellulosic materials (e.g., hazelnut shells) aiming at improving methane production during AD (Bianco et al., 2022). UP was executed for 0 – 60 min with a frequency and an ultrasonic power of 20 kHz and 200 W (Şenol, 2021), respectively. For the calculation of the actual (Eq. (S1)) and nominal (Eq. (S2)) specific energy (SE_A and SE_N, kJ/kg TS, respectively), see Supplementary Materials (see Section S2.). The experimental conditions carried out for the UP are summarized in Table 1.

2.3. Biochemical methane potential tests

The BMP tests were prepared in 500 mL glass reactors with 169 mL of inoculum (VS = 3.19 g) and 100 mL of either raw or pretreated SSTS to reach an inoculum-to-substrate ratio of 1.0 on VS base. Afterward, distilled water was added to achieve a working volume of 400 mL,

Table 1
Exchange parameters, cumulative methane yields (CMYs) and energy balance (ΔE) of pretreated and untreated conditions (P = 200 W, 1 W/mL, V = 200 mL, TS = 4.67%).

Ultrasonic density	Reactors	Sonication times (min)	SE_N (kJ/kg TS)	SE_A (kJ/kg TS)	SE_A/SE_N	ΔSCOD (%)	CMYs (mL/g VS)	ΔE (kJ/kg TS)
1 W/mL	R0	0	0	0	0	0	152.50	–
	R1	5	6,424	4,277	0.666	30.1	181.90	−3243
	R2	10	12,848	7,802	0.607	47.4	202.34	−9416
	R3	15	19,272	10,820	0.561	62.5	226.40	−15,544
	R4	30	38,544	13,933	0.361	68.7	241.56	−34,630
	R5	60	77,088	16,423	0.213	69.4	240.70	−73,184

Note. R0: utreated substrate, R1: 5 min ultrasonic pretreated substrate, R2: 10 min ultrasonic pretreated substrate, R3: 15 min ultrasonic pretreated substrate, R4: 30 min ultrasonic pretreated substrate, R5: 60 min ultrasonic pretreated substrate, SE_N: Nominal specific energy and SE_A: actual specific energy (calculated by the calorific method); ΔSCOD: incremental SCOD. For the ΔE calculations, the energy obtained from methane (kJ/kg TS) was subtracted to SE_N, considering a heat recovery of 85% by heat exchangers from thermal energy generated by sonication.

obtaining a total solid (TS) amount below 3%, and the reactor headspace (i.e., 100 mL) was finally flushed with nitrogen gas. The reactors were operated under manual shaking (i.e., twice a day) at 37 ± 2 °C until the daily methane production was lower than 1% of the CMY (i.e., 35 d) (Oliva et al., 2022c). Each experimental condition was performed in triplicate. A blank test (i.e., inoculum + distilled water) was also conducted to subtract the CMY aliquot obtained with the inoculum from that produced with either the untreated or pretreated SSTS.

2.4. Analytical methods

TS and VS in both the raw SSTS and inoculum were determined in a lab oven (i.e., at 105 °C for 24 h) and a muffle furnace (i.e., 550 °C for 2 h), respectively (Oliva et al., 2022b). The SCOD of the inoculum, and either untreated or pretreated SSTS, was obtained through the closed reflux titrimetric method (Şenol, 2021). pH was acquired from the inoculum and SSTS through a 510 CyberScan pH meter (Ayer Rajah Crescent, Singapore). The biogas was taken from the anaerobic reactors with a gas collection bag (i.e., 10 – 500 mL), and volumetric biogas production was quantified via the liquid displacement method (Oliva et al., 2022a). Then, the methane content was measured via a GC–8A gas chromatograph (Shimadzu, Japan) (Şenol et al., 2020).

2.5. Data analysis

The CMYs retrieved from untreated and pretreated SSTS were fitted with MGM and MLM, as shown in Table S2 (see Supplementary Materials) (Oliva et al., 2022c). The ANN and ARIMA models (Fig. S1 and S2, respectively), here used to predict CMYs in R0 – R5, the Regression Analysis, and the One-way ANOVA test, are described in detail in the Supplementary Materials accompanying the manuscript (See Sections S3.1 – S3.3). The energy balance (ΔE) assessment was performed according to Bianco et al. (2022).

3. Results and discussion

3.1. Effect of ultrasound pretreatment on the secondary sedimentation tank sludge

SE_N, SE_A, SE_A-to-SE_N ratios (SE_A/SE_N), incremental SCOD (ΔSCOD, %) values and final methane yields were analyzed to evaluate the effect of SSTS on hydrolysis efficiencies after UP at different time intervals (0 – 60 min, Table 1). The SE_A/SE_N (Table 1) reflected the efficiency of the applied UPs (Alagöz et al., 2018) and varied between 0.666 and 0.213 (Table 1) by increasing of sonication time. This can be attributed to a linear and non-linear increase of SE_N and SE_A values when UP time was raised from 5 to 60 min, respectively, since SE_A was retrieved from the calorimetric method (Şenol, 2021). The increase of the ΔSCOD values here achieved (Table 1) indicates the solubilization of organic substances in the liquid phase (Yuan et al., 2019) and could be generally related to the increase of SE_N (Oliva et al., 2022c). The highest ΔSCOD values of 68.7 and 69.4% (Table 1) occurred after 30 and 60 min of sonication, respectively, and showed significant differences ($p < 0.05$) compared to the other UP conditions. SSTS biomolecules were broken down, likely due to the cavitation bubbles generating shear forces and the subsequent oxidative stress during the UP (Khanal et al., 2007). The increase of the SCOD value here obtained after 60 min of sonication (Table 1) was similar to the findings previously shown in the literature (i.e., 66.1% after 60 min of sonication) (Şenol, 2021). Reflocculation phenomena can support the fact that the ΔSCOD was similar ($p > 0.05$) when the sonication time was increased from 30 min to 60 min (Table 1) (Pilli et al., 2011). Therefore, in this study, the sonication time suitable for optimizing the SCOD was 30 min. Taking into account a lower sonication time (i.e., 10 min) at 1 W/mL, the ΔSCOD value was only 47.4% (Table 1). However, Joshi & Gogate (2019) reported that the SCOD increased of 61.5% due to UP (i.e., at 0.4 W/mL for 10 min) applied to food wastes containing 7% TS. The discrepancy with this study can be supported by different aspects such as sludge type and properties, TS content, PA value, frequency, ultrasonic density, and temperature (Şenol, 2021).

3.2. Methane yields of raw and pretreated secondary sedimentation tank sludge

The effect of UPs on CMY was investigated by pretreating SSTS at different sonication times (Table 1) under mesophilic conditions (Fig. 1). The CMY of the untreated SSTS in R0 (i.e., about 153 mL/g VS, Fig. 1) was lower compared to the pretreated SSTS in R1-R5 (i.e., about 182 – 241 mL/g VS, Fig. 1) likely due to sonication assisting in floc disintegration, cell lysis and molecule disruption (Table 1) (Gonzalez-Fernandez et al., 2015). The literature has reported that at least 5000 kJ/kg TS of SE_N should be applied to disintegrate and dissolve an organic substrate with UPs (Feng et al., 2009). In this study, SE_N values (Table 1) were above those recommended in the literature, varying between 6424 and 77,088 kJ/kg TS. Accordingly, the methane production (Fig. 1) was improved up to 1.6-fold with the increase of SE_N input up to 38,544 kJ/kg TS compared to R0. This result is corroborated by previous studies reporting an improvement of methane yields comprised between 4.4 and 28.3% by ultrasonically pretreating sludge (Akgul et al., 2017; Ruiz-Hernando et al., 2014). However, there was no significant difference ($p > 0.05$) in CMYs by further increasing the SE_N input up to 77,088 kJ/kg TS (Table 1). This indicates that SE_N values in UPs can enhance the methane yield up to a certain point (e.g., 30,000 kJ/kg TS) by following the ΔSCOD (Table 1), as also shown by (Şenol, 2021). Similarly, Bougrier et al. (2005) showed that the biogas production of waste-activated sludge could be gradually improved by increasing SE_N input to 6950 kJ/kg TS, but no significant changes can be obtained by pushing up to 14,547 kJ/kg TS. The primary concerns about UP are cost and energy aspects (Khanal et al., 2007). The energy required for the UP could not be enough to justify the methane improvement (Table 1). This is likely due to overrated SENs based on lab-scale conditions with not energetically optimized UP devices (Bianco et al., 2022).

3.3. Modeling results

3.3.1. Sigmoidal modeling results

The CMYs of experimental reactors obtained without pretreatment and depending on different sonication times were simulated with MGM and MLM, and the kinetic constants of the models are shown in Table S2. In addition, the CMYs retrieved from the MGM and MLM are plotted in Fig. 1, along with other results. By comparing the CMY and PMY values obtained with MGM and MLM via R^2, SSE, and RMSE performance criteria, MGM provided better fitting parameters than MLM. This is due to an R^2 closer to 1 and lower RMSE and SSE values in MGM than in MLM. Among the kinetic constants, the lag phase (λ) refers to the time required for the anaerobic microorganisms to adapt to the new environmental conditions. For an effective AD process, the λ values are needed to be relatively short. According to the MGM and MLM, the λ values of the pretreated substrate were lower than that of the untreated substrate (Table S2). This is most likely due to the rapid acclimation time of anaerobic microorganisms supported by a more significant presence of dissolved organic matter in the liquid phase after the UPs compared to the absence of pretreatment (Oliva et al., 2022c). The specific methane yield (μ_m) was accordingly enhanced from approximately 10 to 17 mL CH_4/g VS·d (Table S2) in R0 and R5, respectively. About A values (Table S2), which refers to the maximum methane yield, were positively related to the ultimate CMY obtained in this study, as previously reported in the scientific literature (Bianco et al., 2022).

3.3.2. Auto-Regressive Integrated Moving-Average modeling results

Simulating CMY curves with nonlinear regression models in the anaerobic process can provide more detailed information about the

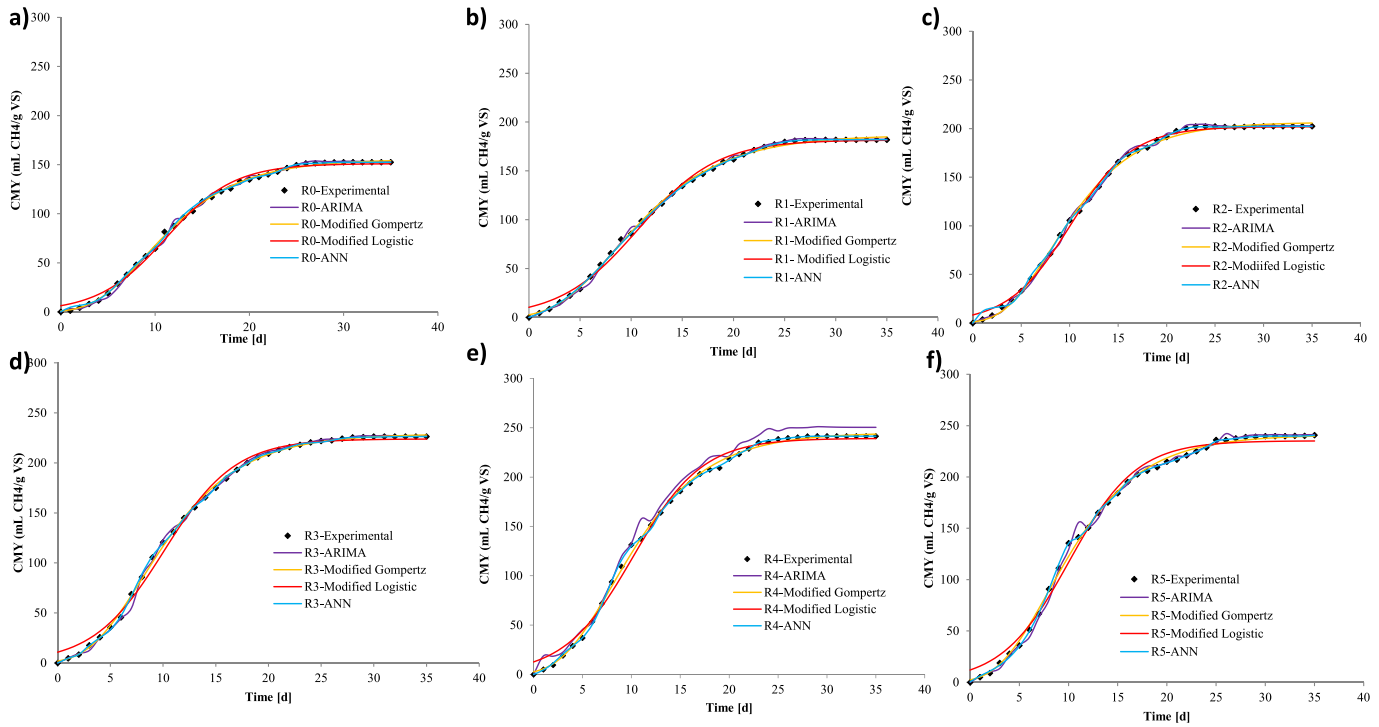


Fig. 1. Artificial Neural Network (ANN), Auto-Regressive Integrated Moving-Average (ARIMA), Modified Gompterz (MGM) and Modified Logistic (MLM) models fitting the experimental cumulative methane yields (CMYs) in R0 (a), R1 (b), R2 (c), R3 (d), R4 (e), and R5 (f).

process (Tufaner & Demirci, 2020). In this study, the ARIMA model from time series was applied for the first time as an alternative to nonlinear regression models to predict CMY curves after the UP. The obtained methane yields through the ARIMA model are shown in Fig. 1, along with other results. The ARIMA model can be understood by evaluating the connection between the temporal series and methane production (Fig. 1). The present methodology can be very responsive to a wide range of full-scale batch plants that will use similar substrates as feeding after applying the UP at specific times. According to the current results, only minimal or no adaptions of the strategy need to be considered to simulate the CMY. The ARIMA model's performance indicators (i.e., R^2, SSE, and RMSE) are reported in Table 2. Considering that as an indicator of high model performance, an R^2 value (i.e., up to 0.9988) near one and the SSE and RMSE (i.e., 1.5382 and 2.3000 in R1, respectively) close to 0, the ARIMA model's employment performed relatively lower than the ANN (Table 2). However, when explicitly examined, there does not seem to be much difference between model performances. On the contrary, the performance of the ARIMA model is better when compared to the MGM and MLM model performance indicators, as shown in Table S2. Thus, due to its robustness and simplicity, the ARIMA model is a handy tool for replicating methane production during AD. This choice is supported by the auto-correlated observations and records (i.e., CMYs) predictable only by time series. Another explanation for using time series analysis is that there were no records of other explanatory covariates to investigate other representative models. Compared to the existing sigmoidal models (Table S2), only a minimum of output parameters are essential for the methane prediction. In addition to the accessibility of the data, it is guaranteed that data originates solely from the individual AD operation, and no extra assumptions based on lab tests are needed. To assess the extent of the reactivity of this approach entirely, future investigations and experiments concerning procedure failures and operating parameters are needed.

3.3.3. *Artificial neural network modeling results*

To make a more accurate comparison with nonlinear regression models (i.e., sigmoidal models), only 'day' was taken as input and 'methane yield' as output in the ANN modeling study. As in the previous study, an ANN model was established with 35 data sets each and 70 data sets for days and methane yields in the 35-day methane production process (Şenol, 2021). Methane yields of the unpretreated and pretreated SSTS were recorded daily (i.e., 35 d in total), and approximately 70% of this data (i.e., 25) were used to teach the ANN model, and the remaining 10 were used for testing and validation (Şenol, 2021; Tufaner & Demirci, 2020). The choise of an appropriate training algorithm is vital to identify the optimal architecture in ANN modeling work. This study used the tangential sigmoid transfer function (tansig) and linear transfer function (purelin) in the hidden and output layers, which performed well in previous studies, respectively (Tufaner & Demirci, 2020). One of the high-performance indicators of a network algorithm is the number of hidden neurons specified in the network (Antwi et al., 2017).

Table 2
Performance criteria for time series and artificial neural network (ANN) models.

Models	Performance criteria	R0	R1	R2	R3	R4	R5
ARIMA	R^2	0.9977	0.9988	0.9987	0.9986	0.9884	0.9975
	SSE	1.7763	1.5382	1.8905	1.6064	8.0818	2.7283
	RMSE	2.7500	2.3000	2.7400	3.0400	3.9900	4.3800
ANN	R^2	0.9992	0.9996	0.9990	0.9998	0.9996	0.9998
	SSE	2.3106	0.6249	1.1003	0.6293	0.9432	0.7376
	RMSE	1.5200	0.7905	1.0489	0.79329	0.9433	0.8589

Note. ARIMA: auto-regressive ıntegrated moving-average. R0 – R5 are the experimental reactors described in Table 1.

The number of neurons in the hidden layer was 10, which gave the lowest MSE (mean squared error) number according to the trial and error method. ANN successfully predicted the methane productions of untreated and pretreated SSTS; the results are given in Fig. S5, along with other modeling results. Fig. S5a shows the regression values of the data allocated for training, validation and testing of the SSTS without preprocessing. R^2, the indicator of the relationship between the output and the target, varied between 0.9934 and 0.9999, and it can be understood from this that the ANN model network completed the learning process. Fig. S5b shows the error values for training, validation and testing the ANN model. Fig. S5c shows the error values for training, validation and test data at each iteration due to network training. In this section, the number of epochs in network training was taken as 21, and the best performance was achieved in the 15th epoch. Similar to Fig. S5, the training performance, regression and error graphs of the ANN model of the pretreated conditions are shown in Fig. S3 – S8 (see Supplementary Materials). It can be concluded that the ANN model better performed than sigmoidal models for predicting CMY data, as previously reported in the literature (Şenol, 2021; Tufaner & Demirci, 2020). This can be due to the good predictive power of the ANN-based model, which can find relationships between input and output in a highly complex system such as AD.

4. Conclusions

In this study, CMYs were successfully enhanced through UP for 0 – 60 min. Employing 30 min of UP (i.e., SE_N = 38,544 kJ/kg TS) resulted in a CMY of approximately 242 mL/g VS, and ΔSCOD was about 69% higher than the control. Afterwards, CMYs achieved by the SSTS were successfully fitted through the ANNs and ARIMA model, which is here proposed for the first time, due to a better prediction performance compared to the MGM and MLM models. Therefore, the ANN model can be a valuable tool for predicting and implementing the CMYs in full-scale biogas plants.

CRediT authorship contribution statement

Halil Şenol: Conceptualization, Data curation, Formal analysis, Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision, Validation, Writing – original draft, Writing – review & editing. **İlkay Türk Çakır:** Funding acquisition, Investigation, Methodology, Project administration, Resources, Software, Supervision. **Francesco Bianco:** Data curation, Supervision, Visualization, Writing – original draft, Writing – review & editing. **Emre Görgün:** Formal analysis, Investigation, Methodology, Project administration, Supervision, Validation, Visualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.biortech.2023.129866.

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