



A fuzzy-logic-based model to predict biogas and methane production rates in a pilot-scale mesophilic UASB reactor treating molasses wastewater

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ABSTRACT

A MIMO (multiple inputs and multiple outputs) fuzzy-logic-based model was developed to predict biogas and methane production rates in a pilot-scale 90-L mesophilic up-flow anaerobic sludge blanket (UASB) reactor treating molasses wastewater. Five input variables such as volumetric organic loading rate (OLR), volumetric total chemical oxygen demand (TCOD) removal rate (R_V), influent alkalinity, influent pH and effluent pH were fuzzified by the use of an artificial intelligence-based approach. Trapezoidal membership functions with eight levels were conducted for the fuzzy subsets, and a Mamdani-type fuzzy inference system was used to implement a total of 134 rules in the IF-THEN format. The product (prod) and the centre of gravity (COG, centroid) methods were employed as the inference operator and defuzzification methods, respectively. Fuzzy-logic predicted results were compared with the outputs of two exponential non-linear regression models derived in this study. The UASB reactor showed a remarkable performance on the treatment of molasses wastewater, with an average TCOD removal efficiency of 93 (± 3)% and an average volumetric TCOD removal rate of 6.87 (± 3.93) kg TCOD_{removed}/m³-day, respectively. Findings of this study clearly indicated that, compared to non-linear regression models, the proposed MIMO fuzzy-logic-based model produced smaller deviations and exhibited a superior predictive performance on forecasting of both biogas and methane production rates with satisfactory determination coefficients over 0.98.

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1. Introduction

Distillery effluents from sugar cane factories using molasses as a raw material are characterized by highly organically polluted wastewaters, and can have serious impacts on the environment if discharge untreated. Molasses-based distilleries are one of the most polluting industries generating large volumes of high-strength wastewater [1]. Apart from high organic content, distillery wastewater also contains high concentration of nutrients in the form of nitrogen (1660–4200 mg/L), phosphorus (225–3038 mg/L) and potassium (9600–17,475 mg/L) [2] that can lead to eutrophication of receiving water bodies [1,3]. The raw molasses wastewater is also characterized by moderately acidic (pH 4–5), very high total chemical oxygen demand (TCOD) (65,000–130,000 mg/L), high concentration of mineral salts and has a bad smell and dark brown color as the melanoidin pigment [1,3–5]. This dark color

hinders photosynthesis by blocking sunlight and is deleterious to aquatic life [6]. Moreover, studies focused on the water quality of a river contaminated with distillery effluent displayed high biological oxygen demand (BOD) values of about 1600–21,000 mg/L within a radius of 8 km [1,3,7]. Adequate treatment is therefore imperative before the effluent is discharged [1,3,4,8].

With environmental regulations becoming more stringent, regulatory compliance has also become a matter of increasing concern to the fermentation and food industries. Therefore, several types of processes have recently been proposed for treating the molasses wastewater to improve the quality of the final discharge in terms of residual pollutant contents. Sirianuntapiboon and Prongtong [9] have conducted studies on the removal of color substances in molasses wastewater by using combined biological and chemical treatment processes. The study concluded that color substances can be removed by simple coagulants (such as CaO for stillage and FeCl₃ for anaerobic treated molasses wastewater (An-MWW)). But to increase the color removal efficiency in coagulation step, the authors suggested the pretreatment of molasses wastewater by aeration with or without sludge-added for about 96 h. In another study conducted by Pena et al. [5], color removal from biologically pretreated molasses wastewater by means of chemical oxidation with ozone were explored. The study concluded that ozonation was an effective treatment to remove color but

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less effective to remove organic matter. The authors reported that, depending on the applied ozone dosage, color removal from 71% to 93% and TCOD reduction from 15% to 25% were reached after 30 min reaction time. In a recent study, Zhang et al. [4] proposed a novel UASB–MFC–BAF (up-flow anaerobic sludge blanket reactor–microbial fuel cell–biological aerated filter) integrated system for simultaneous bioelectricity generation and high-strength molasses wastewater treatment. The study concluded that TCOD, sulfate and color removal efficiencies of the proposed system were achieved of 53.2%, 52.7% and 41.1%, respectively. Moreover, each unit of this system had respective function and performed well when integrated together. Sirianuntapiboon and Prasertsong [10] carried out investigations on the treatment of molasses wastewater by acetogenic bacteria BP103 in a sequencing batch reactor (SBR) system. They used acetogenic bacteria BP103 cells as the absorbent for melanoidin pigment (MP) and molasses wastewater. The study concluded that the strain showed the highest TCOD, BOD₅, TKN (total Kjeldahl nitrogen) and MP removal efficiencies of 65.2%, 82.8%, 32.1% and 50.2% on average, respectively. In another recent work, Liang et al. [8] applied coagulation/flocculation process in the polishing treatment of molasses wastewater on a bench-scale. The study concluded that ferric chloride was found to be the most effective in removing melanoidins from bio-treated molasses wastewater, achieving color and TCOD removal efficiencies of 98% and 89%, respectively. Finally, Sohsalam and Sirianuntapiboon [3] applied a surface flow constructed wetland (SFCW) system with *Cyperus involucratus*, *Typha augustifolia* and *Thalia dealbata* J. Fraser to treat An-MWW under the organic loading rates (OLR) between 612 and 1213 kg BOD₅/ha-day. The proposed SFCW system showed that the highest SS (suspended solids), BOD, TCOD, total phosphorus, ammonia nitrogen, nitrate nitrogen and molasses pigments removal efficiencies of 90–93%, 88–89%, 67%, 70–76%, 77–82%, 94–95% and 72–77% were detected under the lowest OLR of 612 kg BOD₅/ha-day, respectively. Detailed information on several other existing and advances methods applied to the treatment of molasses-based distillery wastewater can be found in a comprehensive review of Satyawali and Balakrishnan [1].

In recent years, anaerobic digestion technology has become a technology of growing importance, especially for high-strength wastewater [11,12]. Although anaerobic digestion has been regarded as one of the beneficial and advantageous processes, particularly in treatment of highly polluted wastewaters, however this type of treatment is thought to be a difficult system due to its instability and complexity in solving this problem in a short time in a real-scale plant.

Modeling is a valuable tool in both design and operation of biological treatment plants, and can be used for process optimization and testing of control strategies at a reasonable cost. Hence, modeling helps to develop a better understanding of the treatment processes and provides a significant potential for solving operational problems as well as reducing operational cost in a specific treatment process. Moreover, model results can be evaluated for different operating data before transferring the concepts to a full scale plant [13]. In the literature, there are numerous studies such as integral dynamic modeling of the UASB reactor [14], mathematical simulation of the sludge blanket of UASB reactor [15], dynamic modeling of a singlestage high-rate anaerobic reactor [16], and mathematical modeling of a batch anaerobic digestion [17], conducted about the comprehensive and complex models to control and simulate several anaerobic treatment systems. In a recent study, Pontes and Pinto [18] conducted studies on the analysis of integrated kinetic and flow (or hydraulic) models for two anaerobic digesters, the UASB and the EGSB (expanded granular sludge bed) reactors. In the study, the flow models were found to be quite different for the UASB and EGSB reactors. Since many of the parameters used in the UASB reactor flow model can be critical for an accurate

simulation, the study concluded that the volume variations of the sections of the UASB reactor were necessary to accurately describe the behaviour of such digester in non-steady-state. Because of the mechanisms associated with anaerobic processes are not adequately understood to formulate reliably, many of conventional models require simplifications of the process representation for a better understanding of the underlying phenomena in anaerobic digestion. Hence, more simple and useful models are required to overcome complexity and applicability [19]. Biyikoglu et al. [20] have reported that conventional numerical methods require much time to obtain accurate results, and sometimes it is not possible to reach a solution due to convergence problems regarding the type of governing equations and boundary conditions.

The performance of anaerobic digestion processes is complex and highly dependent on the configurations of the different reactors, and varies significantly with different influent characteristics and operational conditions [19,21]. Therefore, the system must be continuously monitored and controlled due to its instability in circumstance conditions, particularly in terms of biogas or methane production rates, providing an indication of the overall anaerobic biomass activity in the process [22]. Since anaerobic digestion process is very susceptible to fluctuations in process inputs such as organic loading rates, influent pH, and toxic organic compounds, biogas or methane production rates are highly dependent on the applied process conditions. Therefore, the complicated interrelationships among a number of system factors in the process may be explicated through a number of attempts in developing a representative knowledge-based prediction model allowing the investigation of the key variables in greater detail.

Because of their speed and capability of learning, robustness, predictive capabilities and non-linear characteristics, several artificial intelligence-based modeling techniques, such as artificial neural networks [23–26], fuzzy-logic [27,28], adaptive neuro-fuzzy inference systems [19,29], have recently been conducted in the modeling of various real-life processes in environmental engineering field. Among these methods, fuzzy-logic methodology has been successfully applied in a variety of ecological and environmental applications, ranging from mapping to modeling, evaluation and prediction tasks [30]. Fuzzy-logic-based models have also been conducted by many researchers as an established and promising method for modeling of various types of anaerobic processes [31–39]. However, there are no systematic papers in the literature specifically devoted to a study of an artificial intelligence-based modeling of biogas and methane production rates in a pilot-scale mesophilic UASB reactor treating molasses wastewater using the fuzzy-logic technique. Such an artificial intelligence-based control of real-time gas production rates may provide several potential advantages such as protection of the system from possible risks associated with significant fluctuations in influent characteristics, optimization of the process at a reasonable cost, providing a rapid evaluation and estimation of emissions on energetic basis, and development of a continuous early-warning strategy without requiring a complex model structure and tedious parameter estimation procedures. Therefore, clarification of the place of the present subject in the scheme of fuzzy-logic methodology can be considered as a particular field of investigation to evaluate in real-time biogas and methane production rates that are necessary to control the anaerobic process and to establish fault diagnosis.

Considering the above-mentioned facts, the specific objectives of this study were: (1) to develop a fast predicting MIMO (multiple inputs and multiple outputs) fuzzy-logic-based model for the estimation of biogas and methane production rates in a pilot-scale mesophilic UASB reactor running under various organic, hydraulic and alkalinity loading conditions; (2) to compare the proposed artificial intelligence-based model with the conventional non-linear regression approach by means of various descriptive statistics; and

(3) to verify the validity of the MIMO fuzzy-logic model by several additional testing data sets which were not used in training the model.

2. Materials and methods

2.1. Source of molasses wastewater and feed preparation

Molasses was taken from a commercial sugar factory located in Adapazari, Turkey and stored in the refrigerator at 4 °C to minimize substrate decomposition before the experiment. The content of molasses used as feedstock (in g/kg molasses) was Total Chemical Oxygen Demand (TCOD): 800–900, ammonia nitrogen ($\text{NH}_3\text{-N}$): 25–80, oxygen: 4–8, K^+ : 40–60, and Na^+ : 150–200, respectively. Weight percentages of other components in molasses (%) were determined as saccharose: 50, betaine: 5.5, K_2O : 4.7, and others: 39.8, respectively.

Satyawali and Balakrishnan [1] have reported that although the UASB system is the most popular high-rate digester that has been utilized for anaerobic treatment of various types of industrial wastewaters, however, dilution is required before treatment due to the presence of some inhibitory substances left in solution after pH correction. Therefore, the feed for UASB reactor was prepared by diluting the predetermined amount of molasses with the desired quantity of tap water (1:10–1:3-fold). After the acclimatization of seed culture, the dilution ratio was gradually decreased from 1:10 to 1:3 to increase organic loading rate (from about 2 to 17 kg TCOD/ $\text{m}^3\text{-day}$) and to study the effects of different feed strengths on the digestion performance of the reactor. Since molasses wastewater is characterized by moderately acidic [4], the pH of the feed wastewater was adjusted by the gradual addition of NaHCO_3 (Merck Chemical Corp.), as similarly conducted by Gohil and Nakhla [40]. A nutrient solution/basal media containing essential micro and macro nutrients for an optimum anaerobic microbial growth was also prepared with the following components, and added 1 mL/L of the daily fed substrate at relatively high organic loading conditions [41]: 5 g/L $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, 6 g/L $\text{FeCl}_2 \cdot 6\text{H}_2\text{O}$, 10 g/L $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$, 1 mg/L H_3BO_3 , 1 mg/L $\text{ZnSO}_4 \cdot 7\text{H}_2\text{O}$, 1 mg/L $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 100 mg/L $\text{MnCl}_2 \cdot 6\text{H}_2\text{O}$, 1 mg/L $(\text{NH}_4)_6\text{Mo}_{24} \cdot 4\text{H}_2\text{O}$, 585 mg/L $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$, and 1 g/L $\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$. It is noted that addition of other micronutrients (such as nickel and selenium) to the feed material in the form of their salts may also play an important role on the growth of microorganisms and help to increase biogas production.

2.2. UASB set-up and operation

The molasses wastewater was anaerobically treated in a pilot-scale UASB reactor under different organic and hydraulic loading conditions. The internal diameter, total height and total tank capacity of the system were 20 cm, 190 cm and 90 L, respectively. All parts of the reactor was made of ANSI 316 stainless steel. The reactor had a conical bottom of 20 cm length and a feed inlet pipe of about 1.0 cm diameter to avoid choking during operation. An outlet weir was provided at the top (1.85 m), which was connected to an outlet pipe to the effluent collection tank. The reactor was equipped with five sampling ports, localized at 0.30, 0.45, 0.60, 0.75 and 0.90 from the bottom of the system. The diameter of each sampling port was about 1.5 cm.

Biogas was collected from the headspace on the top of the reactor via a gas collecting system. The gas collecting and measuring system consisted of a gas–solid–liquid (GSL) separator (made from inverted plastic funnels of 15 cm diameter), a gas collecting pipe, a glass water trap used as hydrogen sulfide (H_2S) scrubber and a wet-tip gas meter. The reactor was kept under mesophilic conditions

(35.2 ± 0.7 °C) by circulating the hot water through the external reactor jacket with a Fisher Isotemp 2100 (Fisher Company, Pittsburgh) immersion circulator. Heated water was pumped through the jacket surrounding the reactor and glass wool was used as an isolation material.

Ward et al. [42] have reported that a certain degree of mixing can be beneficial in terms of productivity, as well as of presenting substrate to the bacteria. Although the low speed mixing conditions allow the digester to better absorb the disturbance of shock loading than did high speed mixing conditions, however, excessive mixing can reduce methane production and disrupt the granule structure, reducing the rate of oxidation of fatty acids which can lead to digester instability [43–45]. Moreover, Appels et al. [46] have reported that proper auxiliary mixing prevents both the formation of surface scum layers and the deposition of sludge on the bottom of the tank. Therefore, considering the above-mentioned facts, reactor contents were gently mixed by an adjustable low-speed top mounted mixer shaft coupled with two stainless steel impellers (ANSI 316) driven by a geared DC motor (0.25 kW, 60 rpm).

Depending on the feed flow rates (from 40.5 to 165.1 L/day), the reactor was operated in a semi-continuous mode feeding (i.e. twice an hour for 15 min) by pumping of the fresh feed into the reactor and collecting effluent samples daily. The reactor was run for a period of about 2 years under various organic and hydraulic loading rates. Influent and effluent sampling was carried out once the steady-state period was achieved. In feeding, different target hydraulic retention times (HRTs) were achieved using a peristaltic pump (ColeParmer, Masterflex®). Sufficient up-flow velocity was maintained to achieve proper fluidization inside the reactor. During the feeding of the reactor, the feeding tank was occasionally agitated with a glass rod to prevent the sedimentation of suspended solids, as well as to make the feeding solution homogeneous. In order to increase the efficiency of the digestion process, the reactor were seeded with anaerobic sludge (about 25% of the working volume) taken from the Kartonsan Factory Anaerobic Treatment Facility (Corlu, Istanbul, Turkey). A detailed schematic of the experimental set-up is depicted in Fig. 1.

2.3. Representation of model parameters

Identification of parameters that could be used for monitoring the biological treatment system is an important factor for efficient operation of the anaerobic digestion processes. Choosing the most appropriate model components representing the behaviour of the studied process can help to recognize possible technical faults and to reduce operating costs of plants in the planning stage [12,47]. There are several suggestions in the literature regarding the choice of parameters [22]. On the basis of the existing experimental data, several combinations of parameters were pre-trained until the best input parameters and best fitting input structure were developed. Following to preliminary computations, we selected volumetric organic loading rate (OLR), volumetric Total Chemical Oxygen Demand (TCOD) removal rate (R_V), influent alkalinity, influent pH and effluent pH as the input parameters in our modeling study. Biogas and methane production rates in the UASB reactor were the output parameters of the proposed fuzzy-logic model. The present model components, which are among the most widely used and monitored parameters in the literature, are briefly discussed below:

2.3.1. Effect of organic loading rate

The organic loading rate (OLR) is an important parameter significantly affecting microbial ecology and characteristics of anaerobic systems. This parameter integrates the operational characteristics of the reactor, and bacterial mass and activity into the volume of media [48]. Verma [49] has reported that OLR is a measure of the

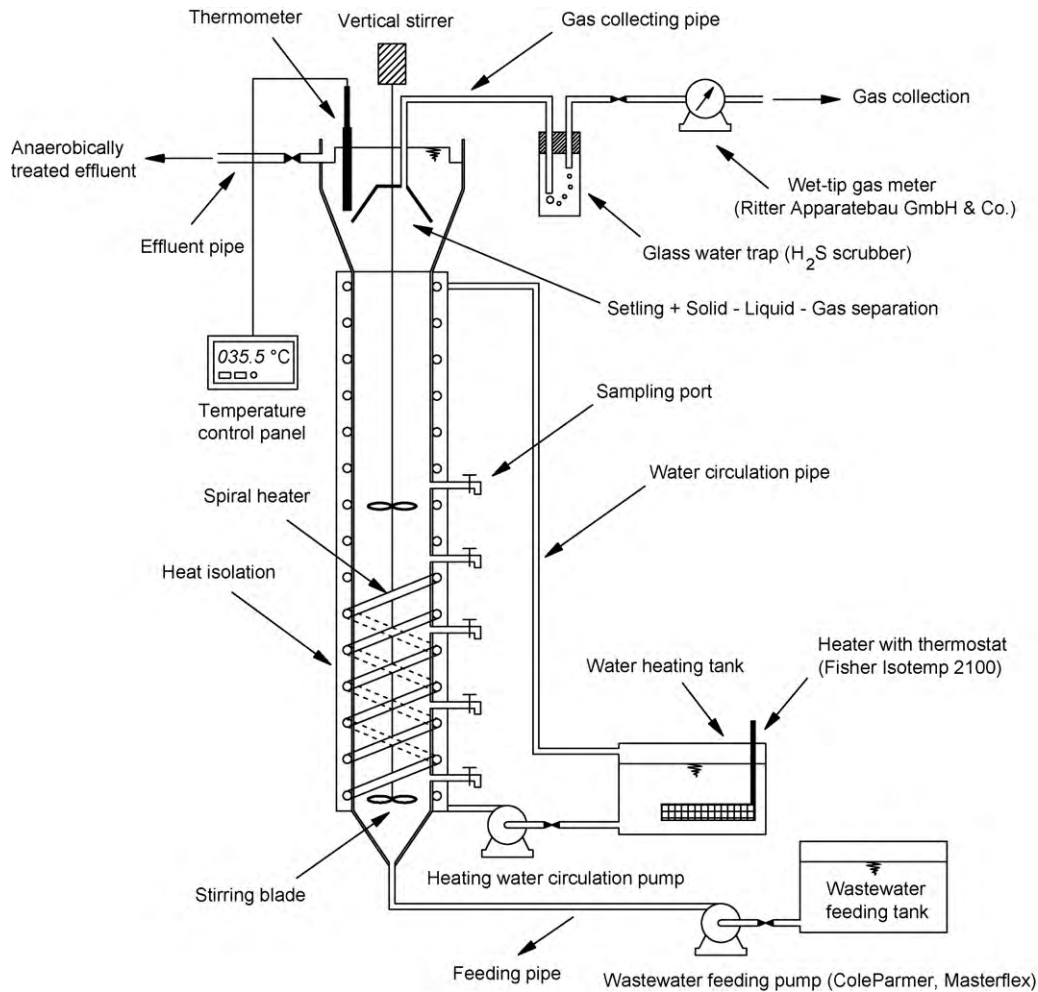


Fig. 1. A detailed schematic of the experimental set-up.

biological conversion capacity of the anaerobic treatment system. It depends on the technology used and on the type of wastewater to be treated [22]. Satyawali and Balakrishnan [1] have reported that, depending on various anaerobic reactor configurations, a wide range of OLRs ranging from 0.6 to 86.4 kg TCOD/m³-day have been successfully applied for treating molasses distillery wastewater.

2.3.2. Effect of volumetric TCOD removal rate

The volumetric TCOD removal rate (R_V) is a function of influent flow rate, working volume of the reactor, and the difference between influent and effluent substrate concentrations [50]. This is an important parameter for biological treatment processes in terms of a measure of substrate utilization efficiency and microbial metabolic activity in real-time. It is clear from the literature that high R_V values have been achieved in treatment of various organic-laden wastewaters such as poultry manure wastewater [47], dairy wastewater [51], specifically with the use of anaerobic processes.

2.3.3. Effect of alkalinity

Alkalinity refers to the ability of a solution to resist changes in pH. Alkalinity is important because as acid is added to solution, carbonates will contribute hydroxide ions, which tend to neutralize the acid. This is known as the buffering effect of alkalinity [52]. Buffer capacity is a more reliable method of measuring digester imbalance than direct measurements of pH, as an accumulation of short chain fatty acids will reduce the buffering capacity significantly before the pH decreases [42]. Moreover, it is noted that

alkalinity is not only important for pH-regulation, but also as the pool for CO₂ in methane production. In general, sodium bicarbonate is used for supplementing the alkalinity, as it is the only chemical that gently shifts the equilibrium to the desired value without disturbing the bacterial activity [22].

2.3.4. Effect of pH

It has been determined that an optimum pH value for anaerobic treatment lies between 5.5 and 8.5 [49]. Methane bacteria are very sensitive to pH value. They need a pH range between 6.5 and 7.8 whereas the acid-producing bacteria have optimum pH value between 5 and 6 [53]. This is an important reason why some designers prefer the separation of the hydrolysis/acidification and acetogenesis/methanogenesis processes in two-stage processes [42]. Liu et al. [54] have reported that the pH range is relative wide in the plant scale and the optimal value of pH varies with substrate and digestion technique.

2.3.5. Biogas and methane production rates

In any anaerobic digester, effectiveness of the process is usually represented in terms of biogas production rate [55]. The most important step in the operation of biogas reactor is the control of the digestion process to maximize the methane production from biological decomposition of organic matters in the waste. Decreases in biogas yield and methane content are the potential indicators of an unstable process condition in the anaerobic digestion. The stability of the system should be attentively examined for the methane

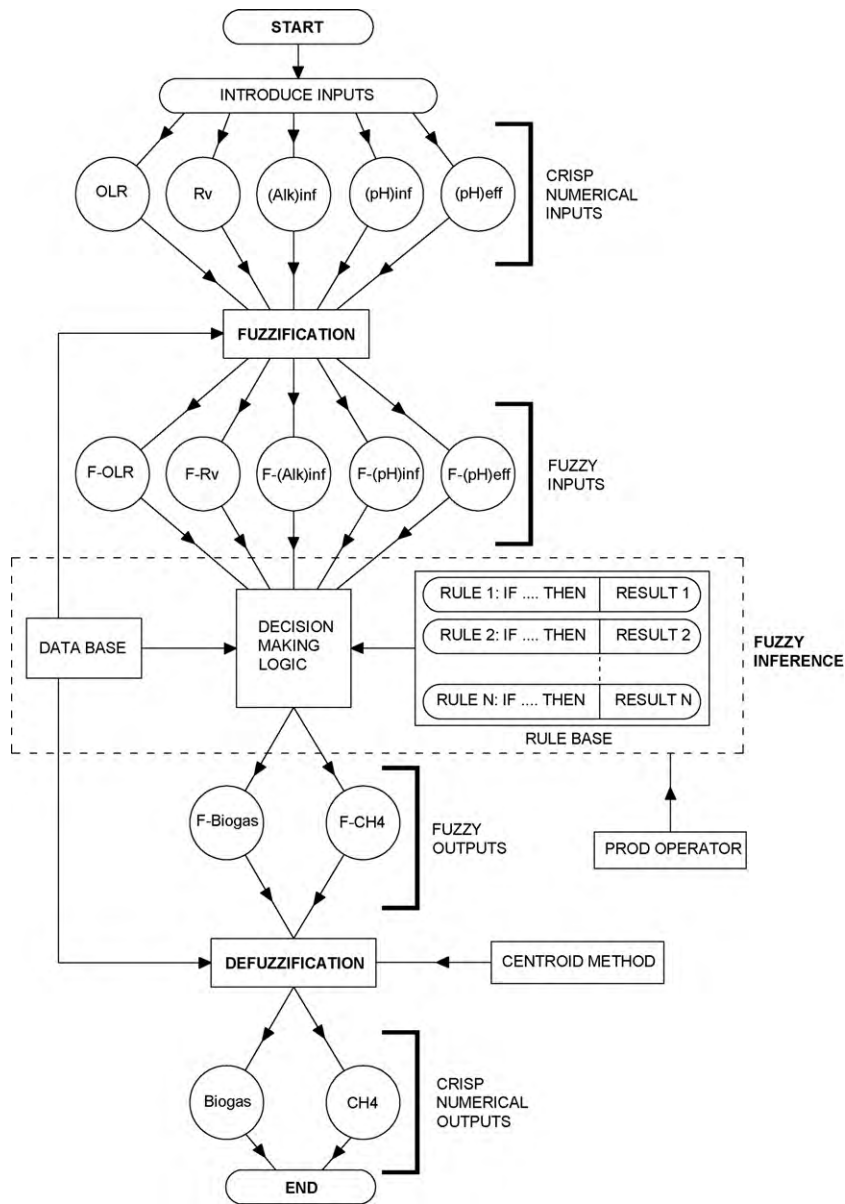


Fig. 2. A detailed schematic of the MIMO fuzzy system applied in this study.

content below 65% [52]. The gas production rates provide an indication of the overall anaerobic biomass activity [22]. Therefore, in this study, we selected biogas and methane production rates as the output parameters of the proposed models.

2.4. Fuzzy-logic methodology

A general fuzzy system has basically four components: fuzzification, fuzzy rule base, fuzzy output engine, and defuzzification [56]. In fuzzification step, numerical inputs and output variables are converted into linguistic terms or adjectives (such as low, high, big, small, etc.), and the corresponding degrees of the one or more several membership functions are determined [28]. Since multiple measured crisp inputs first have to be mapped into fuzzy membership functions, the fuzzification process requires good understanding of all the variables [57]. Akkurt et al. [56] have reported that fuzzy inference engine takes into account all the fuzzy rules in the fuzzy rule base and learns how to transform a set of inputs to corresponding outputs. Two kinds of inference operators, minimization (min) and product (prod), are basically employed in

this step [56,58]. In this study, we conducted the prod technique due to its better performance in collection of all the relations among inputs and outputs fuzzy sets in the fuzzy rule base.

Finally, in the defuzzification step, linguistic results obtained from the fuzzy inference are translated into a real value by using the rule base provided [20]. This phase is responsible for transforming the fuzzy results from the fuzzy system into crisp values [58]. Akkurt et al. [56] have reported that there are many defuzzification methods such as centre of gravity (COG or centroid), bisector of area, mean of maxima, leftmost maximum, rightmost maximum, etc. As conducted by several researchers [28,56,57], in this study, we employed centroid method which is most commonly used defuzzification technique. It is expressed as follows [56,57]:

$$(y_i)_d = \frac{\sum_{i=1}^n \mu(y_i) y_i}{\sum_{i=1}^n \mu(y_i)} \quad (1)$$

where $(y_i)_d$ is the defuzzified output, y_i is the output value (or the centroidal distance from the origin) in the i th subset, and $\mu(y_i)$ is the membership value of the output value in the i th subset. For the

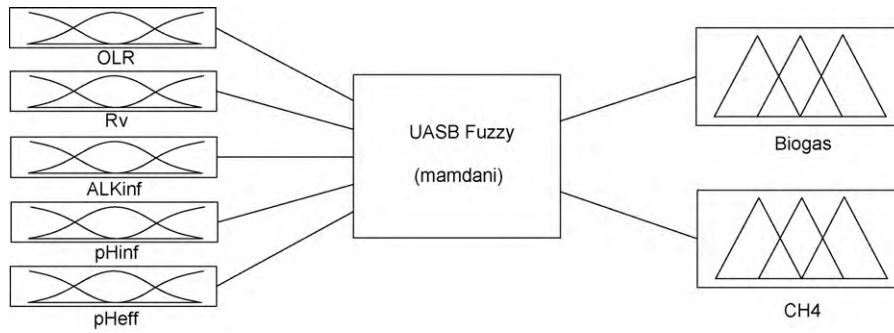


Fig. 3. Input and output variables considered for the present MIMO fuzzy model.

continuous case, the summations in Eq. (1) are replaced by integrals, as given by Sadiq et al. [59]. On the basis of above-mentioned fuzzy steps, a detailed schematic of the MIMO (multiple inputs and multiple outputs) fuzzy system applied in this study is depicted in Fig. 2.

The situations of uncertainties in fuzzy-logic are defined via giving appropriate membership functions to the elements of the set that represent the situation. The value of the variation between 0 and 1 (the highest level) for each element is called membership degree and its value in subset is called membership function [60]. In fuzzy models, the shape of membership functions of fuzzy sets can be triangular, trapezoidal, bell-shaped, sigmoidal, or another appropriate form, depending on the nature of the system being studied [30,59,61]. Among them, triangular and trapezoidal shaped membership functions are predominant in current applications of fuzzy set theory, due to their simplicity in both design and implementation based on little information [62]. As suggested by others [63,64], in this study, we selected trapezoidal shaped membership functions for both input and output variables for optimal software performance. The scalar parameters of trapezoidal membership functions were adjusted until satisfactory outputs were obtained with respect to the set of rules used in the study, as similarly conducted by Mitra et al. [63]. The trapezoidal curve is membership function of a vector, x , and depends on four scalar parameters, a, b, c, d , as follows [28,57,65]:

$$\mu(x) = \mu(x; a, b, c, d) = \begin{cases} 0, & x \leq a \\ \frac{x-a}{b-a}, & a \leq x \leq b \\ 1, & b \leq x \leq c \\ \frac{d-x}{d-c}, & c \leq x \leq d \\ 0, & d \leq x \end{cases} \quad (2)$$

In this work, Fuzzy Logic Toolbox was used to create and to edit the present Fuzzy Inference System (FIS) within the framework of MATLAB® V7.0. The steady-state data obtained from the experimental study were allocated into fuzzy sets to represent different levels of crisp numerical variables. Input and output variables were built by using the FIS Editor, and fuzzified with trapezoidal membership functions (trapmf). Fig. 3 illustrates input and output variables on the MATLAB® numeric computing environment. Each input variable and output variable had eight membership functions namely A, B, C, D, E, F, G, and H. Some extreme values for each variable were also considered with their respective ranges to enhance the prediction flexibility of the model. Organic loading rate (OLR) ranged from 1.9 to 16.7 kg TCOD/m³-day in X-axis. Fig. 4a depicts the shape and range of each level for the first input variable. Volumetric TCOD removal rate (R_v), the second input variable, ranged from 1.9 to 16.7 kg TCOD/m³-day, and the shape and range of its membership functions are shown in Fig. 4b. Influent alkalinity (ALK_{inf}) and influent pH (pH_{inf}), considered as the third and the

fourth input variables, ranged from 230 to 1950 mg CaCO₃/L, and from 4.0 to 7.4, respectively (Fig. 5a and b). The effluent pH (pH_{eff}), the fifth input variable, ranged from 6.4 to 7.6, and the shape and range of membership functions for this input are depicted in Fig. 6c. Biogas production rate, output 1, ranged from 45 to 760 L/day in X-axis. Fig. 6a presents the shape and range of each level for the first output variable. Methane production rate were considered as output 2, and ranged from 35 to 490 L/day, as shown in Fig. 6b. Table 1 summarizes the number of membership functions and their ranks, trapmf[abcd], for each of the input and output variables considered in the present fuzzy sets.

In this study, a total of 134 rules were established with the Fuzzy IF-THEN Rule Editor, and a decision from the combinations of input membership functions (premise part or the antecedent block) to output membership functions (consequent part) was made by experience and the steady-state experimental data set. As an example, Table 2 presents the rule base of 30 rule sets randomly selected from the total 134 sets built on the MATLAB® environment.

In the applications of the fuzzy system in both control and forecasting, there are two types of fuzzy inference systems, namely, Mamdani-type [66] and Takagi-Sugeno-type [67] fuzzy systems [62,68,69]. Because of allowing a simplified representation and interpretation of the fuzzy rules, Mamdani's fuzzy inference method is the most commonly applied fuzzy methodology [61].

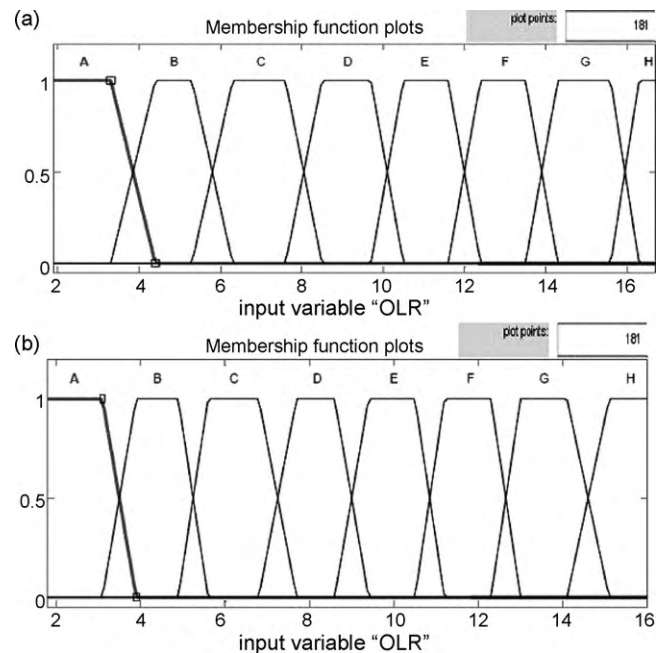


Fig. 4. Fuzzification of organic loading rate (OLR, kg TCOD/m³-day) and volumetric TCOD removal rate (R_v , kg TCOD/m³-day).

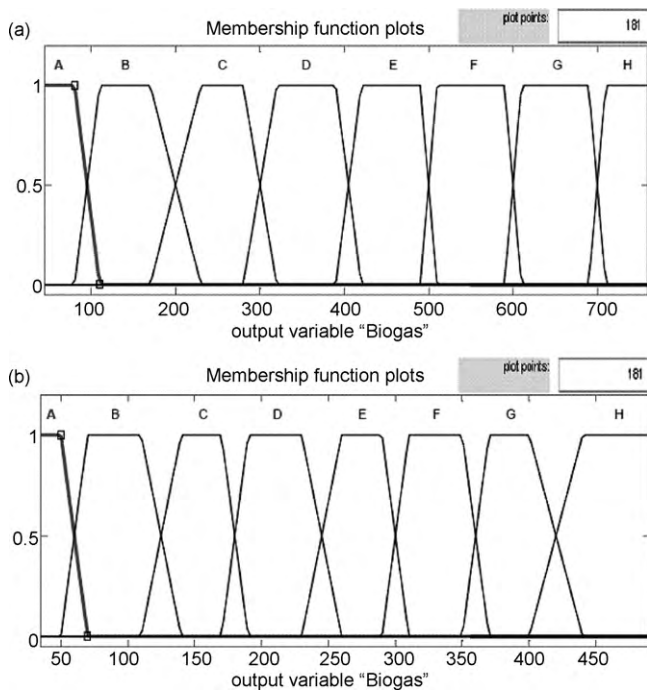


Fig. 5. Fuzzification of influent alkalinity (ALK_{inf} , mg $CaCO_3/L$), influent pH (pH_{inf}) and effluent pH (pH_{eff}).

This kind of system allows a simplified representation and interpretation of the fuzzy rules, and the integration of expert knowledge is thus easily realizable [70]. Therefore, as similarly conducted by several researchers [56,61,65,70–73], in this study, we used a Mamdani-type fuzzy inference system to implement IF-THEN rules for prediction of both biogas and methane production rates.

2.5. Non-linear modeling study

In this study, a non-linear modeling study was also carried out to appraise the performance of the UASB reactor treating molasses wastewater by means of biogas and methane production rates. The steady-state experimental data was evaluated by DataFit® scientific software (version 8.1.69, Copyright© 1995–2005 Oakdale Engineering, RC167) containing 298 two-dimensional (2D) and 242 three-dimensional (3D) non-linear regression models. As similarly done in our previous studies [12,13,47,74], in this study, the non-linear regression analysis was conducted based on the Levenberg-Marquardt method with double precision. The experimental data was imported directly from Microsoft® Excel used as an open database connectivity data source, and then the non-linear regression analysis was performed. As regression models were solved, they were automatically sorted according to the goodness-of-fit criteria into a graphical interface. Moreover, *t*-ratios and the

Table 1
Number of membership functions and their ranks for each of the input and output variables considered in the present fuzzy sets.

Membership functions	Input variables					Output variables	
	OLR	R_v	ALK_{inf}	pH_{inf}	pH_{eff}	Biogas	CH_4
A	[–0.60.5 3.3 4.4]	[–0.30.5 3.1 3.9]	[–2080 380 480]	[3.3 3.5 4.3 4.5]	[6.15 6.2 6.6 6.65]	[–20 1080 110]	[0 20 50 70]
B	[3.3 4.4 5.3 6.3]	[3.1 3.9 4.9 5.6]	[380 480 580 720]	[4.3 4.5 4.8 5.0]	[6.6 6.65 6.75 6.8]	[80 110 170 230]	[50 70 110 140]
C	[5.3 6.3 7.6 8.5]	[4.9 5.6 6.8 7.7]	[580 720 840 940]	[4.8 5.0 5.3 5.4]	[6.75 6.8 6.85 6.9]	[170 230 280 320]	[110 140 170 190]
D	[7.6 8.5 9.7 10.5]	[6.8 7.7 8.6 9.4]	[840 940 1060 1160]	[5.3 5.4 5.7 5.9]	[6.85 6.9 7.0 7.05]	[280 320 390 420]	[170 190 230 260]
E	[9.7 10.5 11.6 12.4]	[8.6 9.4 10.5 11.2]	[1060 1160 1280 1380]	[5.7 5.9 6.2 6.3]	[7.0 7.05 7.1 7.2]	[390 420 490 510]	[230 260 290 310]
F	[11.6 12.4 13.5 14.3]	[10.5 11.2 12.3 13.0]	[1280 1380 1510 1600]	[6.2 6.3 6.6 6.75]	[7.1 7.2 7.25 7.3]	[490 510 590 610]	[290 310 350 370]
G	[13.5 14.3 15.6 16.3]	[12.3 13.0 14.1 15.1]	[1510 1600 1710 1840]	[6.6 6.75 7.1 7.2]	[7.25 7.3 7.4 7.45]	[590 610 690 710]	[350 370 400 440]
H	[15.6 16.3 17.1 17.8]	[14.1 15.1 16.9 17.9]	[1710 1840 2060 2190]	[7.1 7.2 7.6 7.7]	[7.4 7.45 7.75 7.8]	[690 710 810 830]	[400 440 540 580]

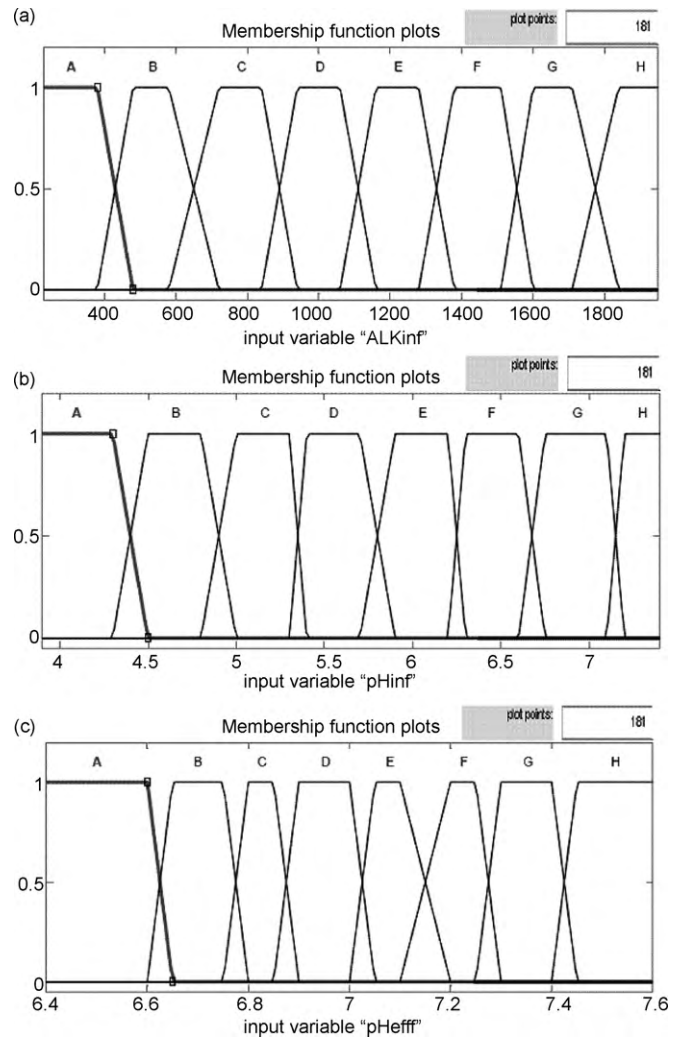


Fig. 6. Fuzzification of biogas (Biogas, L/day) and methane (CH_4 , L/day) production rates.

corresponding *p* values were determined to appraise the significance of the regression coefficients. Descriptive statistics of the residual errors were also provided to better evaluate the model performance. To determine the statistical significance of the predicted results, an alpha (α) level of 0.05 (or 95% confidence) was used in the non-linear modeling study.

2.6. Analytical procedure

Influent and effluent pH values were measured by a pH meter (Thermo Orion 210). Total Chemical Oxygen Demand (TCOD), volatile fatty acids (VFA) and alkalinity analyses were conducted

Table 2

A random selection of 30 rule sets from the total 134 sets.

Input variables					Output variables	
OLR	R_V	ALK_{inf}	pH_{inf}	pH_{eff}	Biogas	CH_4
A	A	D	E	B	A	A
A	A	D	E	C	A	A
A	A	C	D	B	A	A
A	A	C	F	A	A	A
A	A	B	C	C	A	A
A	A	C	B	B	A	A
A	A	A	B	B	B	B
A	A	A	B	A	A	A
A	A	B	B	A	A	B
A	A	C	A	B	B	B
C	C	E	C	C	B	B
C	C	F	H	D	D	D
C	C	B	A	D	E	E
G	G	A	A	D	F	F
A	A	C	B	A	B	B
A	A	B	B	B	B	B
A	A	C	B	A	B	B
B	B	A	A	A	B	B
B	B	A	A	B	B	B
B	B	A	B	A	B	B
B	B	A	A	A	B	B
B	B	B	B	A	B	B
B	B	F	B	B	B	B
D	D	B	B	C	C	C
D	D	B	B	D	C	C
F	F	H	C	G	E	F
F	F	G	C	G	E	G
F	F	H	C	G	E	G
F	G	E	D	D	F	F
H	H	D	D	D	G	H

by the procedures described in the Standard Methods [75]. Biogas produced in the reactor was measured continuously by a drum type Ritter wet-tip gasmeter (Ritter Apparatebau GmbH & Co.), and biogas composition was determined by using a portable Orsat apparatus. Stability of the treatment process and components of wastewater samples were monitored in the Civil Engineering Laboratory at Yildiz Technical University in Istanbul, Turkey. Each experiment was performed in triplicate and repeated at least three times to observe the reproducibility, and experimental results were reported as the mean value of each parameter with standard deviation.

3. Results and discussion

3.1. UASB process

On the basis of the cross-sectional area of the reactor (314.16 cm^2) and applied feed flow rates (40.5–165.1 L/day, mean: $73.4 (\pm 34) \text{ L/day}$), hydraulic loading rates (L_H) were controlled between 1.29 and $5.26 \text{ m}^3/\text{m}^2\text{-day}$, with an average value of $2.34 (\pm 1.09) \text{ m}^3/\text{m}^2\text{-day}$. The UASB reactor were conducted with different HRTs between 0.36 and 1.48 days. Imposed volumetric organic loading rates (OLR) ranged from 1.95 to $16.56 \text{ kgTCOD}/\text{m}^3\text{-day}$, with a mean value of $7.40 (\pm 4.26) \text{ kgTCOD}/\text{m}^3\text{-day}$.

Depending on various organic and hydraulic loading conditions, daily biogas production rates ranged between 46 and 753 L/day. Daily methane (CH_4) production rates ranged from 36 to 490 L/day with a mean value of about 160 L/day. Percentages of the typical components in biogas, such as CH_4 and CO_2 , ranged between 50–70% and 16–44%, with mean values of about 68 (± 6)% and 27 (± 5)%, respectively. Moreover, high volumetric TCOD removals (R_V) were achieved between 1.85–15.97 $\text{kgTCOD}_{\text{removed}}/\text{m}^3\text{-day}$, with an average value of $6.87 (\pm 3.93) \text{ kgTCOD}_{\text{removed}}/\text{m}^3\text{-day}$. Result also showed that the rounded TCOD removal percentages ranged from 84 to 98%, with an average value of 93 (± 3)%.

The average values of influent and effluent pH were about 5.01 (± 0.20) and 6.82 (± 0.21), respectively. The increase in pH can be attributed to the anaerobic bio-conversion of amino acids contained in feed wastewater to ammonia [11,47]. During the experimental study, influent alkalinity ranged from 240 to 1940 $\text{mg CaCO}_3/\text{L}$, and the average increase in alkalinity across the UASB reactor was determined to be about 1280 $\text{mg CaCO}_3/\text{L}$. The increase in alkalinity can be ascribed to the degradation of proteins in molasses wastewater by anaerobic treatment, which resulted in generation of alkalinity due to the reaction of ammonia with carbon dioxide and water, as similarly reported by others [40,76]. Alkalinity increase in the UASB reactor averaged about 0.20 $\text{mg CaCO}_3/\text{mg TCOD}_{\text{removed}}$ during the continuous experimental period. This is comparable to the value of 0.17 $\text{mg CaCO}_3/\text{mg TCOD}_{\text{removed}}$ reported by Gohil and Nakhla [40]. The stability of the anaerobic system was also monitored using the ratio of VFA:alkalinity. The steady-state data indicated that the average effluent VFA:alkalinity ratio was about 0.13, which was less than 0.4 as similarly observed by several authors [77–79].

Mudunge [80] has reported that at steady-state the daily mass of influent TCOD (MS_i) is equal to the daily mass of TCOD leaving the system by means of the daily mass of effluent COD (MS_e), the daily mass of COD in discharged sludge (MS_x), the daily mass of digested sludge (MS_d), and the daily mass of oxidised sludge (MS_o). MS_e and MS_x are contained in the effluent wastewater ($TCOD_{\text{out}}$), while the daily mass of oxidised sludge (MS_o) is incorporated into the biomass. For anaerobic bacteria, the growth rate is very slow that this amount is negligible [11]. On the basis of the present steady-state experimental data, the mole of methane in biogas was calculated using the well-known ideal gas equation, and then theoretical TCOD of methane was determined for its oxygen equivalent, as conducted by Yetilmezsoy and Sakar [11]. The present TCOD mass balance revealed that over 92% of influent organic matters imposed to the system were transformed to biogas on average. A linear regression showed a good agreement between the daily mass of influent TCOD and the daily mass of effluent TCOD, with a high determination coefficient of $R^2 = 0.8409$. In addition, the value of adjusted determination coefficient ($R_a^2 = 0.8397$) was also very high, showing a high significance of the TCOD mass balance. Moreover, a high value of the correlation coefficient ($R = 0.9170$) signified a noticeable correlation between the mass of influent and effluent TCOD values.

Consequently, the molasses wastewater was satisfactorily treated by means of a high-rate anaerobic process, specifically with the use of UASB reactor. Although relatively high incoming OLRs were imposed to the system, the UASB reactor demonstrated a stable performance on the anaerobic treatability of molasses wastewater, and no process failure was recorded. This should be due to acclimatization of both acidogens and methanogens to the gradual flow regime after a well controlled adaptation period.

3.2. Prediction of biogas and methane production rates

In this work, the developed MIMO fuzzy-logic-based model and the non-linear regression analysis-based model were applied to predict biogas and methane production rates obtained from the steady-state experimental data. In the non-linear study, one exponential model and two first-order polynomial models were obtained for prediction of both biogas and methane production rates. Results are summarized in Table 3.

Regression variable results including standard error, the t -statistics and the corresponding p values for the best-fit model are summarised in Table 4. The best-fit models defined as a function of five process variables [Biogas (Y_1) or methane (Y_2) = $f(\text{OLR}, R_V,$

Table 3
Summary of non-linear regression results for prediction of both biogas and methane production rates.

Rank	Model	SEE	SR	RA	RSS	R ²	R _a ²	NNI
Prediction of biogas production rate (output 1)								
1	$\exp(ax_1 + bx_2 + cx_3 + dx_4 + ex_5 + f)$	52.46	-192.38	-1.44	352228.46	0.8899	0.8856	6
2	$ax_1 + bx_2 + cx_3 + dx_4 + ex_5 + f$	55.66	7.2×10^{-11}	5.3×10^{-13}	396607.93	0.8761	0.8712	11
3	$ax_1 + bx_2 + cx_3 + dx_4 + ex_5$	59.80	-61.31	-0.46		0.8558	0.8513	1
Prediction of methane production rate (output 2)								
1	$\exp(ax_1 + bx_2 + cx_3 + dx_4 + ex_5 + f)$	31.82	-101.95	-0.76	129677.34	0.9053	0.9016	5
2	$ax_1 + bx_2 + cx_3 + dx_4 + ex_5 + f$	34.96	1.4×10^{-10}	1.04×10^{-12}	156410.52	0.8858	0.8813	11
3	$ax_1 + bx_2 + cx_3 + dx_4 + ex_5$	39.12	-48.80	-0.36	197439.56	0.8558	0.8513	11

SEE, standard error of the estimate; SR, sum of residuals; RA, residual average; RSS, residual sum of squares; R², coefficient of multiple determination; R_a², adjusted coefficient of multiple determination; NNI, number of non-linear iterations.

Table 4
Model components and regression variable results for the best-fit models.

Independent and original variables	SE ^a	t-Ratio	p value
$Y_1 = \exp[0.0062(OLR) + 0.1097(R_V) + 0.000104(ALK_{inf}) - 0.102(pH_{inf}) + 0.474(pH_{eff}) + 1.72]$			
$x_1 = OLR$ (kg TCOD/m ³ -day)	3.673×10^{-2}	0.1678	0.86698
$x_2 = R_V$ (kg TCOD _{removed} /m ³ -day)	3.862×10^{-2}	2.8408	0.00524 ^b
$x_3 = ALK_{inf}$ (mg CaCO ₃ /L)	5.741×10^{-5}	1.8233	0.07058
$x_4 = pH_{inf}$	3.292×10^{-2}	-3.098	0.00239 ^b
$x_5 = pH_{eff}$	0.1162	4.0781	0.00008 ^b
$Y_2 = \exp[-0.0596(OLR) + 0.1696(R_V) + 0.00022(ALK_{inf}) - 0.142(pH_{inf}) + 0.618(pH_{eff}) + 0.51]$			
$x_1 = OLR$ (kg TCOD/m ³ -day)	3.482×10^{-2}	-1.7109	0.08952 ^b
$x_2 = R_V$ (kg TCOD _{removed} /m ³ -day)	3.648×10^{-2}	4.6490	0.00001 ^b
$x_3 = ALK_{inf}$ (mg CaCO ₃ /L)	5.218×10^{-5}	4.2901	0.00003 ^b
$x_4 = pH_{inf}$	0.0296	-4.7889	0.00000 ^b
$x_5 = pH_{eff}$	0.1069	5.7822	0.00000 ^b

^a Standard error.

^b p values < 0.05 were considered to be significant.

ALK_{inf}, pH_{inf}, pH_{eff})] are given in the following equations:

$$Y_1 = \exp[0.0062(OLR) + 0.1097(R_V) + 0.000104(ALK_{inf}) - 0.102(pH_{inf}) + 0.474(pH_{eff}) + 1.72] \quad (3)$$

$$Y_2 = \exp[-0.0596(OLR) + 0.1696(R_V) + 0.00022(ALK_{inf}) - 0.142(pH_{inf}) + 0.618(pH_{eff}) + 0.51] \quad (4)$$

The larger *t*-ratio indicates the more significant parameter in the regression model. Moreover, the variable with the lowest *p* value is considered the most significant [12]. Based on *t*-ratios and *p* values given in Table 3, volumetric TCOD removal rate and effluent pH had more importance than the OLR, influent alkalinity and influent pH for the derived exponential models in prediction of both biogas and methane production rates. In this study, the MIMO fuzzy-logic-based model was developed based on a total of 134 rules in the IF-THEN format and tested with 40 different experimental data, used as the testing set, randomly selected from the remaining steady-state data set. Briefly, about 77% of the total steady-state experimental data was used as the learning set based on a total of 134 rules, and the remaining (about 23% of the total) was used as the testing set (a total of 40 rules) to verify the prediction performance of the proposed MIMO fuzzy-logic-based model. Fig. 7 shows a head-to-head comparison of performance for experimental data, fuzzy-logic testing outputs and the regression model outputs by means of biogas and methane production rates. Moreover, variations of other variables (organic loading rate and volumetric TCOD removal rate, influent pH and effluent pH, and influent and effluent alkalinity) in the testing set are depicted in Fig. 8. In the testing set, the average values of imposed volumetric organic loading rates and volumetric TCOD removals were about 7.25 (±4.74) kg TCOD/m³-day and 6.74 (±4.39) kg TCOD_{removed}/m³-day, respectively (Fig. 8a). Result indi-

cated that the rounded TCOD removal percentages ranged from 87% to 97%, with an average value of 93 (±2.6)%. The influent and effluent pH averaged about 4.96 (±0.78) and 6.81 (±0.22), respectively (Fig. 8b). The influent alkalinity ranged from 270 to

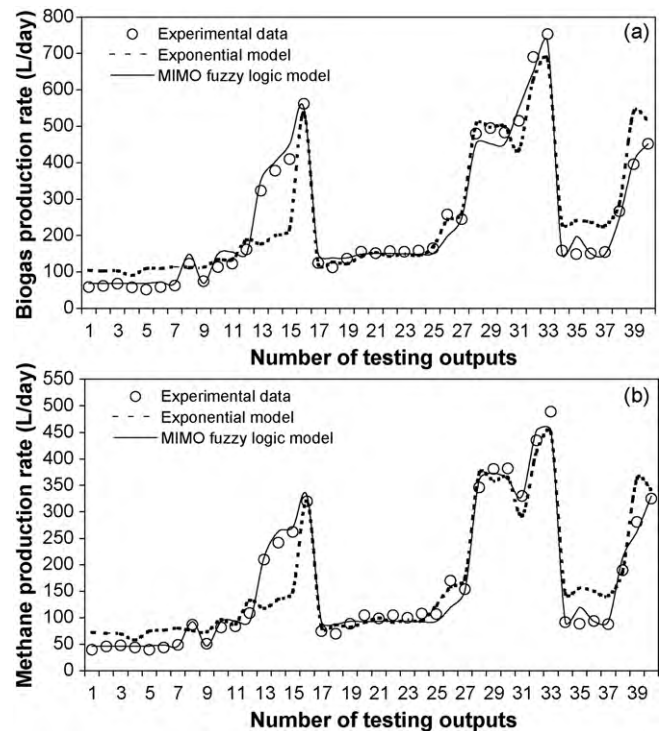


Fig. 7. A head-to-head comparison of performance for experimental data, fuzzy-logic testing outputs (responses for 40 different experimental data used as the testing set) and the regression model outputs by means of biogas (a) and methane (b) production rates.

Table 5
Descriptive performance indices corresponding to the testing data of each model.

Estimator	Testing data used in the modeling study			
	Biogas production rate		Methane production rate	
	FLM ^a	NRM ^b	FLM ^a	NRM ^b
R^2	0.9847	0.8721	0.9848	0.8935
R	0.9675	0.9104	0.9676	0.9216
a	0.9536	0.8469	0.9797	0.8626
b	12.898	41.937	3.5422	25.965
Mean (measured, L/day)	241.475	241.475	161.55	161.55
Mean (predicted, L/day)	243.163	246.436	161.81	165.315
MAE (L/day)	18.34	41.16	11.36	30.09
MB (L/day)	1.688	4.961	0.2575	3.7647
RMSE (L/day)	23.64	66.47	15.33	40.92
RMSE _S (L/day)	8.74	28.72	2.54	17.48
RMSE _U (L/day)	21.96	59.94	15.12	36.99
PSE	0.1583	0.2296	0.02811	0.2232
IA	0.9957	0.9637	0.9961	0.9698
FB	-0.00696	-0.02034	-0.00159	-0.02304
FV	0.03982	0.09768	0.01288	0.09144
FA2	0.9699	0.9456	0.9942	0.9432

^a Fuzzy-logic model.

^b Non-linear regression model.

1840 mg CaCO₃/L, and the average increase in alkalinity across the UASB reactor was determined to be about 1193 mg CaCO₃/L (Fig. 8c). Moreover, the alkalinity increase in the UASB reactor averaged about 0.22 mg CaCO₃/mg TCOD_{removed} in the testing set. The

testing data also revealed that the average effluent VFA:alkalinity ratio was about 0.12, which was less than 0.4 as previously observed in the learning set.

Finally, in order to describe the overall performance of the proposed models, results were assessed with various descriptive statistics such as coefficient of determination (R^2), correlation coefficient (R), mean-absolute error (MAE), root mean-square error (RMSE), systematic and unsystematic RMSE (RMSE_S and RMSE_U, respectively), index of agreement (IA), mean bias (MB), fractional bias (FB), the factor of two (FA2), fractional variance (FV), intercept (a) and slope (b) of the adjusted line between observed and predicted values, and proportion of systematic error (PSE). Detailed definitions and calculations of these estimators can be found in several studies [19,26,81–84]. The obtained results are summarized in Table 5.

The obtained PSE, IA and MB values were in line with those reported by others [18,78]. Present FB and FV values were also in agreement with the values reported by Agirre-Basurko et al. [82]. However, it is noted that differences between the present results and other findings may be ascribed to the characteristics of studied input vectors and mean-squared error performance index, as well as to non-linear nature of the problems. As seen in Table 5, descriptive performance indices such as MAE, RMSE, FV, revealed that the fuzzy-logic-based model produced smaller deviation and exhibited a superior predictive performance on forecasting of both biogas and methane production rates compared to non-linear regression model. The value of determination coefficients ($R^2 = 0.9847$ and 0.9848) indicated that only 1.53% and 1.52% of the total variations were not explained by the fuzzy model in prediction of biogas and methane production rates, respectively. However, for the non-linear regression model, about 12.79% and 10.65% of total variations did not fit the experimental data in estimation of biogas and methane production rates, respectively. Results showed that the non-linear regression did not yield satisfactory predictions of gas production rates as good as the fuzzy model. This can be attributed to the advantage of artificial intelligence-based models on complex interactions between multi-input and output variables in a complex system, such as anaerobic digestion process. The linear regression between the fuzzy-logic testing outputs and the corresponding targets indicated that the forecasted data were obviously agreed with the experimental data compared to non-linear regression model. Consequently, it can be concluded that the proposed MIMO fuzzy-logic model can be a good alter-

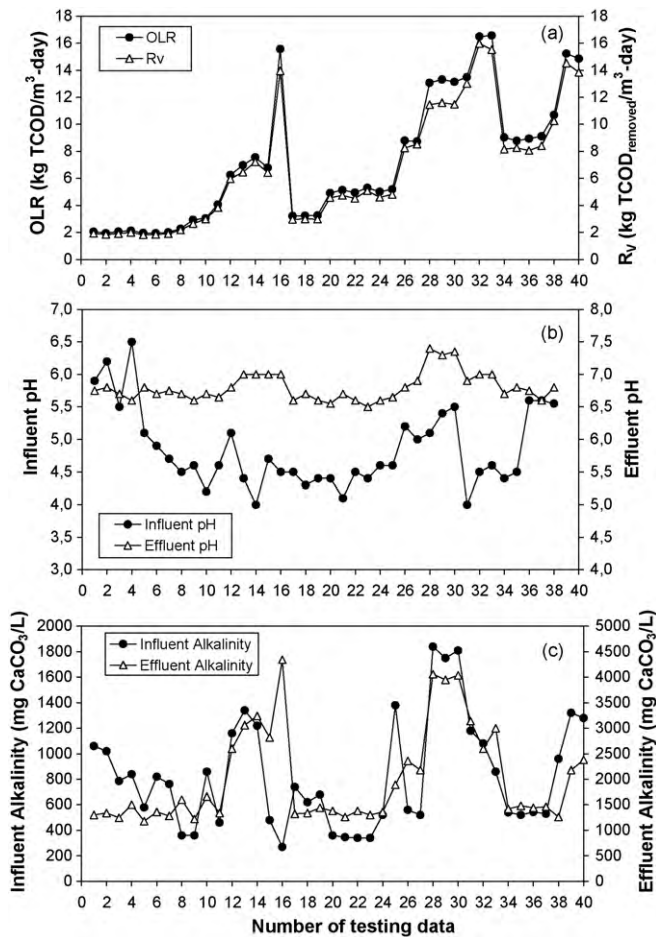


Fig. 8. Variations of other variables in the testing set; (a) organic loading rate and volumetric TCOD removal rate, (b) influent pH and effluent pH, and (c) influent and effluent alkalinity.

native to the conventional multiple regression-based method due to its ability to precisely discriminate the arbitrary non-linear functional relationships without requiring a mathematical model to define complex biochemical reactions between input and output data sets.

4. Conclusions

The pilot-scale UASB reactor showed a noticeable performance on the treatment of molasses wastewater under various organic and hydraulic loading conditions. TCOD removal efficiencies ranged between 84% and 98%, and high volumetric TCOD removal rates (R_V) ranging from 1.85 to 15.97 kg TCOD_{removed}/m³-day) were achieved. TCOD mass balance revealed that over 92% of influent organic matters imposed to the system were transformed to biogas on average. Although relatively high OLRs (1.95–16.56 kg TCOD/m³-day) were imposed to the system, no process failure was observed.

On the basis of the experimental findings, a real-world modeling study was conducted as an important objective to develop an artificial intelligence-based model that could make a reliable prediction on both biogas and methane production rates. For five different model components (OLR, R_V , ALK_{inf} , pH_{inf} , pH_{eff}) the proposed MIMO fuzzy-logic model showed precise and effective predictions with satisfactory correlation coefficients over 0.96. Moreover, two exponential non-linear regression models were also developed as the best-fit models to appraise the performance of the UASB reactor treating molasses wastewater by means of biogas and methane production rates. Non-linear regression variable results showed that R_V and effluent pH had more importance than other model components in prediction of both biogas and methane production rates.

Descriptive performance indices clearly indicated that the proposed MIMO fuzzy-logic-based model showed a superior predictive performance on forecasting of both biogas and methane production rates compared to non-linear regression model. The applicability of the fuzzy-logic model is very simple and there is no need to define the complex reactions and their mathematical or biochemical equations. Moreover, due to highly non-linear structure of the fuzzy-logic model model, it was shown that a complex system such as anaerobic digestion could be easily modelled. Since fuzzy-logic methodology gave encouraging estimation results for the online control of a pilot-scale system, it is believed that this kind of model will help the control engineer to evaluate in real-time production rates that are necessary to control the anaerobic process and to establish fault diagnosis before transferring the concepts to a full scale plant.

On the basis of the advantages of artificial intelligence-based modeling approach, for future studies, an improved MIMO fuzzy-logic-based model, including additional inputs and outputs, will be developed to estimate parameters that are not measured on-line in the process, as well as to evaluate the effects of unexpected input changes on the outputs. Furthermore, different types of membership functions and their combinations will also be tested to enhance the prediction performance of the proposed diagnosis system based on fuzzy-logic.

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