Environmental Research and Technology
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DOI: https://doi.org/10.35208/ert.1232198

Environmental Research & Technology

Research Article

Treatment of citrus juice process wastewater with UASB and biogas production

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ARTICLE INFO

Article history
Received: 10 January 2023
Revised: 23 March 2023
Accepted: 26 March 2023

Key words:
Biogas; Citrus juice process wastewater; COD; Methane; Organic loading rate; UASB reactor

ABSTRACT

A lab-scale up-flow anaerobic sludge blanket (UASB) reactor was used for biogas production from the citrus juice process wastewater (CPWW). The volume of the reactor was 11.5 L. During 200 days of the reactor, the organic loading rate (OLR) value changed from 1.8-21.9 kgCOD./m^3.d, upflow velocities (Vup) 0.1–5.2 m/h and hydraulic retention time (HRT) changed from 0.042 to 4.16 days. The reactor showed a stable performance at all organic loadings. Experimental chemical oxygen demand (COD) removal efficiencies were 71.5±21% and 83.3±16.3% for total COD (TCOD) and soluble COD (SCOD), respectively. The acetic acid concentration changed from 135 to 650 mg/L. The temperature was kept in the range of 35.1±1.4 °C, the pH in the range of 6.6±0.2, and the alkalinity was controlled daily and kept in the range of 411±273 $CaCO_3$ mg/L. After anaerobic reactions, 6283±3476 m^3/d biogas was produced and the methane concentration in the biogas was 65.5±11.5%. Depending on the methane production, the annual energy value potential that can be obtained from the existing UASB reactor is estimated as 48,768 kWh.

Cite this article as: Durak SG, Acarer S, Türkoğlu Demirkol G. Treatment of citrus juice process wastewater with UASB and biogas production. Environ Res Tec 2023;6:1:68–77.

INTRODUCTION

The fruit juice processing industry consists of 3 parts: the clear part where fruits such as apples and cherries are processed, the blurred part where fruits such as peaches and apricots are processed, and the citrus part where fruits such as oranges, lemons, tangerines, grapefruit, limes, bergamot, and lemons are processed [1, 2]. It is common for the citrus line to consume large quantities of water, and wastewater is produced in direct proportion to this consumption. In the citrus juice process facilities, water is used in many stages such as during the transportation of the products, washing, rinsing, pressing the fruits, washing the glass bottles, container washing, filtration, and grinding of the juice [3]. During all of these processes, wastewater is produced in large quantities. The continuous use of clean water at various stages of citrus processing is common. However, the total amount of wastewater is more than 10 times the volume of citrus juice produced [4–6].

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This paper has been presented at Sixth EurAsia Waste Management Symposium (EWMS 2022)/Istanbul, Turkiye / 24–26 October 2022.

In general, CPWW can be thought of as dilute solutions of citrus juice. Wastewater from citrus processing has a high water (80–90%) and organic matter (95%) content [7]. Numerous treatment processes have been applied for citrus processing waste and wastewater from the past to the present. These include ponding and chemical flocculation, spreading, spray irrigation, artificial aeration, yeast production, anaerobic ponding, trickle filtration and activated sludge, anaerobic digestion, and membrane filtration [8]. Due to its high organic matter content and the growth of the microbial mass, biological treatment has been applied in the treatment of CPWW in recent years [9]. Because treatment plants based on physical and chemical treatment are expensive and rarely reliable and efficient, it has limited use for CPWW. For example, the removal of CPWW by evaporation creates a high energy requirement. A disadvantage is the removal of dissolved and total suspended solids (TSS) by flocculation and the high cost of flocculation materials as well as low removal efficiency. The CPWW's contents make biological treatment suitable [2]. Anaerobic treatment is preferred over biological treatment methods. Facultative and anaerobic microorganisms convert organic materials into carbon dioxide (CO_2) and methane (CH_4) in the absence of oxygen [5]. Also, thanks to anaerobic digestion, there is less sludge production, more energy gain, and, above all, less cost [10]. Biogas produced after anaerobic treatment can be used for steam production in boilers and meets the energy demands of the unit [11].

Factors such as temperature, OLR, alkalinity, pH and HRT affect the performance of the bacteria and the yield of the biogas produced [5]. In particular, OLR and HRT are important factors in the set-up phase, as they allow the decision to be made on the amount of feed to the reactor. These two factors determine the final amount of hydrolysis and methanogenesis in the UASB reactor. OLR is an important parameter that must be kept under control for maximum biogas production and high COD removal [12]. The occurrence of anaerobic reactions at lower HRT is an advantage over other digestion methods [13]. Along with all these advantages, the anaerobic digestion of citrus residues makes it possible to reduce wastewater disposal problems. In addition, since it has a high phosphorus content as a final product, it can be applied directly to the soil as a fertilizer or organic substance. The high rate of methane production compared to other industries is the reason why anaerobic methods are preferred for CPWW [2].

Some anaerobic treatment methods applied for CPWW are two-stage anaerobic sequencing batch reactors [8, 14–16], upflow anaerobic sludge bed reactors [17–19], lab-scale completely stirred tank reactor [20], thermophilic downflow stationary fixed film anaerobic reactor [21], anaerobic hybrid reactor [22], anaerobic sequencing batch reactor [23], lab-scale horizontal flow anaerobic immobilized biomass reactor [24], two-stage anaerobic digestion [25] and pilot-scale upflow anaerobic sludge blanket reactor [26] methods.

Table 1. Citrus juice wastewater characterization

Parameter	Wastewater
SCOD (mg/L)	7.760±0.02
TCOD (mg/L)	12.926±0.09
TKN (mg/L)	29.3±1.3
TP (mg/L)	9.7±0.01
pH	4.64±0.3
VFA (as acetic acid) (mg/L)	27.5±1.7
TSS (mg/L)	1.442±0.05
Conductivity (mS/cm)	1.35±0.02

This study aimed to present the results obtained by anaerobic digestion of citrus process wastewater to achieve high biogas production and COD removal. Moreover, the amount of biogas produced was used to estimate the electrical energy potential.

MATERIALS AND METHODS

Wastewater Characteristics

In this study, treatment efficiency and biogas production performance were investigated by using UASB reactor for CPWW treatment. Wastewater was supplied from a fruit juice production facility with a total fruit processing capacity of 125,000 tons. There is an annual inflow of 75,000 tons of citrus fruit to the facility, and the amount of freshwater entering the facility for production is 720,908 m^3 per year on average. The amount of wastewater coming out of the facility is 127 m^3 per day on average.

SCOD, TCOD and volatile fatty acids (VFAs) (as acetate) concentrations, total Kjeldahl nitrogen (TKN), total phosphorous (TP) and TSS concentrations, pH and electrical conductivity values of CPWW are given in Table 1.

The Experimental Setup

The lab-scale UASB reactor in the study was made of plexiglass, with a diameter of 0.12 m, a length of 1.5 m, and an effective volume of 11.5 L (Fig. 1). There was a gas/liquid/solid separator at the top of the reactor. The gas was collected at the upper point as separated and the gas flow was measured with a wet gas meter. Liquid products, on the other hand, were taken to the collection container by weirs from the upper part (Fig. 1-Part 5). The temperature in the reactor was kept constant in the range of 35.1±1.4 °C.

Operating Conditions

Feed solutions were added to the reactor daily. 3 L of 11.5 L was inoculum and the remaining volume was as citrus juice waste. Alkalinity, SCOD, TCOD, OLR, biogas, methane and TSS were measured once in two days; BOD_5, TKN, TP, pH, VFAs, electrical conductivity and temperature parameters

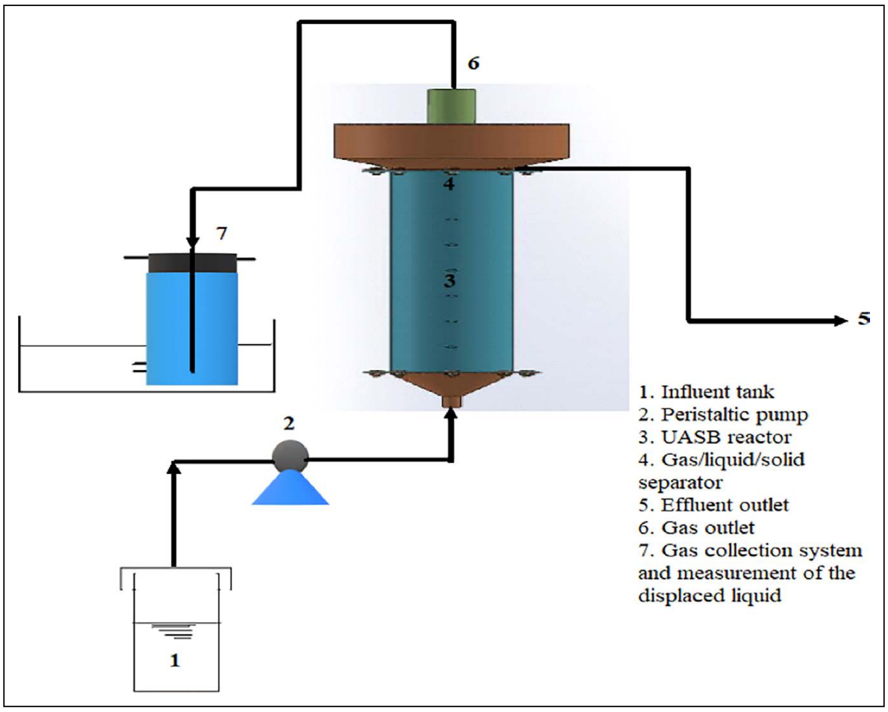

Figure 1. Schematic view of the lab-scale UASB reactor.

were measured as required. The analyzes were carried out to determine the SCOD and TCOD removal efficiency, the amount of biogas produced, and the proportional distribution of the gases in the biogas. The initial HRT value was 0.85 days and the average value of the initial total COD was 7356 mg/L. The OLR varied between 1.8 and 21.9 kgCOD/m^3. d. The alkalinity value at the inlet is 720 $CaCO_3$ mg/L. To keep the COD/N/P ratio around 300/5/1, nutrient was added to the wastewater with ammonium chloride (NH_4Cl) and disodium phosphate (Na_2HPO_4). To provide sufficient alkalinity in the reactor and to buffer CO_2 and volatile acids, 3.3 g/L $NaHCO_3$ was added. According to the alkalinity measurement results, this amount was increased when necessary. A variable-speed peristaltic pump was used to pump feed from a vessel to the reactor every day for 60 days. After the adaptation was achieved, a 200-day operating period was started.

COD analyzes were performed using COD Reactor CR25, DragonLab MX-F, and Spectroquant Colorimeter Picco COD/CSB Merck instruments by EPA 410.4 and ISO 15705 standards [27, 28]. HQ40D Portable pH/Dissolved Oxygen Meter Dual-Channel Multimeter was used for pH and electrical conductivity measurements. TKN, TP, BOD_5 and TSS analyzes were performed using Standard Methods [29].

VFAs parameter was measured by Anderson and Yang [30] method and alkalinity measurements were measured with standard methods 2320 B [31]. Gas measurements were made with the liquid displacement method, 2 days a week during the 200-day operating period. Methane gas was obtained by separating carbon dioxide from biogas by passing it through a liquid containing 3% NaOH, and the amount of methane gas was measured using Draeger X-am® 2500.

Possible Energy Values

The daily produced energy (DPE) that can be obtained from biogas production is calculated by Eq. 1 [32].

$$DPE = C_V \times \%CH_4 \times PB \quad (1)$$

where DPE is the energy produced daily (kJ/d); CV, the theoretical calorific value of methane (35.75 kJ/L) (at 1 atm. pressure and 273 K); The value of methane produced in $\%CH_4$ biogas, PB, is expressed as the measured amount of biogas (L/d) (1 atm. pressure and 273 K temperature).

Eq. 2 is used to calculate the daily energy used (DUE) to produce biogas [32]:

$$DUE = C_P \times \Delta T \times Q \quad (2)$$

where DUE is the energy required for the combustion of biogas (kJ/d); C_p is the specific heat capacity of the wastewater (3.8 kJ/L °C); ΔT is the temperature rise (°C) affecting the reactor due to heating and Q is the average flow rate (L/d) of the UASB reactor.

Heating efficiency is calculated by Eq. 3 [32]:

$$Heating\ Efficiency = \frac{DUE}{DPE} \times 100 \quad (3)$$

To calculate the energy outputs (NEY, kJ/d) in the daily produced energy content, the daily used energy is subtracted from the daily produced energy (Eq.4) [32].

$$NEY = DPE - DUE \quad (4)$$

Table 2. Wastewater characteristics of UASB reactor

Parameter	Unit	Influent	Effluent
SCOD	mg/L	5.466±1.542	610±437
TCOD	mg/L	7.356±1.756	1500±875
BOD5	mg/L	2462.5±374	662±324
TP	mg/L	13.8±0.9	12.9±8.3
TKN	mg/L	51.0±27.0	12±9.1
Temperature	°C	35.1±1.4	33.3±0.7
pH	–	6.6±0.2	6.5±0.5

Table 3. The operation conditions of the UASB reactor

Parameter	Value
OLR (kgCOD/m^3.d)	8.63±5.0
HRT (d)	1.15±0.91
Alkalinity as $CaCO_3$ (mg/L)	411±273
VFA as acetic acid (mg/L)	319±160
Biogas production (m^3/d)	6.283±3.476
Flow (m^3/d)	1.386±868

Eq. 5 is used to determine the energy potential of CPWW [32].

$$EP_{CPWW} = \frac{PB \times \% \, CH_4}{OLR^S_{rem} \times V_R} \times SCOD \times SCOD - RE \times C_V \quad (5)$$

EP_{CPWW}, energy potential (kJ/m$^3_{CPWW}$); OLR^s_{rem} removed dissolved organic loading rate (kg-SCOD /m^3.d), V_R, reactor volume (m^3); SCOD is SCOD entering the reactor (g-SCOD /L_{CPWW}) and SCOD-RE is SCOD removal efficiency (%). Descriptions of other parameters are given in the explanation of Eq. 1.

RESULTS AND DISCUSSION

In the study, using the lab-scale UASB reactor, the treatment of CPWW for 200 days and the biogas content formed by the reactor were monitored. At the end of 60 days, the reactor reached to steady-state, and granular sludge formation started. The characteristics of the wastewater and the operation conditions of the UASB reactor are given in Table 2 and Table 3.

SCOD and TCOD Removal in UASB Reactor

The OLR parameter was initially kept at 7 kgCOD/m^3.d and an attempt was made to operate without reducing it below 4.2 kgCOD/m^3.d (approximately 60%). The OLR increased to OLR of 10 kgCOD/m^3.d approximately in two weeks. According to the results obtained, the effluent TCOD concentrations varied from 41 mg/L to 4,590 mg/L during the monitoring period. The TCOD removal efficiency changed from 51% to 93%, respectively (Fig. 2). SCOD removal concentrations ranged from 20 mg/L to 1,853 mg/L, and SCOD removal efficiencies ranged between 67% and 99.6% (Fig. 3). It is also seen from Figure 2 and 3 that as the influent concentration increases, the removal efficiency increases [33].

Effect of OLR on UASB Reactor

OLR is important for microorganisms to perform their vital activities. Because it is the amount of organic matter required for the growth and reproduction of microorganisms in a system [34]. The treatment efficiency increased with the increase of OLR in many wastewaters. However, in case of an excessive increase, problems such as excessive foaming at the gas-liquid interface in the sludge blanket, its flotation and gas-liquid-solid separator, and accumulation of undigested components arise [35]. In steady-state conditions, the OLR reached 21.9 kg COD/m^3.d, and the TCOD removal efficiency was 54% and the SCOD removal efficiency exceeded 89% [10, 36]. Hajiabadi et al. [37] (2009) states that high OLR provides high TCOD removal. However, in the current study, as the OLR increased, there was a decrease in the removal efficiency of TCOD (Fig. 4). While the TCOD removal efficiency was 90% when the OLR was 1.8 kg/m^3, there was a general decrease in the TCOD removal efficiency as the OLR increased.

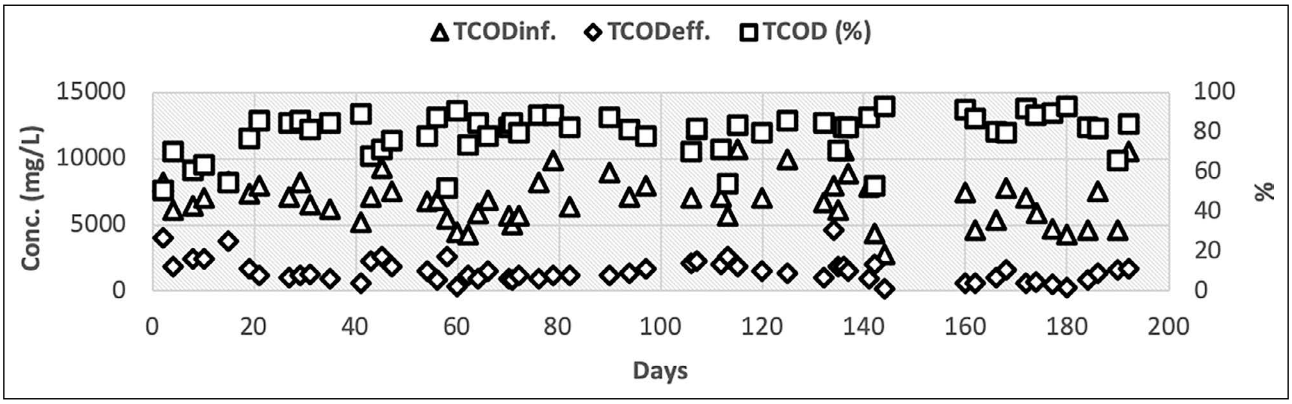

Figure 2. TCOD influent and effluent concentrations, and removal efficiencies.

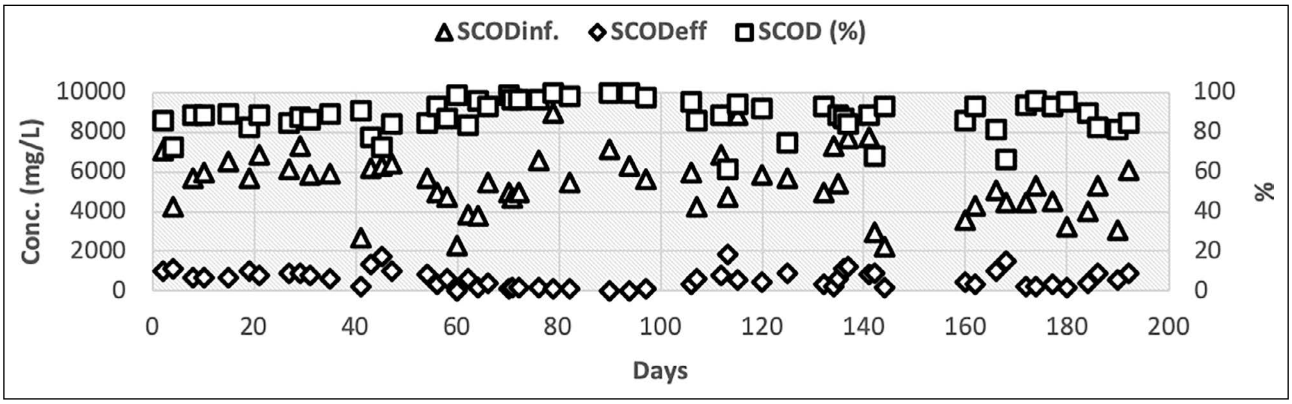

Figure 3. SCOD influent and effluent concentrations, and removal efficiencies.

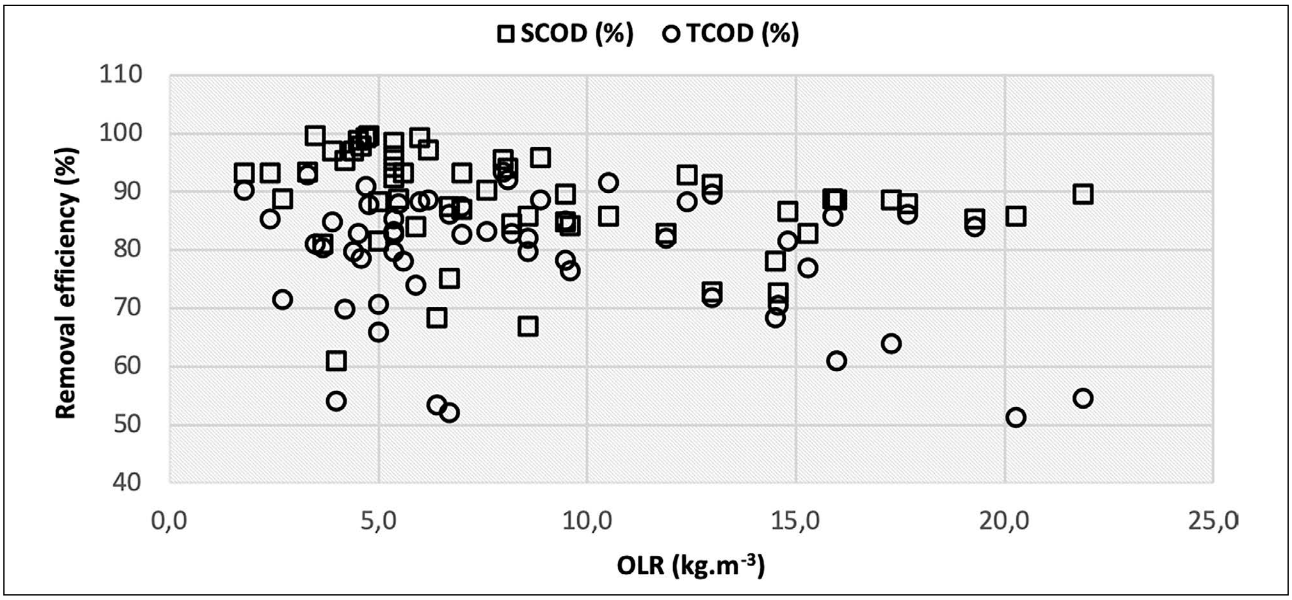

Figure 4. Variation of SCOD and TCOD removal efficiencies according to OLR.

The Effect of Alkalinity on the UASB Reactor

Low pH slows down anaerobic degradation. The VFA concentration is a function of pH. In acidic conditions, the amount of VFAs increases [38]. When Figure 5 is examined, the amount of VFA (acetic acid) is low in conditions where alkalinity is high, and the amount of VFA is high in conditions where it is low [39]. In the study, the amount of propionic acid and butyric acid was ignored as they were in trace amounts.

The Impact of Upflow Velocity on the UASB Reactor

HRT can cause compaction and coalescence of solids in the sludge blanket as a result of its relationship to the V_{up} and the solid's contact time in the reactor [35, 40]. V_{up} is the main factor affecting reactor efficiency. It affects sludge retention and is the basis of the settling characteristic. Increasing the V_{up} increases the collision rate between suspended particles and sludge, and increases the removal efficiency [35, 41–43].

Table 4. Approximate energy values that can be obtained from the UASB reactor

Parameter	UASB reactor
DPE (kWh)	71,715
DUE (kWh)	2,633.4
Heating efficiency (%)	3.67
NEY (kWh)	69,081.6
EP_{CPWW} (kJ/m^3_{CJIW})	3,859

When sufficient contact time between the sludge and wastewater is provided, the biomass is separated from the gas [44]. With the increase in V_{up}, HRT and removal efficiency also decrease [35, 45]. HRT, which was determined as 69.3 hours at 0.1 m/h V_{up} speed, decreased to 9.7 hours when V_{up} reached 1.9 m/h. In addition, as shown in Figure 6, TCOD removal efficiency decreased as HRT decreased [44, 46, 47].

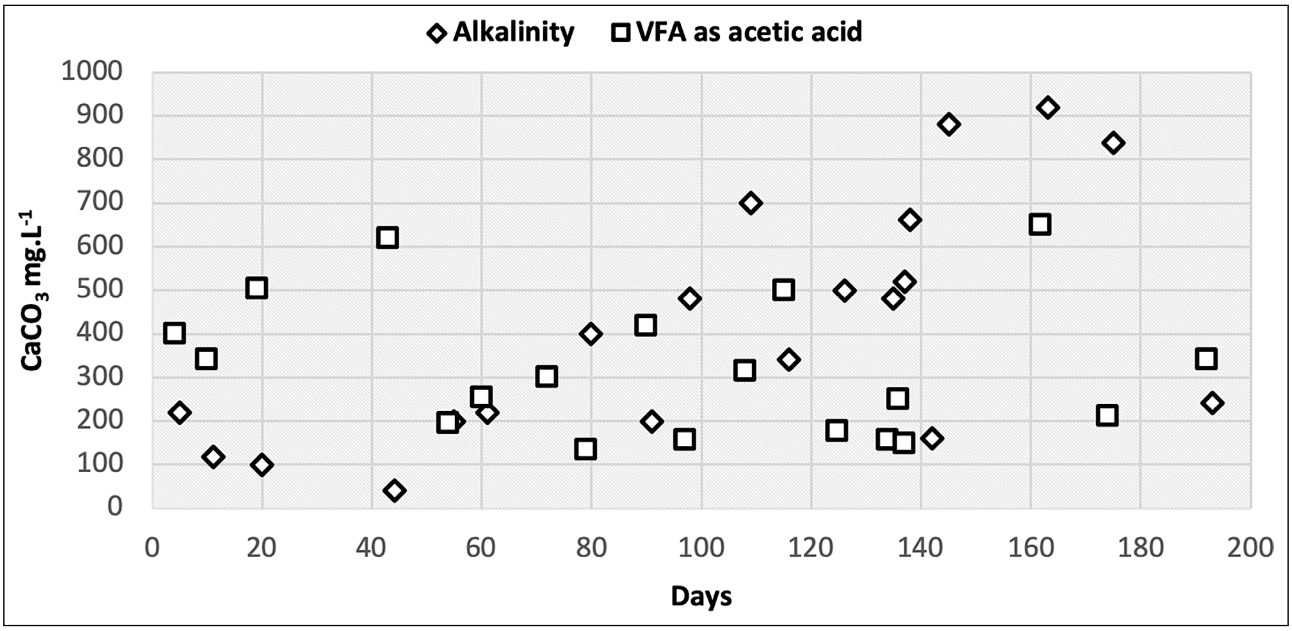

Figure 5. Variation of Alkalinity and VFAs concentration during operation.

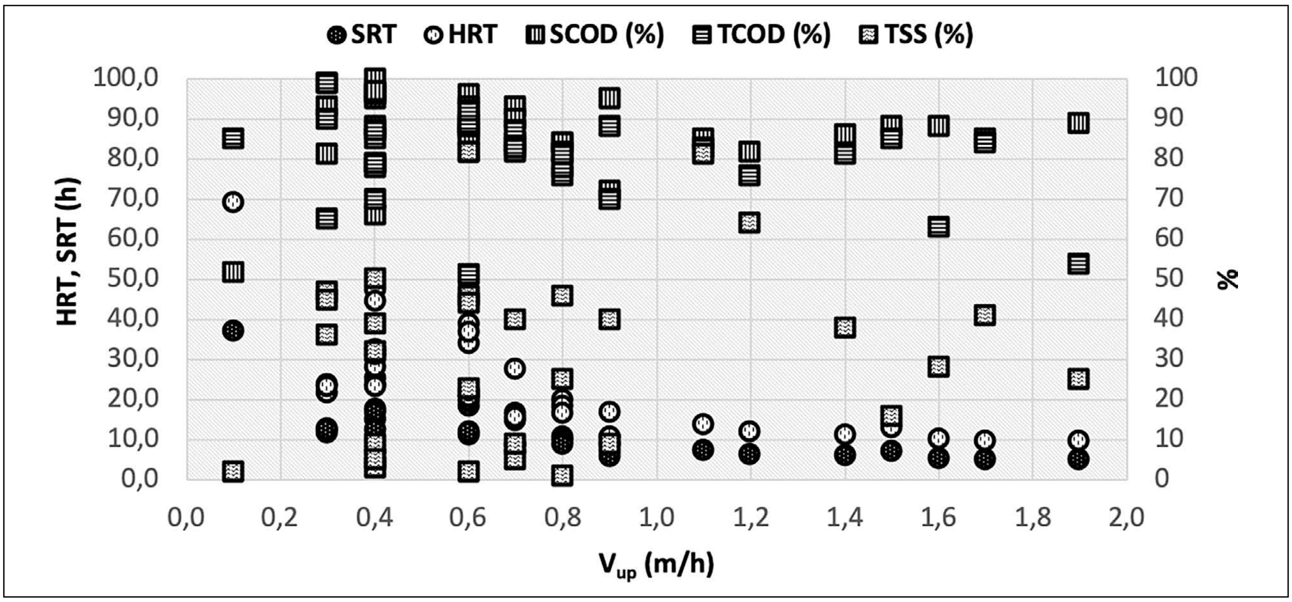

Figure 6. The effect of upflow velocity and variation of HRT and SRT on SCOD, TCOD, and TSS removal efficiencies.

Biogas Production and Composition in the UASB Reactor

Produced biogas values are given in Figure 6 and Figure 7. The biogas production amount is 6283±3476 m^3/d on average. The biogas content consists of methane gas at a high rate (Fig. 7). On average, biogas contains 62% CH_4, 32% CO_2, and traces of H_2S. The studies show similarities with the available data. Accordingly, it is possible to obtain from 54% to 77% methane [47–50] and 30–50% CO_2 [51] from biogas and convert methane into energy.

While there was more biogas production at the beginning, as time passed, a decrease in biogas production occurred. The formation of granular sludge is essential for efficient biogas production. The diameter of the granules is small at the beginning but increase over time. The decrease in biogas production after day 143 can be explained by the increase in granule size. However, the amount of CH_4 in the biogas content is also higher in the initial phase and decreases similar to the decrease in biogas production. When Figure 5 and Figure 7 are examined, it is seen that there is a similar change between VFA amount and biogas production [39].

The amount of biogas production varied between 6283±3476 m^3/d and there was a directly proportional in-

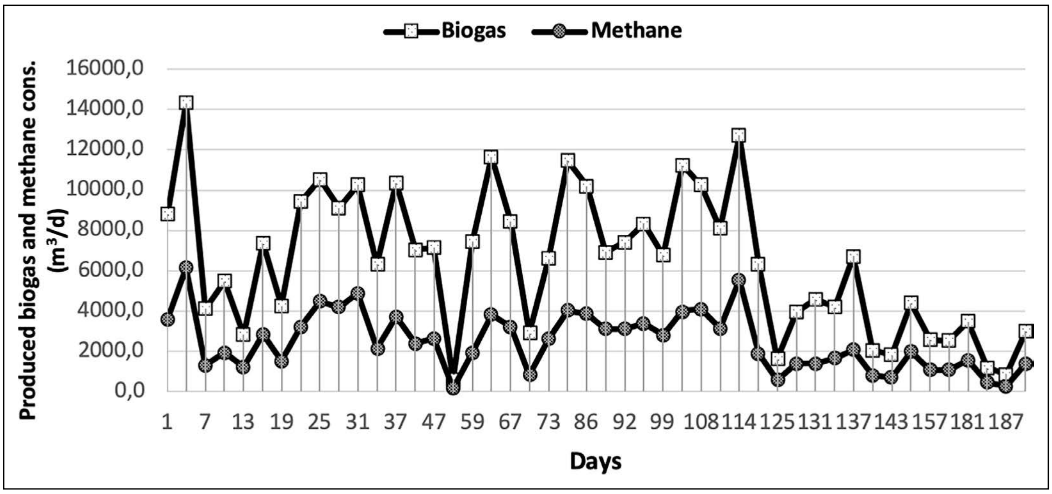

Figure 7. Biogas, and the amount of methane production in biogas' content.

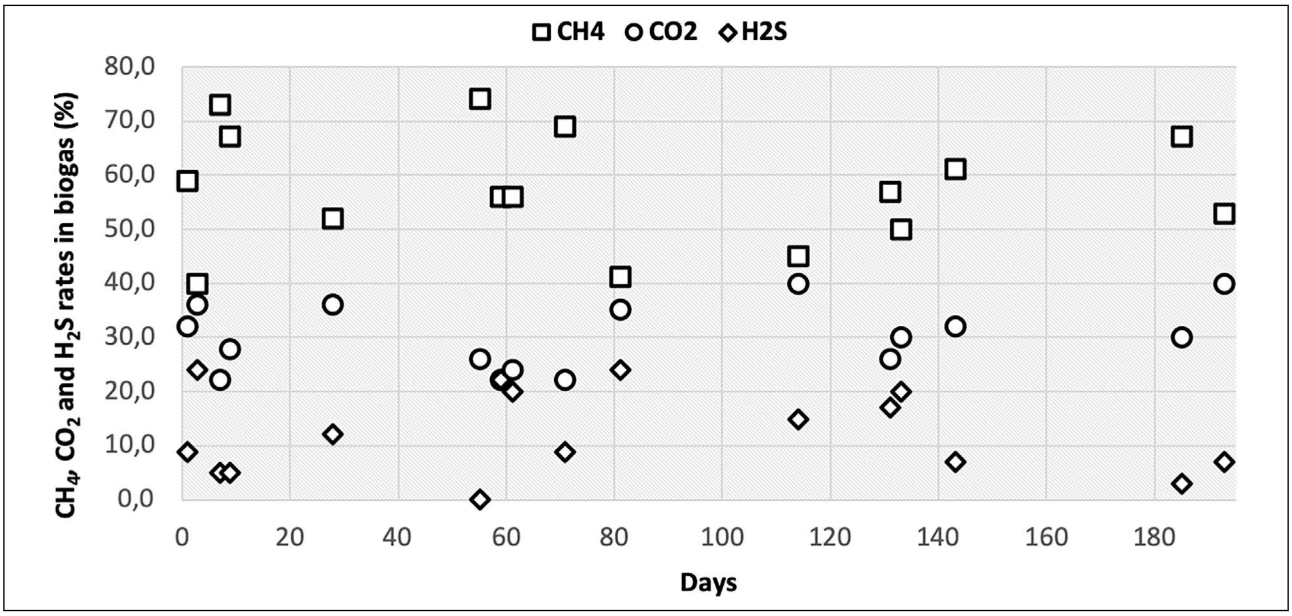

Figure 8. The amount of biogas formation and the percentage of gases.

crease in biogas production as OLR increased (Fig. 9). The linear regression showed a high R^2 coefficient (0.8405).

When calculations are made with the equations given in the possible energy values section, DPE, DUE, Heating Efficiency, NEY, and EPCPWW values are given in Table 4.

The daily energy produced under operating conditions is more than the average daily energy use. The lower the DUE, the higher the heat losses. NEY represents 96.3% of the DPE from the UASB reactor (71,715 kWh). This result shows that the reactor is a useful system. EPCPWW has been calculated considering the operating parameters of the reactor. The increase in temperature increased the EP, accordingly biogas production and anaerobic biomass activity increased. The amount of wastewater released from citrus juice production is approximately 127 m^3 per day (3,800 m^3 per month). The efficiency of a potential power source produces 3,859 kJ/m^3 CPWW (3.85 MJ/m^3 CPWW) can reach 14,630 MJ or 4,064 kWh per month. The annual amount may correspond to 48,768 kWh. In addition, the heat generated while the biogas is converted into electricity can be directed to the reactor, as the efficiency of the reactor will increase if the temperature is high. This increases both the biogas production and the NEY value.

CONCLUSION

With the increase of OLR, biogas production also increased. It is possible to say that the interaction of the particles deteriorates after a certain speed for the V_{up}. HRT and SRT decreased significantly with increasing V_{up} rates.

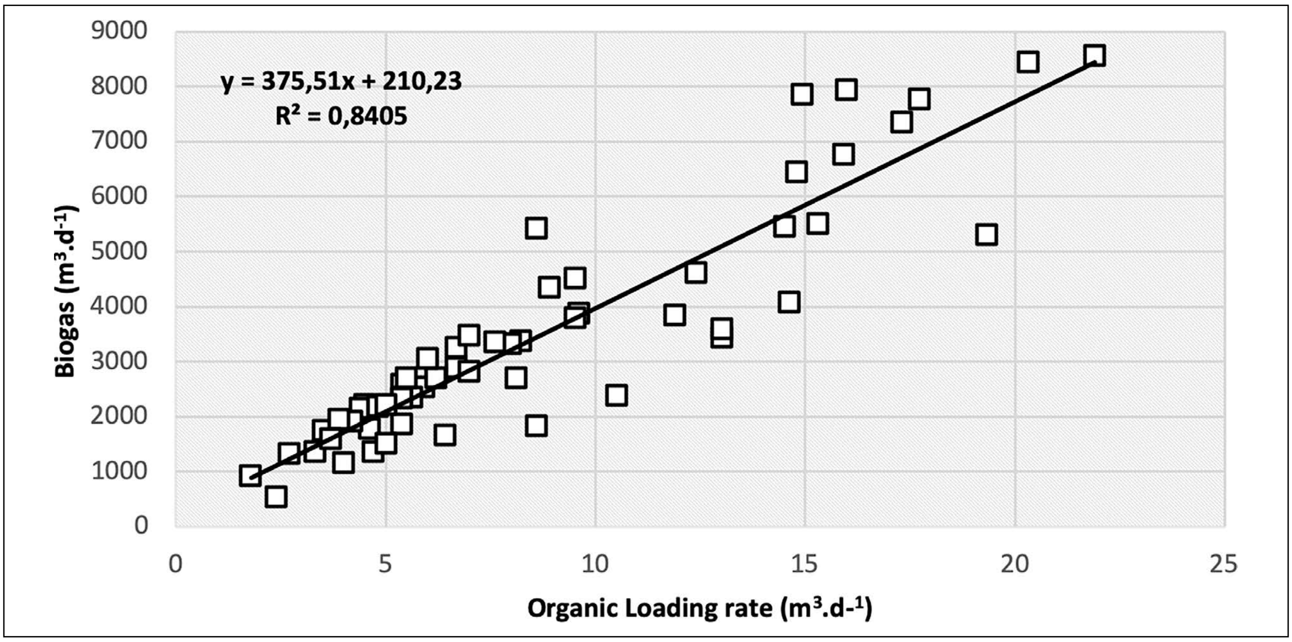

Figure 9. Change of biogas production amount according to organic loading rate.

According to the analysis results, it is possible to say that there is a negative correlation between VFA as acetic acid and alkalinity. When the energy values are examined, it is possible to obtain 48,768 kWh of energy annually from the system. This energy can be used to increase the temperature of the reactor and accordingly increase biogas production. Due to the high organic matter content of citrus juice wastewater, it appears that using UASB reactors is an appropriate option because of their ability to generate energy. In some citrus juice units, increasing the wastewater fed to the UASB reactor and therefore its volumetric capacity can satisfy the required energy needs by generating electricity. Further, considering that the average household consumption of electricity is 238 kWh per month, the energy obtained monthly can serve approximately 17 households. The removal efficiencies of the SCOD and TCOD parameters indicate that the treated wastewater from the UASB reactor can be reused in appropriate sections of the appropriate industries (cooling towers, ash irrigation, flue gas scrubbing, etc.).

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

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