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Two-Phase Anaerobic Digestion Model of a Tannery Solid Waste: Experimental investigation and Modeling with ANFIS

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Abstract Anaerobic digestion of limed fleshing solid waste from leather-processing industry and effluent treatment liquid waste was studied in a two-phase digester. The hydrolysis and acidification stages resulted in the formation of volatile fatty acid (VFA), where the maximum concentration of total VFA observed in the acidogenic reactor was 18,225 mg/L and the average VFA concentration was 10,574 mg/L for hydraulic retention time (HRT) of 10 days. The hydrolyzed predigested material was tested in a methanogenesis reactor for biogas generation at HRT of 20 days. The two-phase process of the digestion system has been modeled using adaptive neuro-fuzzy inference system (ANFIS) with HRT, pH and organic loading rate (OLR) as input parameters and cumulative gas production as output parameter. The average CH_4 production rate over the entire study period was $0.31\,m^3\,CH_4/kg$ VS destroyed and $0.15\,m^3\,CH_4/kg$ VS fed at an overall average OLR of $1.05 \pm 0.05\,kg\;VS/m^3$ day. Modeling with ANFIS using HRT, OLR and pH as inputs and cumulative production of gas as output produced a mean-square error of only 0.00049 which is relatively more accurate with reference to available literature.

Keywords Anaerobic digestion · Hydrolysis · Methanogenesis · Biogas · ANFIS modeling

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1 Introduction

Limed and sulfide fleshing solid wastes are formed in plenty in tannery which results in environmental hazard because of their high putrid and biodegradable nature. Anaerobic digestion process is the best way to dispose these wastes [1,2]. Anaerobic digestion is the biological degradation by complex microbial ecosystem of organic and occasionally inorganic substrates in the absence of an oxygen source. During the process, organic material is converted to mainly methane, carbon dioxide and biomass. The nitrogen not utilized in growth is generally released as such or reduced to ammonia. Treatment of solid waste using anaerobic technology is significantly dependent on advancements in high-rate anaerobic bioreactors [3,4].

Anaerobic systems can be operated as single-phase or two-phase systems, and single-phase systems involve only one reactor for the microorganisms to digest the organic matter, whereas two-phase systems separate the hydrolysis and acidogenic and methanogenic organisms into two separate reactors. Recently, many novel reactors were designed [5], and the difference between the methods is based on the maintenance of microorganisms in bioreactor or by how the acidogenic/methanogenic bacteria are retained in two phases to decrease the limitations of anaerobic digestion processes [6,7]. Bo and Pin-jing [8] has analyzed the performance of two-stage anaerobic digestion system of kitchen wastes using batch experiments.

A key constraint of anaerobic digestion of tannery solid wastes containing biodegradable organics in single-phase system is rapid acidification and larger VFA production resulting in less pH in the reactor, which stressed and decreased the activity of methanogenic bacteria [9]. Smith and Almquist [10] has investigated on anaerobic digestion and evaluated the performance of two-phase reactor system.

Table 1 Operating parameters of various digester systems

Type of digester system	Temperature (°C)	HRT (days)	OLR (kg/m^3/day)	VS destruction (%)	Specific gas production (m^3/kg VS)	Rate of specific gas production (m^3/kg VS/day)
Conventional sewage digesters (displacement type)	Ambient cold digestion	55–75	1.33	97–94	–	–
Standard sewage works digester	Heated	25–30	2.6–3.4			
	35	11–46 (avg. 27)	0.73–3.3	97–94	0.43–0.56	0.017–0.022
High rate	35	15	5.2–11.5			
Optimum sewage		12–15	2.4			
Rowett research	35	10	2.6–3.7	95–94	0.42	0.042
Piggery wastes		7			0.38	0.054
(High rate)		5				
	30	10			0.39	0.039
Auchincruive	Heated	23	3.2	90	0.39	0.017
Imhoff mixed farm wastes	Heated	36	2.8	86–90	0.31	0.008
Gobar gas plant	25–35	28	2.9	91–93	0.24–0.54	0.009–0.02
Fry original experiments. Piggery	35	ca. 60	2.9	86–88	0.47	0.008
Suggested optimum		35–40	2.8			0.012
Biomechanics High rate, sludge—recycle, piggery	Heated	5–10		84–90	0.5	0.05–0.1
Meat packing	33	2	1.3		0.47	0.24
Yeast manufacture		2	1.8		0.25	0.12
Maise starch	23	3.3	1.8		0.44	0.14

Hence, the two-phase system has emerged as competent technology for anaerobic digestion of tannery fleshing. The main benefit is due to OLR buffering that occurs in the first phase and stable loading rate in the methanogenic second phase [11,12]. Table 1 shows the operating parameters of various digester systems used in the literature [1–12].

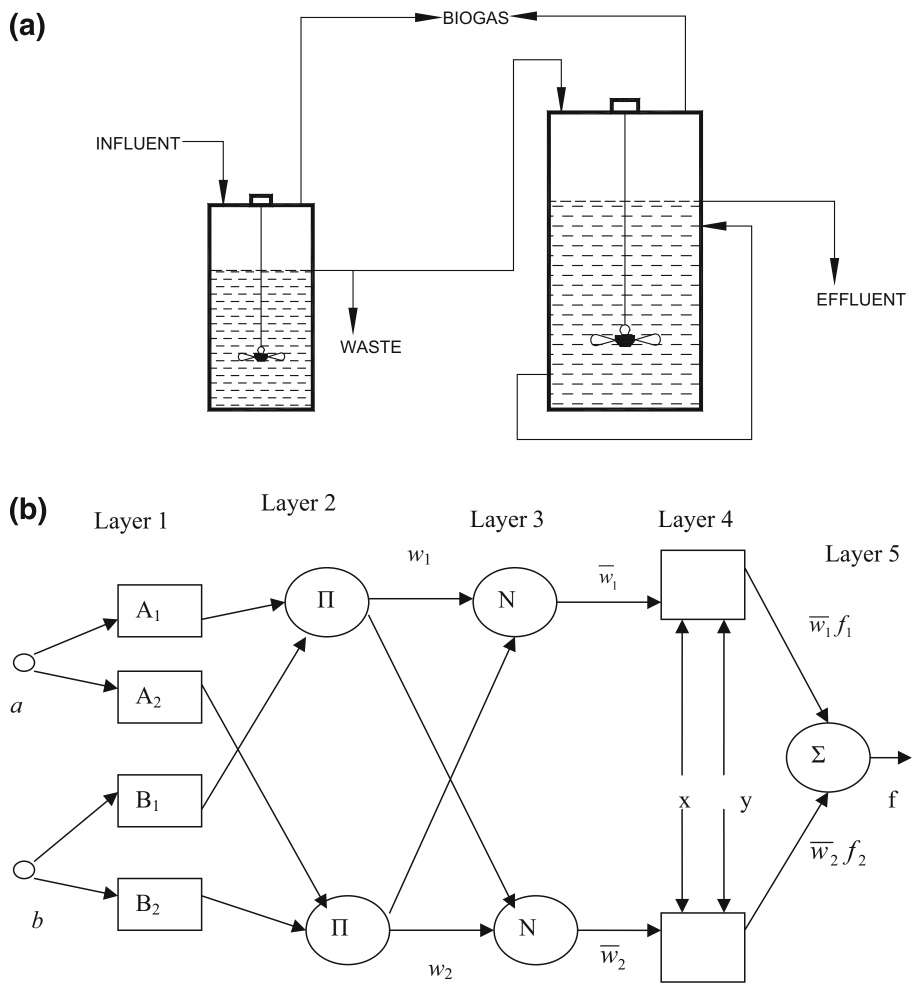
The neuro-fuzzy technique hybridizes neural and fuzzy concepts, where the learning from neural networks helps in mapping its knowledge to fuzzy logic [13]. Variants of neuro-fuzzy algorithms are fuzzy aggregation/inference/modeling networks [14], network-driven fuzzy reasoning, fuzzy associative memory systems etc. Mathematical modeling of anaerobic digestion of biomass waste has been presented in [15]. ANFIS is an important neuro-fuzzy model [13] which is a graphical representation of TSK model with training by gradient descent method. The sigmoid-ANFIS [16,17] uses only sigmoidal membership functions, and ANFIS unfolded-in-time [18] duplicates ANFIS for integrating temporal information. The five layered self-organizing fuzzy neural network [19,20] is an online implementation of TSK [19]. Dynamic evolving neuro-fuzzy inference system [21] is used to perform the prediction where new fuzzy rules are created and updated during the operation of the system. Similarity in fuzzy rule-based systems and artificial neural networks was studied by Zhang [22]. Jang [13] proved that a fuzzy system has similar functionally as radial basis function networks. CANFIS is a scalable version of ANFIS that has multiple inputs [20].This research work analyzes the two-phase anaerobic digestion of tannery solid wastes, and the results are modeled using ANFIS.

2 Materials and Methods

2.1 Design of the Reactor with Operational Conditions

The two-phase system shown in Fig. 1a comprised of two separate reactors for acidogenic phase and methanogenic phase.

Fig. 1 **a** Schematic diagram of two phase system, **b** ANFIS architecture



The acidogenic and the methanogenic reactors were continuously stirred tank-type reactors in series (CSTR), each having total volume of 4 and 10 L respectively. The two-phase system is designed in such a way that the size of methanogenic reactor is larger when compared to the acidogenic reactor as it requires longer HRT. The active volume acidogenic and methanogenic reactor was 3.50 and 6.5 L, respectively. The acidogenic phase was run in the pH range of 7.0–7.4 without buffering system and HRT as 10 days. The acidogenic phase reactor was fed with mixture of tannery solid and liquid wastes in the ratio of 1:1 (wt/wt) with constant weight of 80 g each. Similarly, the methanogenic reactor was run for 20 days of HRT and pH between 7.50 and 8.30. The two-phase systems first reactor was fed with combination of solid and liquid wastes from tannery, and the digested material exhausted from first phase reactor was fed to second reactor (methanogenic reactor).

2.2 Analysis and Fermentation Parameters

Tannery and effluent treatment plant solid waste samples were analyzed once a week for physicochemical parameters. Samples from acidogenic and methanogenic reactors were also analyzed twice a week. The feed and effluent samples from hydrolysis and methanogenesis reactors were analyzed for pH, volatile solids (VS), total solids (TS), total volatile fatty acids (total VFA), in accordance with standard methods [23]. The amount of biogas generated by each reactor was calculated daily by water displacement technique, and biogas methane content was analyzed by both alkali- scrubbing technique and gas chromatography (GC) procedure periodically [23].

2.3 Two-Phase System

This system was operated to estimate the outcome of phase separation on tannery solid waste treatment. In this section, the data collected from both the reactors of the two-phase system are reported, and performance of the each phase of the system has been correlated with the overall performance of the two-phase system.

2.4 Acidogenic Reactor

The acidogenic reactor acidifies the tannery solid and liquid wastes for improving the methanogenic reactor performance by rising TS and VS removal and yield of methane. Sometimes, acidogenic reactor may cause reduction in shock loadings to methanogenic reactor thereby improving two-phase system stability. The acidogenic reactor was batch-fed with tannery solid and liquid wastes and operated for a 10-day cycle period which lasted for 123 days.

2.5 Operation of Acidogenic Reactor

From the literature survey, operating conditions of acidogenic reactor [24] was set after analyzing the acid-phase reactors treating dairy wastewater [25,26], brewery wastewater [27], cheese wastewater [28]. Most literature analyzed the effects of pH, HRT and temperature on substrate degradation, acidogenic conversion degree from VS to VFAs, variations in the sharing of VFAs formed and yield of methane and hydrogen. It was found that acidification and biodegradability degree of carbohydrates, lipids and proteins in industrial solid wastes increased with HRT [24].

The acidogenic reactor was operated at a HRT of 10 days which was chosen because of high organic content (average VS was 10.4 g/L) of solid waste, and the treated wastewater was compared with the studies reported in the literature for other solid waste. The pH in the acidogenic reactor was in the range 7.0–7.4. The average composition of limed fleshing, primary sludge and inoculum used in batch experiments provided in Table 2.

2.6 ANFIS Architecture

A fuzzy inference system (FIS) encompasses fuzzy set theory, fuzzy if-then rules and fuzzy reasoning and is used in modeling real-time systems. The parameterized nonlinear map [13] f can be represented as

Table 2 The average composition of limed fleshing, primary sludge and inoculum used in the batch experiments

Constituents	Limed fleshing	Primary sludge	Pre digested sample (inoculum)
pH	12.10	7.41	8.00
Total solids (TS)	13.37 %	6.98 %	2.45 %
Volatile solids (VS)	55.60 %	35.24 %	35.57 %
Oil and grease (crude lipid)	4.79 %	2.57 %	10.1 %
Protein (crude)	56.5 %	28.39 %	34.47 %
Volatile fatty acid (as acetic acid equivalent)	–	1,845 mg/L	360 mg/L

$$f(x) = \frac{\sum_{l=1}^{m} y^l \left(\prod_{i=1}^{n} \mu_{A_i^l}(x_i)\right)}{\sum_{l=1}^{m} \left(\prod_{i=1}^{n} \mu_{A_i^l}(x_i)\right)} \quad (1)$$

y^l is output singleton depending on Mamdani or Sugeno reasoning [17] is applied. The ANFIS architecture [13], with 2 rules is presented as follows.

$$\text{Rule 1}: \text{if } (a \text{ is } A_1) \text{ and } (b \text{ is } B_1), \quad \text{then } (f_1 = p_1 a + q_1 b + r_1) \quad (2)$$

$$\text{Rule 2}: \text{if } (a \text{ is } A_2) \text{ and } (b \text{ is } B_2), \quad \text{then } (f_2 = p_2 a + q_2 b + r_2) \quad (3)$$

The five layered ANFIS architecture shown in Fig. 1b has a and b as inputs and f as output with fixed node indicated by circle and adaptive node indicated by square.

Layer 1: Adaptive nodes in this layer find the membership function and are represented by

$$O_{1,i} = \mu_{A_i}(a), \quad \text{for} \quad i = 1, 2, \text{ or} \quad (4)$$

$$O_{1,i} = \mu_{B_{i-2}}(b), \quad \text{for} \quad i = 3, 4, \quad (5)$$

Fuzzy set in parametric form is represented as A_i and B_i, and depending on membership function, it is calculated. If bell function [13] is used, then

$$\mu_{A_i}(x) = \frac{1}{1 + \left[\left(\frac{x - c_i}{a_i}\right)^2\right]^{b_i}}, \quad i = 1, 2. \quad (6)$$

a_i, b_i and c_i representing the premise parameters.

Layer 2: The function of fixed nodes is to multiply the input member function, and the output is given by

$$O_{2,i} = w_i = \mu_{A_i}(a)\mu_{B_i}(b), \quad i = 1, 2 \quad (7)$$

Layer 3: Fixed nodes represented as N performs normalization given by

$$O_{3,i} = \overline{w_i} = \frac{w_i}{w_1 + w_2}, \quad i = 1, 2. \quad (8)$$

Layer 4: Adaptive nodes here calculates the product of layer 3, and a first-order polynomial containing consequent parameters p, q, r given by

$$O_{4,i} = \overline{w_i} f_i = \overline{w_i}(p_i a + q_i b + r_i), \quad i = 1, 2. \quad (9)$$

with p_i, q_i, and r_i representing design parameters.

Layer 5: Fixed node here finds summation of all inputs and is given by,

$$O_{5,1} = \sum_i \overline{w_i} f_i = \frac{\sum_i w_i f_i}{\sum_i w_i}, \quad i = 1, 2. \quad (10)$$

During forward pass, premise parameters are fixed and consequent parameters use least-square estimator, while during backward pass, premise parameters use gradient descent and consequent parameters are fixed

3 Experimental Results

3.1 Removal Efficiencies

The profiles of TS and VS contents in the feed and drain of hydrolysis process are shown in Fig. 2a, b, respectively. The average TS and VS concentrations in the feed were 17.68 and 10.4 g/L, respectively, and the average drain TS and VS concentrations were 12.84 and 7.10 g/L, respectively. Because of low solids retention time (SRT) in acidogenic reactor, less solids destruction has occurred in the reactor. SRT was maintained less otherwise at high SRT, more methane production and solids destruction take place.

3.2 VFA Production

The six important VFAs found in effluent were acetic, propionic, butyric (*n*-, *iso*-) and valeric (*n*-, *iso*-) resulting in 95 % of total VFA concentration. Acetic, butyric and propionic acids are important VFAs identified in acid-phase reactors that treats dairy wastewaters [25,26], and according to the operating conditions, the concentrations of VFA varies.

Fang and Yu [25] analyzed the products of two acidogens present in anaerobic digestion systems during acid phase. The first acidogen produced acetate, butyrate, carbon dioxide and hydrogen with second producing acetate, propionate and some valerate with production of less biogas. Both acidogenic microorganisms did not produce ethanol. Later Fang and Yu [25] found a third acidogenesis which produced ethanol, acetate, hydrogen and carbon dioxide at 4.5 pH. Depending on VFA in acidogenic reactor, it can be assumed that all three acidogens were present due to detection of acetic, butyric, propionic and valeric acid. As the presence of carbon dioxide and hydrogen were not checked in biogas and the effluent concentration of alcohols were not quantified, the presence of all types of acidogenic microorganisms cannot be ruled out.

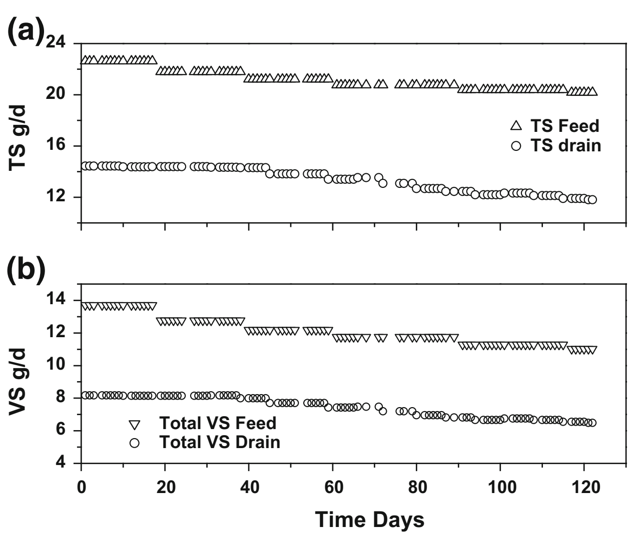


Fig. 2 **a** Total solids content in feed and drain (hydrolysis). **b** Volatile solids content in feed and drain (hydrolysis)

Figure 3 shows the OLR and corresponding total VFA concentration that prevailed in acidogenic reactor effluent during the study. It was noted that the effluent total VFA concentration was sufficiently high to use as feed to the second phase and suggests that the acidogenesis of tannery solid and liquid wastes was rapid for HRT of 10 days. The maximum concentration of total VFA observed in the acidogenic reactor was 18,225 mg/L. The average VFA concentration was found to be 10,574 mg/L. Even though the purpose of acidogenic reactor was to hydrolyze the tannery solid and liquid wastes and generate significantly high VFA concentration in a short HRT, it was found that acidogenic reactor behaved like equalization tank than acid-phase reactor as the fraction of easily biodegradable matter present in the feed is low.

3.3 Biogas and Methane Production

The daily and cumulative gas productions are shown in Figure 4a, b respectively. The acidogenic reactor produced nearly 184 mL of biogas every day, and the methane content was <10 %. Acidogenic reactor was not anticipated to create considerable amount of methane, but the literature shows of 5–15 and 7–27 % of methane in the biogas generated from acidogenic reactor [25,26]. The biogas produced by acidogenic reactor in this research was found to contain methane in the range of 5–10 % and the balance composed of mainly carbon dioxide and very low concentration of hydrogen. The average specific gas production is 0.02 L/g VS fed.

3.4 Methanogenic Reactor

The operation of acidogenic reactor helped in growth of acid-forming microorganisms, and methanogenic reactor helped in methane-forming microorganisms growth. The products generated from acidogenic reactor was batch-fed to methanogenic reactor and was operated on a 20-day cycle for 123 days. The reactor pH varied from 7.6 to 8.3 with average pH of 7.7 ± 0.5 throughout the study.

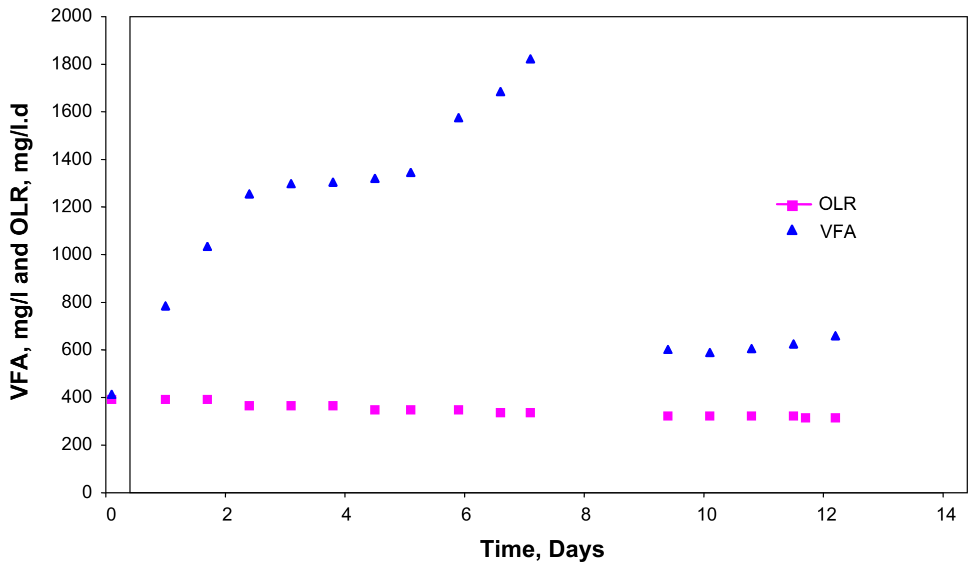


Fig. 3 Variation of OLR and VFA concentration (hydrolysis)

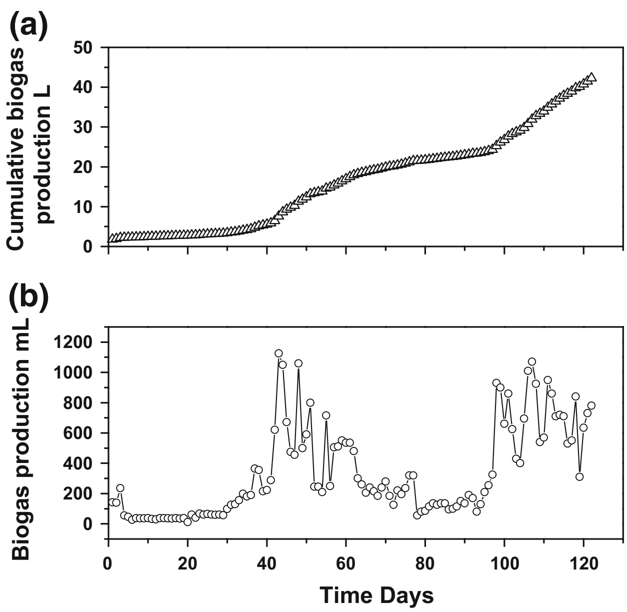


Fig. 4 **a** Cumulative biogas production (hydrolysis). **b** Daily biogas production (hydrolysis)

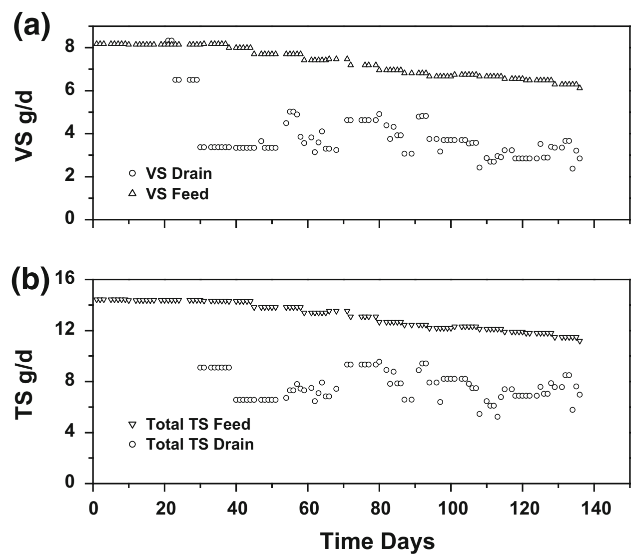


Fig. 5 **a** Volatile solids content in feed and drain (methanogenesis). **b** Total solids content in feed and drain (methanogenesis)

3.5 Methanogenic Reactor Operation

The methanogenic reactor was operated at constant feed volume, where no significant variation in OLR was observed and the feed volume was set based on concentration of VS in the drain from acetogenic reactor. The methanogenic reactor HRT was maintained at 20 days.

3.6 Removal Efficiencies

The concentrations of effluent, influent and performance with TS and VS removal percentage during the period of study is presented in Figure 5a, b. It appears that reasonably high TS and VS removal rates were achieved. Under steady-state operation, the average removal efficiencies of TS and VS were 40 and 46 % respectively.

3.7 Biogas and Methane Production

Initially, VFA was gradually increasing and reached a steady state after 40 days of operation. The average concentration of VFA in the reactor during the study period was 8,987 mg/L. There is a steady state observed beyond 40 days, indicating a dynamic balance between VFA conversion and biogas generation and stabilization of the methanogenesis process.

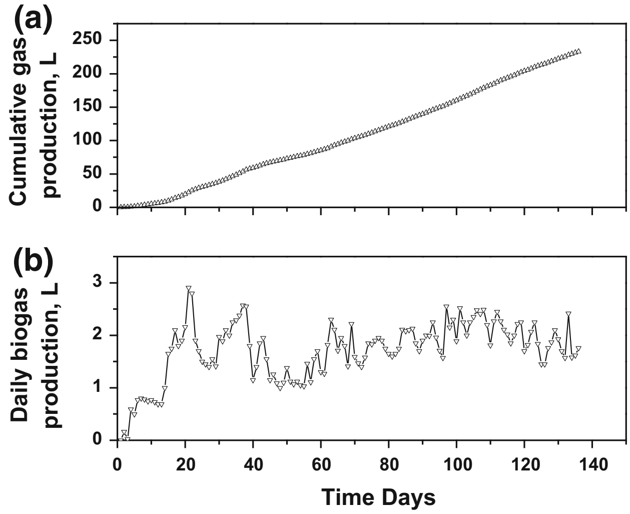


Fig. 6 **a** Cumulative biogas production (methanogenesis). **b** Daily biogas production (methanogenesis)

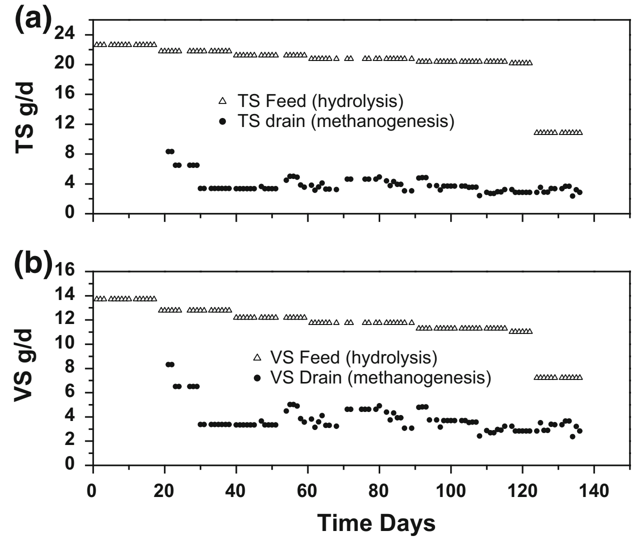


Fig. 7 **a** Total solids content in feed (hydrolysis) and drain (methanogenesis). **b** Volatile solids content in feed (hydrolysis) and drain (methanogenesis)

Biogas yield obtained from a fed of raw cheese whey wastewater by Ghaly et al. [29] was 0.05 and 0.1 m^3/kg VSS added from methanogenic reactor of two-phase anaerobic digester. While treating synthetic whey substrate in upflow anaerobic filter, Yilmazer et al. [28] obtained a biogas production rate of 0.55 m^3/kg COD removed. Strydom et al. [30] evaluated the performance of two-phase anaerobic hybrid reactor for treating cheese, milk and butter factory wastewaters obtaining CH_4 production rates of 0.36, 0.33, and 0.29 m^3/kg COD removed, respectively. The methane yield obtained by Lo et al. [31] was 0.15 and 0.17 m^3/kg VS added treating industrial wastewater in anaerobic rotating biological contactor (ARBC) reactor. From the literature, biogas methane percentage from methanogenic reactor was found to be 52, 67 and 75 % [25,27,29].

The daily gas production and cumulative gas production for the entire study period are shown in Fig. 6a, b, respectively. The methanogenic reactor produced an average of 1.152 L of biogas/day, with an average methane content of 66 %. The pH of the reactor was found to range from 7.6 and 8.2. During the entire study, the average methane production rate was 0.31 m^3 CH_4/kg VS destroyed and 0.15 m^3 CH_4/kg VS fed at an overall average OLR of 1.05 ± 0.05 kg VS/m^3 day. This methane production rate observed in the present investigation resembled the observation reported by Strydom et al. [30]

3.8 Two-Phase Systems Overall Performance

The aim of two-phase systems was to improve the anaerobic biodegradation of tannery solid and liquid wastes by restricting the hydrolysis and acidogenic reactions to systems during first phase and methanogenic reactions during second phase. The tannery solid and liquid wastes were given to the acidogenic reactor, and its drain was given to methanogenic reactor. Acidogenic reactor was expected to decrease the shock loadings to methanogenic reactor as it would behave like equalization tank.

The variations in TS and VS in the feed to first phase and the drain from the second phase are shown in Fig. 7a, b respectively. The average TS and VS removal efficiencies are 65 and 67 % respectively. The cumulative gas productions and daily gas production for the entire study period are shown in Fig. 8a, b respectively. The specific gas production is found to be 0.14 m^3/kg VS fed and 0.21 m^3/kg VS destroyed.

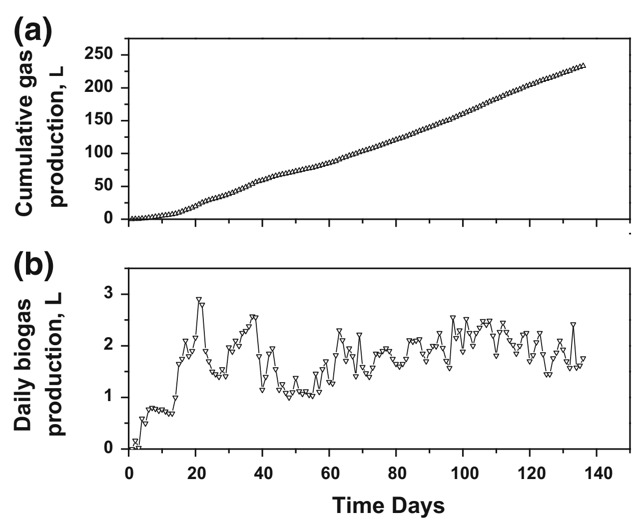
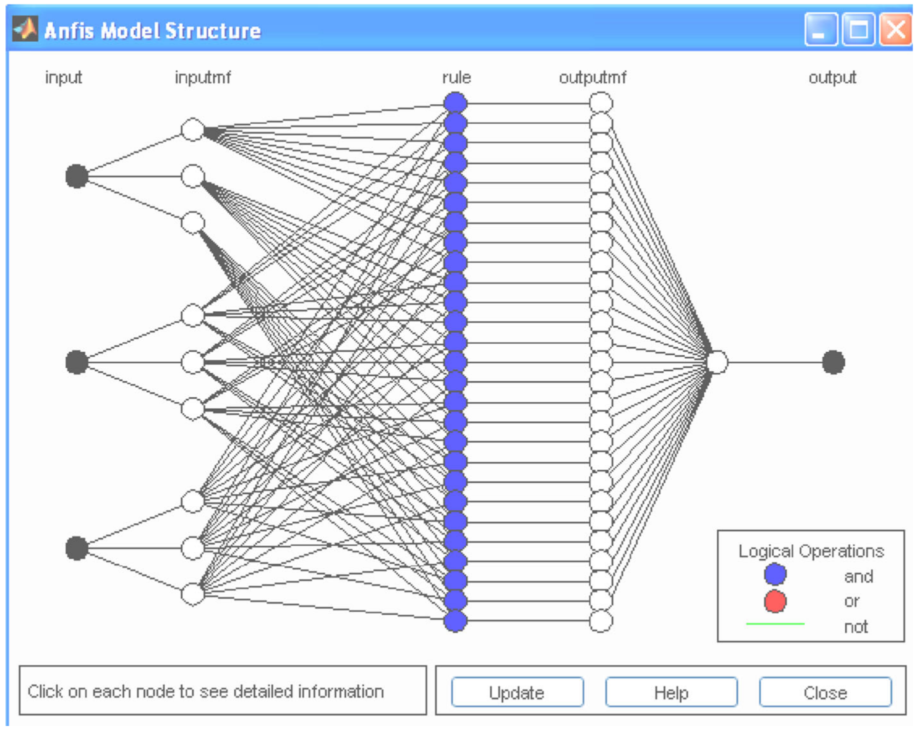


Fig. 8 **a** Cumulative biogas production (hydrolysis + methanogenesis). **b** Daily biogas production (hydrolysis + methanogenesis)

Fig. 9 ANFIS model structure



4 Modeling Using ANFIS

ANFIS [13] uses the learning capability of neural networks [14] and human-like decision-making capacity of fuzzy systems to find the membership function parameters of FIS. A mixture of back propagation gradient descent methods [14] and least squares are utilized for training FIS, which is later, used to model a given set of input/output data. Various hybrid stable learning approaches of ANFIS was presented by Shoorehdeli et al. [32].

For modeling with ANFIS, pH, HRT and OLR are taken as input parameters and cumulative production of gas is the output parameter. Using MATLAB 7, adaptive neuro-fuzzy modeling has been done and the evolved ANFIS model structure is shown in Fig. 9, which has the following layers.

input: HRT, pH and OLR are the three input nodes in this layer which are adaptive. Sigmoid MF has been used for the evolved model.
inputmf: Nodes which are fixed are used in this layer, and it is a simple multiplier of MFs.
rule: Fixed nodes here produce the firing strength normalization.
outputmf: Adaptive nodes in this layer has output, which is the result of the first-order polynomial and normalized firing strength.
output: The fixed node in this layer adds up the outputs from layer 4.

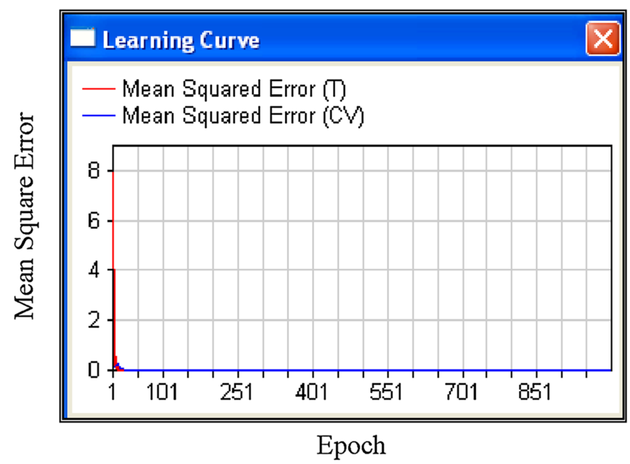


Fig. 10 Learning curve of network error convergence of ANFIS

For the ANFIS model generated, the design details are:

Total quantity of nodes: 78.
Total quantity of linear parameters: 108.
Total quantity of nonlinear parameters: 27.
Total quantity of training data pairs : 232.
Total quantity of fuzzy rules: 27.

Twenty percent of the data have been used for generalization protection. Figures 10 and 11 shows the learning curve and the output versus desired plot for cumulative gas production, respectively. From Fig. 10, it can be inferred that learning in ANFIS happens with less number of epochs. From Fig. 11, it can be inferred that the experimental value and

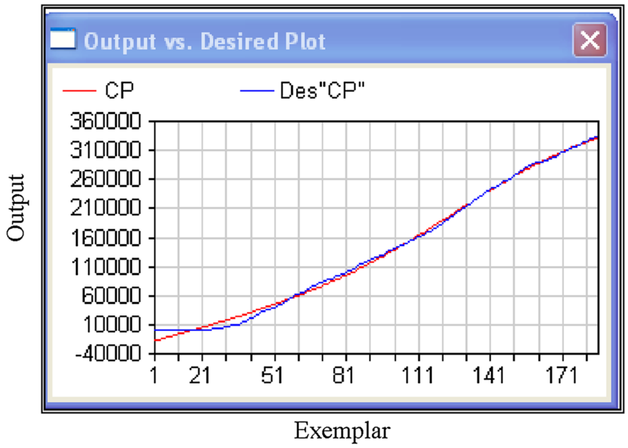


Fig. 11 Output versus desired plot for cumulative gas production

ANFIS predicted value of cumulative gas production almost overlaps with less error value which shows the modeling efficiency of ANFIS.

5 Conclusions

It is observed that the specific gas production obtained in the second phase (0.23 m^3/kg VS added) in the present study is in reasonable agreement with performance reported by Ghaly et al. [29] and Lo et al. [31]. The methane content of 66 % in the biogas is also in good agreement with the values reported by Ghaly et al. [29], and Ince [26]. The higher methane content in the second phase indicates the presence of highly active methanogens in the reactor and the dynamic balance between VFA conversion and biogas generation rate at an OLR of 1.05 ± 0.05 kg VS m^3 day.

According to Fricke et al. [33], protein-rich residues with C:N ratio below 10 can be treated only in two-phase process. In the present study, the fleshing material rich in protein and C/N ratio of 3 has been anaerobically digested in two-phase process with an OLR of 3.47 g VS/L day, and the overall VS conversion efficiency of 67 % has been achieved. The co-digestion of protein-rich solid wastes having C/N ratio of 3 with liquid waste used as diluents minimizes ammonia toxicity in two-phase process as the bacteria cultures in the two-phase process are more robust against high ammonia concentration. Hence, it is concluded that the two-phase digestion system can be operated with protein-rich substrates at higher loading rates without any process instabilities of fatty acid accumulation.

The whole process with three inputs pH, HRT and OLR and cumulative gas production as output has been modeled using ANFIS with mean- square error of 48.935563×10^{-5} which shows the effectiveness of ANFIS modeling.

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