

Enhanced biogas production from anaerobic digestion of wastewater from the fruit juice industry by sonolysis: experiments and modelling

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ABSTRACT

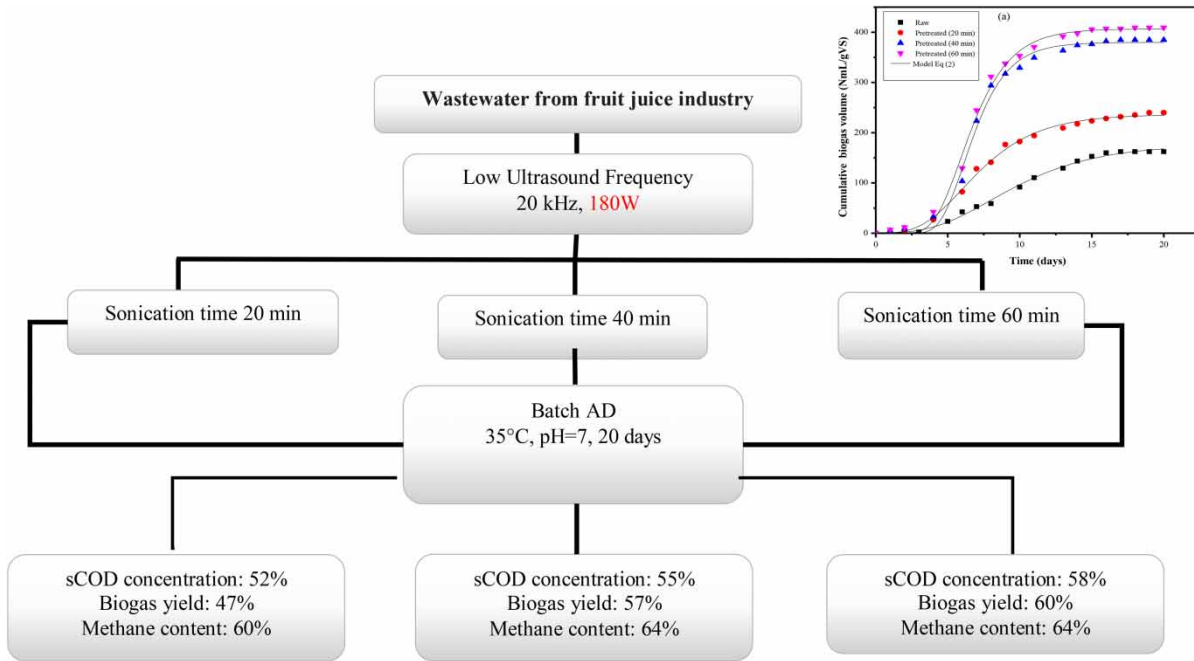
The aim of this study is to investigate the use of ultrasound pretreatment as potential technique to solubilize organic matter and fermentation of fruit juice effluents in anaerobic batch reactor. The efficacy of ultrasound pretreatment has been assessed at low frequency of 20 kHz and at different sonication times (20, 40 and 60 min). Compared with control, the amount of biogas produced increased by 47, 57 and 60% for sonication times of 20, 40 and 60 min, respectively. Methane content of the produced biogas was about 59% in the control and 64% in the case of effluent subjected to ultrasonication for 60 min. After 20 days of anaerobic digestion of the fruit juice effluents, the efficiency of chemical oxygen demand (COD) increased by 9, 31 and 35% with respect to control for sonication times of 20, 40 and 60 min, respectively, corresponding to total sugars uptake efficiency of about 35, 51 and 54%, respectively. The modified Gompertz equation was used to describe the cumulative biogas production. A good agreement was found between simulated and experimental data.

Key words: biogas, fruit juice wastewater, industrial effluents, modelling, pretreatment, ultrasound

HIGHLIGHTS

- Biogas production from fruit juice processing industry.
- Ultrasound pretreatment to improve biogas production.
- High methane content is obtained for prolonged sonication time.
- The COD removal increased by 35% for sonication time of 60 min.

GRAPHICAL ABSTRACT



NOMENCLATURE

$B(t)$	Cumulative biogas production (NmL _{biogas} /gVS)
BOD ₅	Biological oxygen demand (g/L)
COD	Chemical oxygen demand (g/L)
d	Difference between measured and predicted values
E_i	specific energy (MJ/kg TS)
m	Number of data pairs
MS	Mineral solids
OMW	Olive mill wastewater
P	Power (W)
R^2	Correlation coefficient
R_m	Maximum production rate (NmL _{biogas} /gVS .day)
RMSE	Root mean square error
(sCOD)	Soluble oxygen demand (g/L)
T	Temperature (°C)
t	time (h)
TS	Total solids (g/L)
TSS	Total suspended solids (g/L)
US	Ultrasound
V	volume (L)
VFA	Volatile fatty acids
VS	Volatile solids (g/L)
VSS	Volatile suspended solids (g/L)
y	Measured values

Greek symbols

λ	lag phase duration (day)
γ	Biogas production potential (NmL _{biogas} /gVS)

INTRODUCTION

Effluents generated by juice fruit industries include high concentrations of organic matter as well as high chemical oxygen demand (COD). These effluents have different characteristics depending on the specific types of fruit processing operations, including wastewater from washing, rinsing and sanitizing operations during the fruit crushing and cleaning of tanks (Noronha *et al.* 2002), and sustainable management of these wastes has become a major concern (Kaparaju & Rintala 2006). They can be a source of smell generation, toxic gas emissions, insects' attraction, and pathogen increase, which may lead to contamination of groundwater or surface water. The releases of these wastes into municipal wastewater treatment plants are not regulated and further treatments are required to reduce the organic load and to produce energy. Among the available techniques for biological waste stabilization, anaerobic digestion (AD) of fruit juice industries is suggested as a truly sustainable process which can handle the high organic loads and produces methane (Zerrouki *et al.* 2015).

Anaerobic digestion is a biochemical process that converts organic substrates in an oxygen-free environment. The organic substrates can be divided into molecular components such as sugar, amino acids and fatty acids. These intermediate components are transformed into acetate, hydrogen and carbon dioxide via bacterial activities of methanogens (Szuhaaj *et al.* 2016); then, they are converted into biogas. Anaerobic digestion of fruit juices can provide a clean and environment-friendly gas. Biogas is composed mainly of methane and carbon dioxide, as well as a variety of other compounds such as ammonia, hydrogen, hydrogen sulfide, etc., which will then be converted into electricity and heat in the combined heat/power generators (Mateescu *et al.* 2008). The conversion pathway of the substrate can take place according to four stages: hydrolysis, acidogenesis, acetogenesis, methanogenesis. In anaerobic digestion the hydrolysis step is identified as the rate-limiting factor (Kim *et al.* 2003). To improve the anaerobic digestibility of substrate by making it more readily accessible to anaerobic biodegradation, the disintegration techniques and their combinations are often employed.

The most available techniques that are widely applied to pretreat the raw material before fermentation are: biological (e.g., rot fungi, bacteria), chemical (e.g., acid, alkali, organic solvents, ozone), physical (e.g., hydrothermolysis, microwave, ultrasound), physicochemical (e.g., steam explosion, ammonia fibre explosion) and combined techniques, etc. (Demirel *et al.* 2005; Rivas *et al.* 2011; Kavitha *et al.* 2016; Pei *et al.* 2016; Yuan *et al.* 2019; Sinbuathong & Sillapacharoenkul 2020). Wang *et al.* (1995) pointed out that the level of methane produced from activated sludges is increased by 64, 30 and 27% when using ultrasonic disintegration (9 kHz, 30 min), thermal pretreatment by autoclave (120 °C, 30 min), thermal pretreatment with hot water (60 °C, 30 min) and freezing (−10 °C, 900 min), respectively.

Ultrasonication appears as a promising mechanical disruption technique that improves the biodegradability and the methane percentage in biogas with energy recovery; 1 kW of ultrasound energy generates 7 kW of electrical energy (Pilli *et al.* 2011). Ultrasound (US) irradiation process is a potential option for structural deconstruction of biomass, thus improving the performance of anaerobic digestion (AD). This technique is regarded as being useful to all wastewater treatment plants in terms of treatment and disposal of sewage sludge (Pilli *et al.* 2011). It has been found that ultrasonic pretreatment of both primary sludge (Harrison 1991; Hogan *et al.* 2004) and secondary sludge (Harrison 1991) improves the AD efficiency due to the fast hydrolysis step. This results in a release of extracellular polymeric substances and intracellular materials into the soluble phase; as a consequence, the rate and biogas production are increased. The use of US induces monolithic cavitations, including physical and chemical effects in liquid solutions. Two main mechanisms can be distinguished during the sonication of organic compounds: cavitation, which benefits at 'low frequencies', and chemical reactions at 'high frequencies' involving OH, HO₂, and H radicals (Alagöz *et al.* 2018). Note that the mechanical vibrations of ultrasound propagate in liquid phase; they compress and stretch the liquid characterized by a repetitive pattern of compression and rarefaction (Mason 1999; Khanal *et al.* 2007). The continuous irradiation of liquid phase creates a negative pressure and generates microbubbles. According to Suslick (1998), the process in which the dissolved gas within the liquid is transformed into bubbles by the sound field is called rectified diffusion.

For several complex industrial wastewaters such as fruit juice effluents, low ultrasound frequency has a promising potential in improving rate hydrolysis. At this frequency greater acoustic energy can be generated, which may then result in cavitation in liquids (Mason & Lorimer 2002). Cesaro *et al.* (2012) previously reported that ultrasound processes performed at low frequencies facilitate the particle solubilization of organic matter, thus ensuring the availability of large quantities of readily digestible organic matter in the liquid phase. The effects of ultrasound, ozonation and thermal pretreatment techniques have been investigated in waste activated sludge. It has been found that the sludge is nearly completely biodegradable within 24 days with sonication (6,250 or 9,350 kJ/kg TS) and thermal pretreatment (170 or 190 °C). Ultrasound has little

effect on solubilization of sludge but reduces particle size, which increases the biodegradability of substrate, while the main thermal treatment effect concerns solid solubilization and does not affect remaining particulate biodegradability. They find no difference between sludge biodegradability results regarding sonication and thermal pretreatment techniques (Bougrier *et al.* 2006).

The energy supplied during sonication results in different steps based on the hydrolysis and the sludge solubilization: hydro-mechanical shear forces, formation of radicals ($\cdot\text{OH}$, $\cdot\text{O}$, $\cdot\text{H}$), thermal decomposition of volatile hydrophobic substances, and temperature increase (Junior *et al.* 2020).

Oz & Uzun (2015) employed low frequency ultrasound to enhance biogas production from olive mill wastewater (OMW). It has been found that the optimum ultrasonic condition reached for diluted OMW is about 20 kHz for 10 min. The application of ultrasound to OMW increased sCOD/COD ratio from 0.59 to 0.79. They found that the anaerobic batch reactor fed with diluted OMW pretreated by ultrasound produced approximately 20% more biogas and methane compared with the untreated biomass. The ultrasound effect on biogas production from food waste and waste activated sludge has been investigated by Naran *et al.* (2016). The highest amount of organic matter removal was about 11.1% and 39.5% for food waste and activated sludge, respectively, compared to the removal reached in the case of untreated substrate. Ultrasonic pretreatment of these wastes increased methane yield by 56.2%. Li *et al.* (2018) have studied the effect of ultrasonic effect on biogas production from activated sludge. The authors demonstrated increased methane content by as much as 53.8% with the yield of about 36.2 mL/gVS.

Ultrasound was investigated on different kinds of cheese whey substrates to improve methane yield and increase the rate of CH_4 production kinetics. The findings showed a significantly faster kinetics (up to 46% increase in CH_4) and an increase in methane value (maximum +16%) at low ultrasonic energy of 251.4–693.7 Wh/kg VS. An increase in ultrasonic energy up to 1,387.5 Wh/kg VS has not resulted of higher production of methane. This may be explained by the formation of soluble compounds, several of which are not readily treated by anaerobic biomass (Mainardis *et al.* 2019).

The aim of this study was to investigate the opportunities to improve biogas production and methane content from fruit juice effluents by assessing the efficacy of ultrasound pretreatment in solubilization of organic matter. For this purpose, the experiments were conducted in anaerobic batch reactor at low frequency of 20 kHz and at different sonication times (20, 40 and 60 min).

The relationship between methane produced, total sugars and COD reduction during anaerobic digestion was also examined. Furthermore, the modified Gompertz equation was selected to describe the cumulative biogas production.

MATERIALS AND METHODS

Experimental set-up

The fermentation process was carried out using a 500 mL Erlenmeyer flask; the volume of fruit juice effluent in the fermenter was about 375 mL. The operating temperature and agitation speed were maintained constant during the AD process at 35 ± 2 °C and 150 rpm, respectively.

On the top of the reactor, several ports were placed in the tight fitting lid; one serves to monitor the output of biogas, while the others control several parameters through inserted probes such as pH and temperature, and collect liquid samples for subsequent analysis. The experiments were performed at an initial pH of 7, which is adjusted by adding NaOH or HCl (respectively at 2 N and 1 N). Samples were withdrawn daily from the sampling port to measure COD, VFA, proteins, carbohydrates and pH. Biogas formed was followed up by liquid displacement method (Syaichurrozi & Sumardiono 2013). The digestion of high fruit juice substrate was carried out in batch operation and the tests were performed in triplicate. The fermentations were conducted for 20 days.

Substrate characterization

The substrate selected in this study was the effluents provided from a fruit juice processing plant. The plant is located at the industrial park of Didouche Mourad, near Constantine region (Algeria). These effluents are considered as pollutants due to their high concentration of organic matter (500 g COD/L). The substrate to inoculum ratio (S/I) is taken as 2.26. The substrate was stored at 4 °C until use. Table 1 provides the composition and the characteristics of the substrate. It can be seen that the BOD_5/COD ratio of the fruit juice wastewater was about 0.3. This ratio is used as an indicator for biodegradation capacity of organic matter of effluents. To ensure a ready biodegradability, the minimum BOD_5/COD ratio required is about 0.4 (Andrion *et al.* 2019). Besides, substrate pretreatment is required to decompose the complex structure of organic compounds

Table 1 | Substrate characteristics

Parameters	Raw
pH	3.09 ± 0.01
Conductivity (mS cm ⁻¹)	1.83 ± 0.1
Oxidation reduction potential (mV)	200 ± 0.01
Total sugars (g L ⁻¹)	130 ± 0.1
COD (g L ⁻¹)	500 ± 10
sCOD (g L ⁻¹)	208 ± 10
BOD ₅ (g L ⁻¹)	150 ± 4.3
BOD ₅ /COD	0.30
TS (g L ⁻¹)	110 ± 1.23
TSS (g L ⁻¹)	3.59 ± 0.03
Protein (g L ⁻¹)	0.16 ± 0.001
Total Kjeldahl nitrogen (mg L ⁻¹)	84 ± 0.25
VS (g/L)	0.85 ± 0.03
Acetic acid (mol/L)	0.042
Butyric acid (mol/L)	0.020
Propionic acid (mol/L)	0.013

(polysaccharides, proteins, lipids) into simpler compounds (glucose, fatty acids, amino acids). In this case, ultrasound biomass pretreatment process appears as a promising technique to improve biogas production and methane content.

Ultrasound pretreatment

Experiments have been carried out at different sonication times: 20, 40 and 60 min, using an ultrasonic bath (Model: EMMIH30, power 180 W) at a temperature of 35 °C and frequency of 20 kHz. The specific energy input can be calculated using the following formula (Oleszek & Krzemińska 2021):

$$E_i = (P \times t) / (V \times TS) \quad (1)$$

where P is power (W), t is exposure time (s), V is sample volume (L), and TS is content of total solids in the sample (kg/L).

The specific energy input was 5.242, 10.484 and 15.728 MJ/kg TS for a fixed power input 180 W and sonication times of 20, 40 and 60 min, respectively.

Analytical methods

Total sugars before and after AD were measured by the phenol-sulfuric acid method (Dubois *et al.* 1956), and protein concentration was determined by using Lowry method (Lowry *et al.* 1951). Chemical oxygen demand (COD), soluble oxygen demand (sCOD), biological oxygen demand (BOD₅), total Kjeldahl nitrogen (TKN), VS and TSS were measured according to the Standard Methods (APHA 1998). High-performance liquid chromatography (HPLC) (Agilent YL9100) was used in order to estimate the evolution of the VFA concentrations using a C8 column (5 μm – 25 × 0.46). The mobile phase was sulfuric acid (0.005 M) at a flow rate of 0.7 mL/min. The column temperature was set at 30 °C and the detection was made spectrophotometrically at a wavelength of 210 nm. Coupled gas chromatography and mass spectrometry (GC/MS) (Perkin Elmer Clarus 600, Perkin Elmer, Waltham, MA, USA) was used to analyse the biogas composition. The GC was fitted with an Rtx[®]-VMS Column (60 m × 250 μm ID) (Restek Corporation, Bellefonte, PA, USA). Helium was used as the carrier gas at a flow rate of 1 mL/min. The oven temperature was set to 40 °C with a rate increase of 3 °C/min until the temperature of 180 °C was reached; then it was kept at this temperature for 3 min. The injector and detector temperatures were set to 180 °C. The pH value, redox potential, conductivity and temperature were measured using an Inolab (Prolabmas, Murni Swadaya, Jakarta, Indonesia) multi-parameter 720 device.

KINETIC BIOGAS PRODUCTION

Kinetic study of anaerobic digestion of wastewater from the fruit juice processing was carried out using an empirical non-linear kinetic model. This model links between the cumulative biogas and the potential of biogas produced. This model is suitable to describe the behaviour of sigmoidal functions (Zwietering *et al.* 1990). Kinetic parameters are estimated by fitting the experimental cumulative biogas production curve with (Equation (2)) (Zerrouki *et al.* 2015):

$$B(t) = \gamma \cdot \exp \left\{ - \exp \left[\frac{R_m \cdot e}{\gamma} (\lambda - t) + 1 \right] \right\} \quad (2)$$

where $B(t)$ is the cumulative biogas production during the digestion time t (L/g VS), γ is the biogas production potential (L/g VS d), R_m is the maximum production rate (L/g VS d), and λ is the lag phase duration (d).

Regression coefficients (R^2) and root mean square error (RMSE) between experimental data and estimated during fermentation process are estimated according to Kafle & Kim (2013).

$$RMSE = \left(\frac{1}{m} \sum_{j=1}^m \left(\frac{d_j}{y_j} \right)^2 \right)^{\frac{1}{2}} \quad (3)$$

where m is the number of data pairs, j is the j th values, y is the measured values and d is the deviations between measured and predicted data.

RESULTS AND DISCUSSION

Effects of ultrasound on biogas production

Cumulative biogas production in anaerobic batch reactor under mesophilic temperature ($T = 35^\circ\text{C}$) and modelled are given in Figure 1(a). It can be seen that the biogas production increases with the increase of time for selected tests. The juice effluents pretreated by ultrasound provide higher biogas production compared to untreated effluent; the values reached are about 409 and 162 $\text{NmL}_{\text{biogas}}/\text{gVS}$, respectively. This increase is more pronounced after 8 days of AD. Then, the biogas production rates gradually decreased and dropped to low level after 17 days. It is important to note that the increase of sonication time of fruit juice effluents increases the yields of biogas production, to reach the value of 409 $\text{NmL}_{\text{biogas}}/\text{gVS}$ after 60 min of pretreatment. There is no significant biogas production difference between sonication times of 40 and 60 min. These findings are consistent with the results obtained by Wang *et al.* (1999), who found that under prolonged sonication time, more than 30 min, the increment of methane generation is similar to the response reached for sonication time of 30 min. On the other hand, the kinetic parameters of the modified Gompertz model are almost identical for sonication times of 40 and 60 min (Table 2). Li *et al.* (2018) recommended less than 80 min of sonication time prior to conducting anaerobic digestion of activated sludge. This increase is attributed to the increase of COD removal and total sugars uptake during ultrasonic pretreatment, which resulted in the solubilization of organic matter.

Moreover, the increase in the energy input of about 5.242, 10.484 and 15.728 MJ/kg TS increases the biogas production to the values of 47, 57 and 60%, respectively (Table 3). These results are similar to those found by Oz & Yarimtepe (2014). However, Mainardis *et al.* (2019) found that an increase in ultrasonic energy up to the value of 1,387.5 Wh/kg VS does not lead to an increase in methane production from cheese whey. This is due to the formation of soluble compounds, several of which are not readily treated by anaerobic biomass; according to these authors it appears advantageous that the system operates at a higher ultrasound power, rather than longer ultrasound time.

As ultrasound waves propagate in the liquid phase, cavitation is produced (Pilli *et al.* 2011). These microbubbles are generated by acoustic forces (Suslick 1990) due to the surface tension and gradients in the pressure waves. The increase in biogas yield is due to the increase in the net surface area of the particles and solubilization of the complex organic compounds. Compared to the untreated fruit juice effluent (control), the amount of biogas produced increased by 47, 57 and 60% for sonication times of 20, 40 and 60 min, respectively. These results are in agreement with those previously reported by Oz & Uzun (2015), who applied low frequency ultrasound pretreatment to olive mill wastewaters. The treated effluent was found to yield 20% more biogas compared to control (untreated effluent), while the biogas production is significantly improved by 27–104% in the case of anaerobic digestion of sludge in mesophilic conditions (Zhen *et al.* 2017).

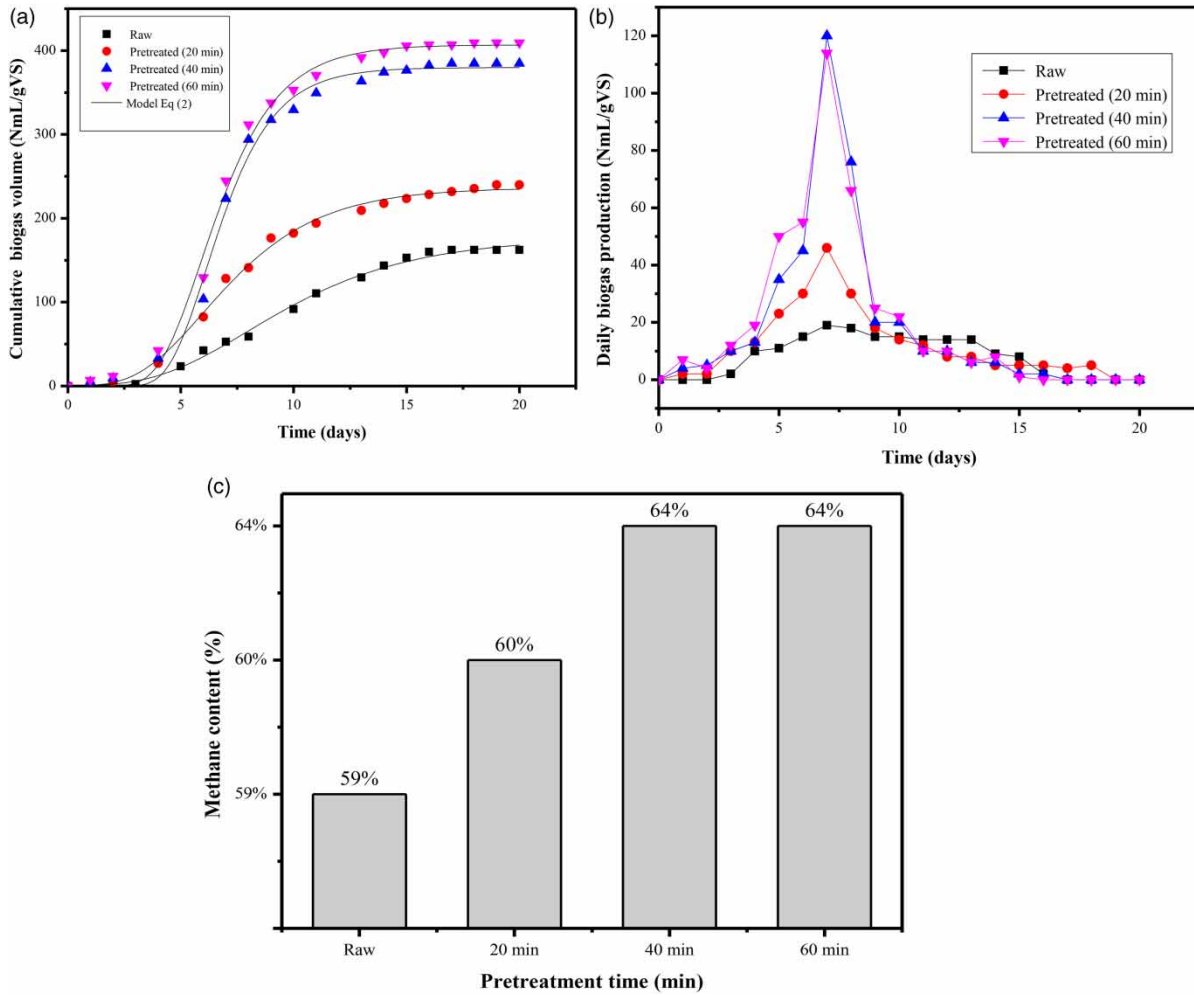


Figure 1 | Performance comparison of the biogas production. (a) Comparison of the calculated values and experimental data of biogas production. (b) Daily biogas production. (c) Methane content.

Table 2 | Modified Gompertz model: kinetic constants parameters for biogas production

	$B(t)$ (NmL _{biogas} /gVS)	γ (NmL _{biogas} /gVS)	R_m (NmL _{biogas} /gVS .day)	λ (day)	R^2	RMSE
Raw	162	161.490	12.623	4.186	0.994	0.081
Pretreated (20 min)	240	238.096	24.542	3.817	0.995	0.080
Pretreated (40 min)	384	383.333	62.258	3.405	0.991	0.086
Pretreated (60 min)	409	408.666	63.768	3.119	0.993	0.083

Table 3 | Increment in the fruit juice wastewater digestibility

Batch ultrasonic system				Yields of fermentation parameters (%)		
Power (W)	Frequency (kHz)	Sonication time (min)	Specific energy consumption (MJ/kg TS)	SCOD	Biogas production	Methane content
180	20	20	5.242	52	47	1.69
		40	10.484	55	57	8.47
		60	15.728	58	60	8.47

Moreover, the increase of sonication time of the sludge increases the methane yield about 12%, 31, 64 and 69%, corresponding to sonication times of 10, 20, 30 and 40 min, respectively (Wang *et al.* 1999). In the present study, the methane level in the biogas was about 59% in untreated substrate and 64% for sonication time of 60 min (Figure 1(c)). This indicates that the ultrasound technique solubilizes the complex substrate into more readily biodegradable compounds (Naran *et al.* 2016). Low frequencies (20–40 kHz) facilitate mechanical effects that improve the methane yield due to the greater availability of macromolecular components such as proteins and carbohydrates for the anaerobic digestion process (Junior *et al.* 2020). Note that the biogas components are influenced by reactor temperature, feeding material mode, and bacterial growth (Jena *et al.* 2017). Based on these results, it appears that the ultrasound pretreatment of fruit juice effluents is a more efficient technique to produce clean energy, particularly biogas rich in methane.

A good agreement is observed between the modified Gompertz model and the experimental data, with $R^2 > 0.99$, and the RMSE value fell within the range of 0.080–0.086 in all cases. The kinetic parameters of the modified Gompertz model are given in Table 2. It was found in the case of untreated juice effluent that the γ -value is lower compared to the values obtained in the case of ultrasound pretreated substrate; it was about 161.490; 238.096; 383.333 and 408.666 (NmL_{biogas}/gVS) for sonication time of 0, 20, 40 and 60 min, respectively. It is worth mentioning that the γ -value represents the biogas production that can be potentially produced for a given process. Furthermore, the R_m (maximum biogas production rate) is about 63.768 NmL_{biogas}/gVS.day for the fermentation conducted with fruit juice effluents subjected to ultrasonic pretreatment for 60 min, while it was only about 12.623 NmL_{biogas}/gVS.day in the case of untreated substrate (control). This agrees well with the results previously found by (Naran *et al.* 2016) regarding the ultrasound pretreatment of food waste and waste activated sludge where the maximum biogas production (γ -value) and biogas production rate (R_m) are significantly enhanced by 423.6 mL CH₄/g VSS and 26.6 mL CH₄/g VSS day, respectively. The lag phase period was shorter in the case of sonicated effluents compared with untreated effluent, which indicates that the methane production was induced immediately in the case of sonicated effluents. In this case, the ultrasound pretreated substrate provides better operating conditions for biomass growth and biogas production, thus promoting the activity of methanogen bacteria.

Effects of ultrasound on sCOD concentration and COD removal

The COD removal during anaerobic digestion of ultrasound pretreated fruit juice effluents and untreated effluent is given in Figure 2. It can be seen that the COD removal is significantly (40%) greater when the substrate is subjected to ultrasonic compared with the untreated effluent. The ultrasound pretreatment improved the total COD removal during the AD process. This is due to the sonication process, which improves the solubilization of complex organic compounds. The average sCOD level reached is about 208, 432, 464 and 496 gO₂/L for sonication times of 0, 20, 40 and 60 min, respectively. In this case, the sCOD increased by 52, 55 and 58% for sonication times of 20, 40 and 60 min, respectively. Moreover, the increase in the energy input increases the sCOD concentration from 52 to 58%. These results are in agreement with those previously

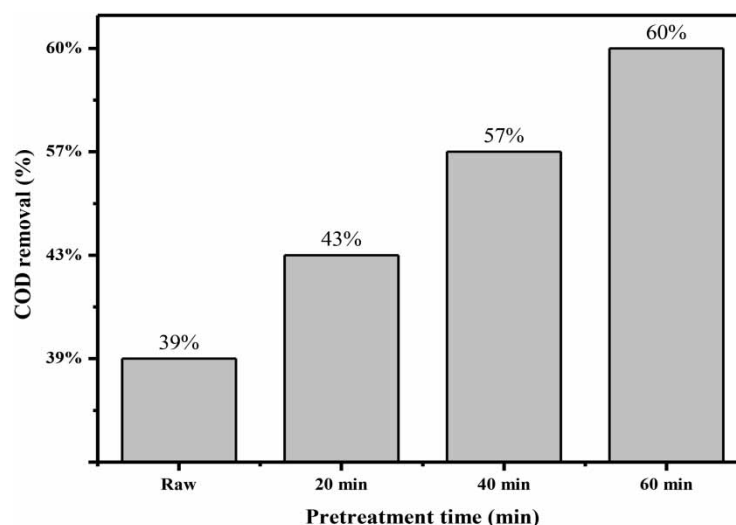


Figure 2 | Effect of ultrasonic pretreatment time on the COD removal.

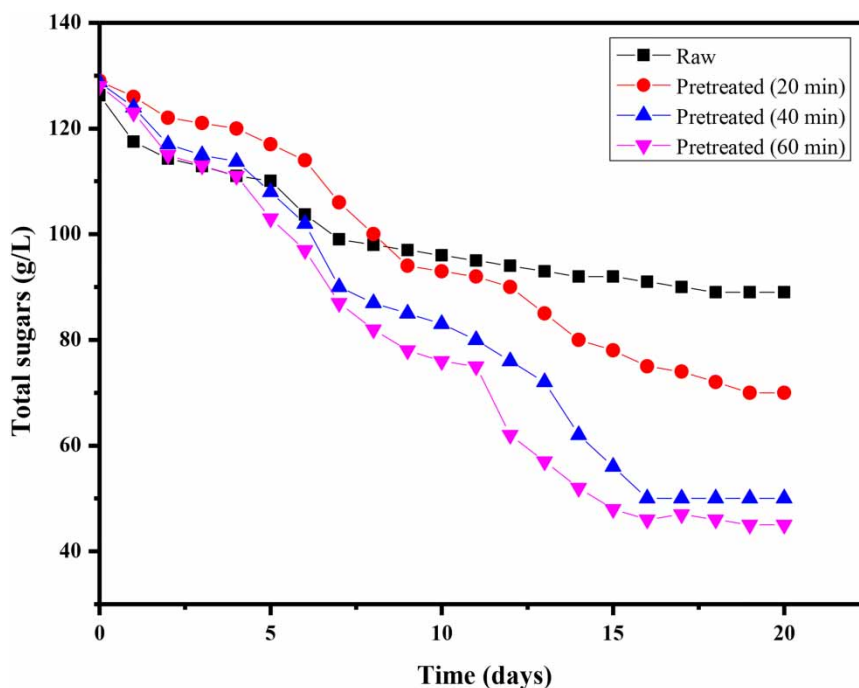


Figure 3 | Effect of ultrasonic pretreatment time on the total sugars uptake.

found by Grönroos *et al.* (2005). The organic matter breaks down the bonds in complex molecules and increases digestibility (Kavitha *et al.* 2015). After 20 days of anaerobic digestion, the efficiency increased by 9, 31 and 35% for sonication times of 20, 40 and 60 min, respectively, compared to the control. Similar results were obtained by Oz & Yarimtepe (2014), who found that the COD removal efficiency of raw and ultrasonically pretreated leachate was about 44 and 67%, respectively. Naran *et al.* (2016) found that the COD removal is about 50% in the case of co-digestion of food waste and waste activated sludge ultrasonically pretreated at low frequency.

Effects of ultrasound on total sugars uptake

The main sugars in fruit juices are glucose, sucrose and fructose. Analyses carried out by Kaparaju *et al.* (2010) revealed that the glucose is the major component, accounting for almost 60% of sugars. Its presence may serve as a source of energy to keep cells alive. Thus, it is important to assess the amount of sugars taken up by the cells during anaerobic digestion (Figure 3). The uptake of the sugars is performed exponentially, and it seems more pronounced and extremely fast in the case of sonication time of 60 min. Compared with control, the total sugars uptake reached is about 35, 51 and 54% for sonication times of 20, 40 and 60 min, respectively. This means that as the sonication time is prolonged, the energy in the form of chemical energy contained in sugars improved bacterial growth of methanogens and their capacity for sugars uptake; as a consequence, the organic load biodegradability and biogas production are significantly improved.

pH control

The variation of pH values during fermentation of fruit juice effluents is given in Figure 4. It can be seen that the pH level of fruit juice effluents decreases gradually for selected tests after 7 days. Then, it tends to fluctuate between 5.7 and 5.2. The reactor medium was acidified due to the accumulation of volatile fatty acids (VFAs), and resulted in the inhibition of the biomass growth, probably due to the activity and/or the concentration of acid bacteria, which could not stimulate total methane production but promoted the rate of methane production at the beginning of the AD. These results are similar to those of Syaichurrozi *et al.* (2018). Measurement of VFAs composition shows three acids, namely, acetic acid (0.042 mol/L), butyric acid (0.020 mol/L) and propionic acid (0.013 mol/L). These findings are consistent with the results obtained by Staley *et al.* (2011). Therefore, to ensure high biogas production, it is necessary to keep the stability of the process of fermentation, i.e., the pH value constant at 7 (Zerrouki *et al.* 2015).

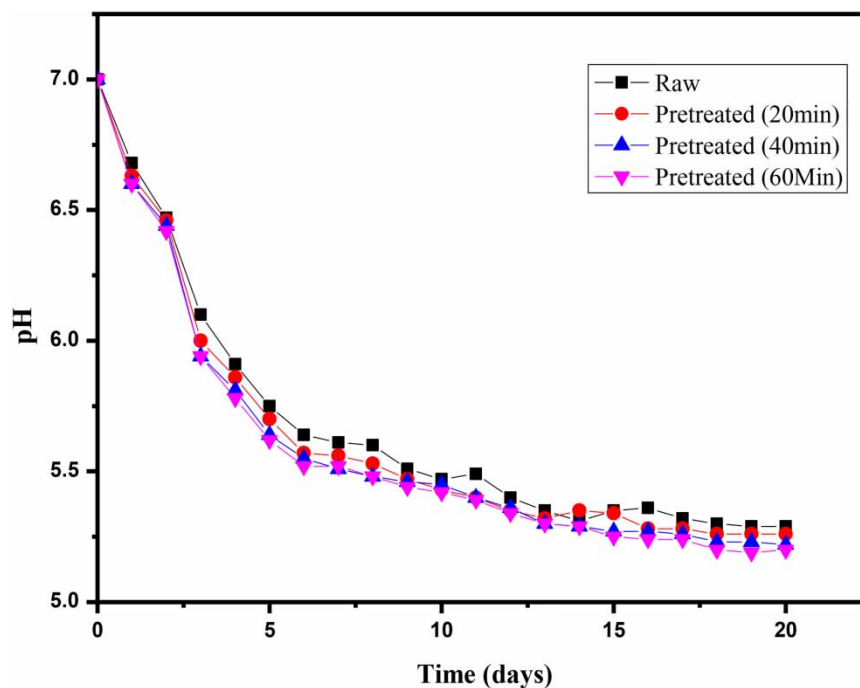


Figure 4 | pH value changes during anaerobic fermentation.

Furthermore, a continuous methanization mode has been suggested to avoid accumulation of volatile fatty acids and keep substrate concentrations constant. The continuous mode can be expected to provide better methane efficiency of the pilot-scale fruit juice processing plant. To better investigate the potential of ultrasonic pretreatment technique, an energy and economic analysis must be assessed.

CONCLUSION

This study has been carried out to investigate the use of ultrasound pretreatment as a potential technique to solubilize organic matter and fermentation of fruit juice effluents.

The main results are:

1. Compared to the untreated fruit juice industry, the amount of biogas increased by 47, 57 and 60%, for sonication times of 20, 40 and 60 min, respectively. Methane content in the biogas was about 59% in untreated substrate and 64% for sonication time of 60 min.
2. The ultrasound pretreatment improved the COD removal during anaerobic digestion process. The efficiency increased by 9, 31 and 35% for sonication times of 20, 40 and 60 min, respectively, with respect to control.
3. Compared with control, the total sugars uptake reached is about 35, 51 and 54% for sonication times of 20, 40 and 60 min, respectively.
4. The modified Gompertz equation was used to describe the cumulative biogas production. A good agreement was found between simulated and experimental data.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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