Journal of Oil Palm Research Vol. 37 (3) September 2025 p. 506-518
DOI: https://doi.org/10.21894/jopr.2024.0030

EVALUATING THE EFFECT OF LOW AND HIGH TEMPERATURE MODE OF SUBCRITICAL WATER PRE-TREATED EMPTY FRUIT BUNCHES ON CO-DIGESTION PERFORMANCE AND KINETIC STUDY FOR METHANE PRODUCTION

ADILA FAZLIYANA AILI HAMZAH[1]; MUHAMMAD HAZWAN HAMZAH[1,2]*; KHAIRUDIN NURULHUDA[1,2]; HASFALINA CHE MAN[1,2]; MUHAMMAD HEIKAL ISMAIL[3] and PAU LOKE SHOW[4,5,6,7]

ABSTRACT

Anaerobic digestion of oil palm empty fruit bunches (EFB) is considered an effective method for non-renewable energy substitution through biogas production. However, lignocellulosic recalcitrance structure of EFB is one of the main difficulties in achieving high biogas production for anaerobic co-digestion with palm oil mill effluents (POME). In this study, EFB was pre-treated with subcritical water (SCW) at low (120°C) and high (180°C) temperatures for 10-30 min to enhance biogas production. The characteristics of EFB after SCW pre-treatment were evaluated to identify changes in physicochemical characteristics. The combination pre-treatment of 180°C for 10 min with 546.18 mL g^{-1} volatile solid (VS) biogas yield and 421.41 mL CH_4 g^{-1} VS methane yield revealed the highest biogas production. Meanwhile, co-digestion of SCW pre-treated EFB with POME led to a removal of more than 66% VS. The sugars released were analysed in liquid fraction of SCW pre-treated EFB where glucose, xylose, cellobiose, mannose and galactose were detected. Notably, kinetic study of biogas production of pre-treated EFB using modified Gompertz model revealed that pre-treatment improved the lag phase, and the highest biogas production rate was observed at 19.80 mL g^{-1} VS. day. In conclusion, co-digestion of EFB with POME for methane production can be improved with the use of SCW pre-treatment.

Keywords: biogas, co-digestion, methane, pre-treatment, subcritical water.

Received: 7 October 2023**; Accepted:** 19 February 2024**;**

INTRODUCTION

The world's oil palm production is predicted to reach 240 million tonnes by 2050 as the market for palm oil products expands (Sulaiman *et al*., 2022). Malaysia's palm oil sector can currently generate 115.86 million tonnes of fresh fruit bunches (FFB), making it the second-largest producer of palm

---

[1] Department of Biological and Agricultural Engineering,
Faculty of Engineering, Universiti Putra Malaysia,
43400 UPM Serdang, Selangor, Malaysia.

[2] Smart Farming Technology Research Centre,
Faculty of Engineering, Universiti Putra Malaysia,
43400 UPM Serdang, Selangor, Malaysia.

[3] Department of Environment,
Faculty of Forestry and Environment,
Universiti Putra Malaysia,
43400 UPM Serdang, Selangor, Malaysia.

[4] Department of Chemical and Petroleum Engineering,
Khalifa University, P.O. Box 127788, Abu Dhabi,
United Arab Emirates.

[5] Zhejiang Provincial Key Laboratory for Subtropical Water
Environment and Marine Biological Resources Protection,
Wenzhou University, Wenzhou 325035, China.

[6] Department of Chemical and Environmental Engineering,
Faculty of Science and Engineering,
University of Nottingham Malaysia,
Jalan Broga, 43500 Semenyih, Selangor, Malaysia.

[7] Department of Sustainable Engineering,
Saveetha School of Engineering, SIMATS,
Chennai 602105, India.

* *Corresponding author e-mail: hazwanhamzah@upm.edu.my*

oil in the world (Awoh *et al*., 2023). However, the process also produces a significant amount of waste. Following the process of extracting crude palm oil (CPO) from FFB, palm oil empty fruit bunches (EFB) are subsequently produced. For every tonne of palm oil produced, typically between 23% and 25% or 1.07 t of EFB, are created (Dolah *et al*., 2021). EFB can be used as a raw material for biogas production. Meanwhile, POME, which is also produced from the same stream as EFB, contains numerous organic matter, solids that are suspended in the water, different nitrogenous compounds, and a mixture of smaller organic and mineral parts that can contribute to water pollution (Tan & Lim, 2019). Due to the rapid expansion of the palm oil industry, a large volume of oil palm waste is produced, thereby raising environmental concerns. These events highlight the need for effective management to ensure environmental sustainability and agricultural productivity (Liew *et al*., 2021). To sustain the oil palm industry, waste treatment is one of the top priorities of palm oil companies. An interesting approach is to use biotechnological methods to transform this waste into an energy resource, which may bring additional financial benefits to the industry (Mahmod *et al*., 2021).

Palm oil wastes such as EFB, decanter cake, and trunk have a high potential for biomethane production. However, due to the high C/N ratio, the use of oil palm wastes as a single substrate in mono digestion often results in poor digester performance (Park *et al*., 2021). The high carbon content in these wastes led to poor buffering capacity and excessive volatile fatty acid production in the biogas digester. Nevertheless, co-digestion with nitrogenous feedstock would eliminate the problem (Hamzah *et al*., 2024). Co-digestion of EFB with another waste stream from palm oil production is considered a viable waste management option in palm oil mills. Liew *et al*. (2021) found that co-digestion of EFB and POME produced relatively high methane-rich biogas, with a performance that was 2.36 times better than using each of them as a single feedstock. Despite anaerobic co-digestion, biogas yield is not satisfying due to the intricate nature of the composition. The application of pre-treatment before biogas production breaks down the lignocellulosic bond, subsequently making it accessible for hydrolysis (Aili Hamzah *et al*., 2023).

Due to the resistant structure of the EFB, the rate of anaerobic digestion of the EFB is constrained at the hydrolysis stage. Consequently, biogas production can be severely impacted by the inability of lignocellulosic components like lignin and cellulose to degrade into simple sugars (Saritpongteeraka *et al*., 2022). The disruption in the morphological structure of lignocellulosic substrates through pre-treatment has gained remarkable attention as a viable strategy for biogas enhancement (Ahmad *et al*., 2018). Several studies have demonstrated the effectiveness of biological, chemical, and hydrothermal pre-treatment of palm oil solid wastes in enhancing the yield of methane-rich biogas (Mamimin *et al*., 2021; Saritpongteeraka *et al*., 2022; Sitthikitpanya *et al*., 2018). SCW pre-treatment is widely recognised as a sustainable and greener technology that facilitates structure-breaking and the accessibility of substrates. Moreover, the pre-treatment does not need recycling of acid and is environmentally friendly (Chen *et al*., 2021). Water in the SCW region penetrates the lignocellulosic structure and subsequently hydrates cellulose, dissolving hemicellulose and eliminating lignin partially (Sarker *et al*., 2021). Thus, SCW pre-treatment can facilitate enzymatic hydrolysis and digestion of lignocellulosic biomass, leading to fast and significant biogas production.

To date, limited studies are available detailing the physiochemical properties of EFB after SCW pre-treatment. Most previous studies focused on the impacts of the SCW pre-treatment on biogas production. Tian *et al*. (2020) conducted a pre-treatment for wheat straw and sludge at a temperature of 175°C. Resultantly, the co-digestion process of the pre-treatment improved biogas production, thus promoting 52.0% of biogas production as compared to untreated samples. At temperatures of 120°C and 180°C, Xiang *et al*. (2021) observed an increased production of biogas from the pre-treated rice straw by 38.0% and 14.0%, respectively. Lignin in the solid fraction was also reported to increase as time increased due to recombination between lignin and hemicellulose (Ahmad *et al*., 2018). Pre-treatment at 120°C resulted in a 4.9% reduction of total solids (TS), which was further reduced to 6.4% upon increasing the pre-treatment time (Dasgupta & Chandel, 2019). Furthermore, Aili Hamzah *et al*. (2023) suggested that energy can be saved by less reaction time than by longer reaction time.

However, the effectiveness of the pre-treatment regarding the unique characteristic of SCW pre-treatment is based on the process parameters itself as a function of reaction temperature and time. The sugar production increased with temperature (170°C-210°C), and temperatures below 120°C were not high enough to cause differential breakdown of hemicellulose and cellulose into simple sugar (Saritpongteeraka *et al*., 2022). On the other hand, methanogenesis inhibitors known as furan derivatives were formed at pre-treatment temperatures higher than 200°C (Lee & Park, 2020). It was observed that a 6.9% reduction in methane yield after pre-treatment at 175°C (Tian *et al*., 2020). At 180°C, Wang *et al*. (2018) reported that biogas produced from pre-treated rice straw improved 3.0% compared to untreated rice straw.

While temperature 210°C presented a 30.0% reduction with a more extended lag period. The optimum biogas yield was obtained at low severity of 2.65. The SCW on cocoa pod waste observed that temperature influences the lignin solubilisation compared to reaction time (Antwi *et al.*, 2019). Pre-treatment temperature up to 120°C resulted in enhanced methane content as pre-treatment time was increased. While, higher SCW temperature has resulted in more significant degradation and soluble sugars produced from hemicellulose solubilisation (Dasgupta & Chandel, 2019). SCW is aimed to improve biogas production but at the same time balance the fermentable sugar yield with lower operational cost. The SCW pre-treatment parameters, temperature and reaction time are the three main factors that influence pre-treatment efficiency and subsequently, anaerobic digestion.

This study aims (1) to investigate the effect of SCW pre-treatment at low (120°C) and high-temperature (180°C) modes on the physicochemical properties of EFB, (2) to determine the sugar production after SCW water pre-treatment, (3) to evaluate the biogas profiles and yield from the co-digestion of SCW pre-treated EFB with POME, and (4) to identify the changes in initial and final effluent of anaerobic digestion process parameters and their kinetic analysis.

MATERIALS AND METHOD

Sample Collection

The EFB and POME used in this study were obtained from the Palm Oil Mill, Selangor in Malaysia. The inoculum was collected from the anaerobic digester treating POME. The inoculum was pre-incubated for three days at 37°C ± 1°C before being used to remove any background methane from the inoculum and adjust the microorganisms to mesophilic environments (Chan *et al.*, 2021). The EFB was dried at 60°C and then went through a grinding process to 500 µm size using a universal cutting mill pulverisette 19 (Fritsch, Germany). The POME and inoculum were stored at 4°C in the refrigerator before further use.

Subcritical Water Pre-treatment

The SCW pre-treatment was carried out in a high-temperature reaction bath with reactors fitted on a rocking bed. In a 40 mL steel reactor, EFBs were poured into the reactor along with water at a ratio of 1:10 (EFB:Water). Thereafter, the reactor head was assembled and tightened after nitrogen gas was purged for 1 min at 2 mL min^{-1}. The SCW pre-treatment was conducted at 120°C and 180°C at 10 and 30 min, respectively. Then the oil was heated to achieve the desired temperature before each pre-treatment. The reactor was attached to the rocking bed in the oil bath, and once the pre-determined amount of time had passed, they were taken out and quickly being immersed in the ice bath. To lower the pressure inside, the mixtures were left prior to usage. The SCW pre-treated EFBs were then co-digested with POME to produce biogas. To quantify the SCW pre-treatment combined effect of time and temperature, the pre-treatment severities (S) were measured for each set of pre-treatments. The S was calculated using Equation (1), where t is the time (min) and T is the SCW temperature (°C) (Vakalis *et al.*, 2022).

$$Log\ S = Log\left[t.EXP\left(\frac{T\text{-}100}{14.75}\right)\right] \tag{1}$$

Biogas Production

The batch co-digestion using SCW pre-treated EFB and POME was performed in 125 mL serum bottles at mesophilic temperature. The working volume of each digester was 100 mL with an organic

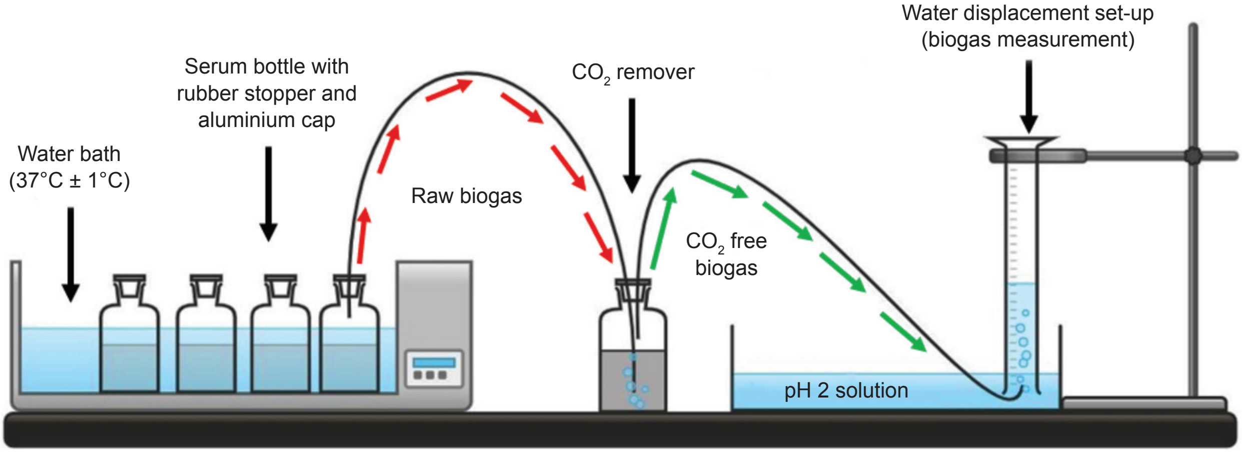

Figure 1. Anaerobic co-digestion set-up.

loading of 11 g VS L^{-1} as described by Liew *et al.* (2021). The bottles were submerged in a water bath during the digestion period at 37°C ± 1°C (Memmert, Germany). Meanwhile, the substrate's pH was adjusted to 7 using 3M sodium hydroxide and hydrochloric acid. Batch testing was conducted until the biogas generation rate was constant (60 days). This procedure was conducted until the plateau of the generated gas was achieved. Each sample was prepared in triplicate to increase the reliability of the measures. Gas production was collected daily using the water displacement method (*Figure 1*).

Analytical Methods

The total solid (TS), VS and total ammonia nitrogen (TAN) were determined using the standard method (APHA, 1998). The TAN measurements were performed according to standard procedure #4500-D. The CHN628 Series (LECO, United States) was used to analyse the carbon (C) and nitrogen (N) contents, whereas a pH meter was employed to measure the pH value. The percentage of lignin in EFB waste was determined based on the Technical Association of the Pulp and Paper Industry (TAPPI) standard procedure (TAPPI, 1950), while the measurement of sugars was performed using a high-performance liquid chromatograph (HPLC) with a refractive index detector (RID). Gas chromatography (GC) with a thermal conductivity detector (TCD) analyser (Agilent 6890N, United States) was used to identify the methane composition of the biogas.

Kinetic Study

A modified Gompertz model equation kinetically analysed the cumulative biogas production data. The gas production curve corresponds to a slow, flat curve when complex solid organic materials are used as substrates (Xiang *et al.*, 2021). The kinetic study provides several essential parameters, such as the lag phase and the maximum biogas production rate for the digestion process. The lag phase (λ) is also an important factor used to reflect the efficiency of the digestion process. The modified Gompertz [Equation (2)] was applied to obtain a better understanding of the behaviour of the process (Tian *et al.*, 2020). The assumption using this model is that the biogas produced in the batch test corresponds to the specific growth rate of the methanogenic bacteria in the digester.

$$B = B_0\, exp\left\{-\, exp\left[\frac{\mu . e}{B_0}(\lambda\text{-}t)+1\right]\right\} \tag{2}$$

where B is biogas production yield (mL g^{-1} VS), B_0 is the maximum biogas yield (mL g^{-1} VS), μ is the maximum biogas production rate (mL g^{-1} VS.day), λ is the lag phase period, t is time and e is an Euler's function = 2.71828. B and t were determined from the experimental data, while B_0, μ and λ were determined from the model. R^2 is a coefficient of determination of the relationship between experimental and predicted values of biogas (Lim *et al.*, 2021). The high R^2 closer to 1 showed that the data adequately fit the model. The accuracy of the studied model is compared by computing the root mean square error (RMSE). The RMSE is calculated as the standard deviation between the predicted and experimental values, as shown in Equation (3) (Manogaran *et al.*, 2023). A smaller RMSE is considered more desirable.

$$RMSE = \sqrt{\sum\nolimits_{i=1}^{n} \frac{(Y_{i,}\, experimental - Y_{i,}\, predicted)^2}{n}} \tag{3}$$

Statistical Analysis

The data were presented as mean ± standard deviation, and statistical analyses were performed using SAS software (Version 9.4). The data were analysed using analysis of variance, followed by Tukey's multiple comparison test. The *p*-value less than 0.05 was considered statistically significant.

RESULTS AND DISCUSSION

Properties of EFB

Table 1 depicts the main characteristics of EFB, POME, and inoculum used in evaluating the potential of biogas production from SCW pre-treated EFB. As shown in the characterisation results, TS and VS of the EFB were 95.77% and 94.38%, respectively. These findings are consistent with the results by Tepsour *et al.* (2019), in which the values of TS and VS were 96.38% and 90.22%, respectively. The C/N ratio is crucial in anaerobic digestion, as it indicates the nutritional supply for the microorganisms in the reactor. It is appropriate to add POME in this instance due to the EFB's high C content (43.24%) as a carbon source to improve biogas yield. Typically, the reported C/N ratio of EFB is 60-80 (Liew *et al.*, 2021; Mamimin *et al.*, 2021). In the present study, the C and N of the POME were 35.65% and 2.04%, thereby resulting in a C/N ratio of 17.47. In terms of pH, the acidity obtained is similar to that reported in a previous study (Tan & Lim, 2019). The TS, VS and pH of the inoculum were 1.75%, 35.56% and 7.55, respectively.

The solid yields of EFB decreased with an increase in the SCW pre-treatment temperature (*Table 2*). Moreover, the solid yield decreased with an increasing pre-treatment time, from 10 to 30 min. The lower solid yields at the longest pre-treatment time were due to the extension of the pre-treatment time, which increased delignification and carbohydrate dissolution (Jomnonkhaow *et al.*, 2022). Similarly, the VS of the EFB was also reduced with SCW temperature. An increase in the SCW pre-treatment time from 10 to 30 min further reduced the VS of SCW pre-treated EFB. Higher pre-treatment temperature and time resulted in solubilisation, structural changes and modification of the lignocellulosic fraction in the biomass, thereby reducing the solid fraction (Chen *et al.*, 2021). Higher pre-treatment severity also led to a relatively higher solid recovery in the pre-treated biomass (Ahmad *et al.*, 2018). Pre-treatment severity reflects the intensity of the pre-treatment, whereby the severity increases in line with an increment in the pre-treatment temperature and time. Low pre-treatment severity might be insufficient to overcome the recalcitrance structure of biomass, thus leading to poor microbial performance (He *et al.*, 2022).

The severity of SCW pre-treatment in this study ranged from 1.59-3.83. Meanwhile, the pH of SCW pre-treated EFB decreased towards a higher pre-treatment severity. According to Simanungkalit *et al*. (2017), a decrease in pH resulted from the formation of soluble sugar and organic acids in the liquid fraction along with pre-treatment severity. Notably, the lignin content of SCW pre-treated EFB in the present study increased in comparison to the untreated EFB at a higher temperature and longer reaction time. As found in previous studies, increasing pre-treatment time and temperature, along with pre-treatment severity, were reported to increase the biomass's lignin content (Ahmad *et al.*, 2018; Jomnonkhaow *et al.*, 2022). The increase in lignin may be attributed to the formation of pseudo-lignin under severe pretreatment conditions (He *et al.*, 2022). From *Table 2*, it can be seen that hemicellulose content reduced from 22.40%-16.90% as the temperature and time increased. This result supports the hemicellulose trend from previous observations in which the hemicellulose fraction of wheat straw reduced from 24.52%-17.84% as the temperature increased from 125°C-175°C (Tian *et al.*, 2020). As mentioned before, hemicellulose can be degraded at a temperature above 150°C (He *et al.*, 2022). Hemicellulose degradation altered lignocellulosic structural complexity and improved microbial accessibility to cellulose, improving anaerobic digestibility (Wang *et al.*, 2018). While for the cellulose fraction, a slight increase in cellulose can be observed from the study. This result further supports the observation from the previous study that the cellulose fraction did not change significantly when the pre-treatment temperature was promoted to 130°C, and the percentage increased more noticeably at higher temperatures (Xiang *et al.*, 2021). This is likely related to the complexity of the cellulose, which is only partially soluble in water or organic solvents at temperatures below 200°C (Phuttaro *et al.*, 2019).

TABLE 1. CHARACTERISTICS OF EFB, POME AND INOCULUM USED IN THIS STUDY

Characteristics	EFB	POME	Inoculum
TS (%)	95.77 ± 0.34	6.36 ± 0.03	1.75 ± 0.05
VS (%)	94.38 ± 0.21	79.62 ± 0.15	35.56 ± 0.55
C (%)	43.24 ± 0.05	35.65 ± 0.16	NA
N (%)	0.53 ± 0.01	2.04 ± 0.00	NA
C/N	82.21 ± 0.04	17.47 ± 0.28	NA
pH	NA	4.55 ± 0.02	7.55 ± 0.06

Note: Data in the table are exhibited in the form of mean ± standard deviation, NA - not available; TS - total solid; VS - volatile solid; C - carbon; N - nitrogen; C/N - carbon/nitrogen.

TABLE 2. PHYSICOCHEMICAL PROPERTIES OF SCW PRE-TREATED EFB AFTER PRE-TREATMENT

Pre-treatment	Severity	pH	Solid yield (%)	VS reduction (%)	Lignin (%)	Cellulose (%)	Hemicellulose (%)
120°C, 10 min	1.59	5.38 ± 0.05^{a,b}	57.66 ± 0.11^{b}	4.51 ± 0.02^{a,b}	15.38 ± 1.29^{a,b}	50.36 ± 1.13^{a}	22.44 ± 1.29^{a}
120°C, 30 min	2.07	5.21 ± 0.01^{a,b}	47.56 ± 0.14^{d}	5.61 ± 1.33^{b}	2.79 ± 0.64^{a}	52.01 ± 1.18^{a,b}	20.36 ± 0.64^{a,b}
180°C, 10 min	3.36	4.78 ± 0.04^{a,b}	54.56 ± 0.10^{c}	2.89 ± 0.86^{c}	16.86 ± 0.74^{b}	54.85 ± 1.27^{a,b}	18.55 ± 1.27^{a,b}
180°C, 30 min	3.83	4.00 ± 0.05^{c}	36.07 ± 0.04^{e}	3.66 ± 0.39^{a,c}	17.32 ± 2.06^{b}	57.79 ± 2.06^{b}	16.90 ± 1.59^{a}
Untreated	0.00	5.79 ± 0.05^{b}	100 ± 0.00^{a}	0 ± 0.00^{d}	16.19 ± 0.10^{b}	49.53 ± 4.96^{a}	24.43 ± 4.96^{b}

Note: Data in the table are exhibited in the form of mean ± standard deviation. Different lowercase letters indicate significant differences at $p<0.05$ using Tukey test.

Sugars Production

The SCW pre-treatment enhances biomass hydrolysis and organic matter breakdown during anaerobic digestion. Lignin, cellulose and hemicellulose are the main components in lignocellulosic biomass, whereas other extractive components may include ash, fats, protein and inorganics. Carbohydrate hydrolysis produces fermentable sugar, which can be used to produce biogas (Sarker *et al*., 2021). Cellulose and hemicellulose are transformed into monosaccharides (glucose and xylose), which can later facilitate microbial hydrolysis in anaerobic digestion (Saritpongteeraka *et al*., 2022).

In the present study, the concentration of glucose in the liquid fraction after SCW pre-treatment increased with increasing pre-treatment temperature (*Table 3*). A similar increasing trend was observed for cellobiose, arabinose, galactose, and mannose after SCW pre-treatment. Likewise, Akita *et al*. (2021) reported an increase in the concentration of glucose, cellobiose and mannose in the liquid fraction of pre-treated EFB along with the increasing temperature. Meanwhile, xylose composition was not detected at a lower temperature. A previous study revealed a similar result, as 346.5 mg L^{-1} of xylose was detected at 180°C for 45 min of pre-treatment time (Zerback *et al*., 2022).

Results also revealed an increase in the cellobiose concentration in the liquid fraction from 206.94-333.79 mg L^{-1}. Through SCW pre-treatment, the EFB is hydrolysed and subsequently, hemicellulose and cellulose are broken down into cellobiose (Kurnin *et al*., 2016). Since xylose cannot be detected in less severe conditions, furfural may be formed from the decomposition of arabinose (Zerback *et al*., 2022). As for arabinose, the highest concentration (1,023.37 mg L^{-1}) was detected at the highest temperature (180°C). These findings align with the results by Zerback *et al*. (2022), who reported an increasing trend of arabinose content in a liquid fraction when pre-treating wheat straw within a temperature range of 160°C-180°C. A previous study also indicated that xylose and glucose can be degraded to furans by dehydration cyclisation (He *et al*., 2022). Furans inhibit the biogas production process and are generated during the breakdown of hemicellulose into monosaccharides, oligosaccharides, and monomers (Dasgupta & Chandel, 2019). Furans may result in reduced biogas production in the anaerobic digestion process, depending on their availability and concentration levels throughout the process (Phuttaro *et al*., 2019).

Biogas Production

The impact of the SCW pre-treatment condition on co-digestion of EFB and POME is presented in *Figure 2*. The biogas production of the low-temperature groups (120°C) reached a significant peak on the 4th day after digestion started. The rapid digestion of the easily soluble components in EFB may be responsible for the rapid biogas generation, which shifted the peak from the 6th day of the untreated EFB to the 4th day. Compared to untreated EFB, at 180°C for 10 min of pre-treatment, the biogas continued to produce peak after the 26th day. The continuous peak observed might have resulted from the changes in the lignocellulosic structure of EFB due to the SCW pre-treatment, which appears to promote biogas production by changing the physical and chemical structure of pre-treated EFB (Kim *et al*., 2015). The second biogas peak at SCW pre-treatment of 180°C and 30 min was detected after the 16th day of the digestion period. Similarly, Xiang *et al*. (2021) experienced a delay in the biogas production of rice straw at high temperatures. The researchers reported that biogas production from rice straw pre-treated at a temperature of more than 180°C produced a second peak after the 14th to 15th day post-digestion.

Figure 3 depicts the improvements in SCW pre-treated EFB; there was no significant difference in the biogas output compared to those that were not SCW pre-treated at lower temperatures and for shorter periods (120°C, 10 min). This explains why the shorter reaction time and low temperature did not affect the biogas production from SCW pre-treated EFB. Previously, Park *et al*. (2021) observed that the reaction time of biogas produced at a temperature of 125°C was not significant and had no effects on biogas production. In addition to the water-to-solid ratio, Aili Hamzah *et al*. (2022) further highlighted that only the temperature of SCW pre-treatment reflected a significant impact on biogas yield.

TABLE 3. SUGAR YIELDS FROM THE RESIDUAL LIQUID FRACTION OF SCW PRE-TREATED EFB

Pre-treatment	Cellobiose (mg L^{-1})	Glucose (mg L^{-1})	Arabinose (mg L^{-1})	Galactose (mg L^{-1})	Mannose (mg L^{-1})	Xylose (mg L^{-1})
120°C, 10 min	206.94	335.05	520.10	416.38	ND	ND
120°C, 30 min	313.78	418.40	ND	449.72	656.30	ND
180°C, 10 min	315.09	637.68	1,028.37	505.08	ND	ND
180°C, 30 min	333.79	734.22	ND	513.77	2,405.24	400.08

Note: ND – not detected.

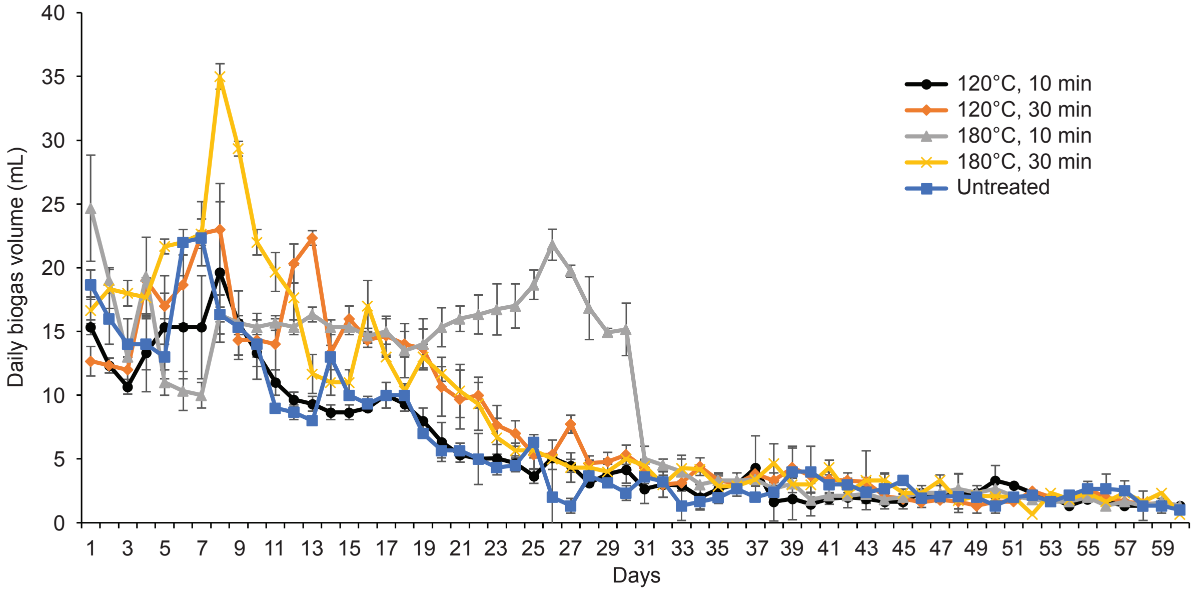

Figure 2. Daily biogas production of co-digestion of SCW pre-treated and untreated EFB with POME.

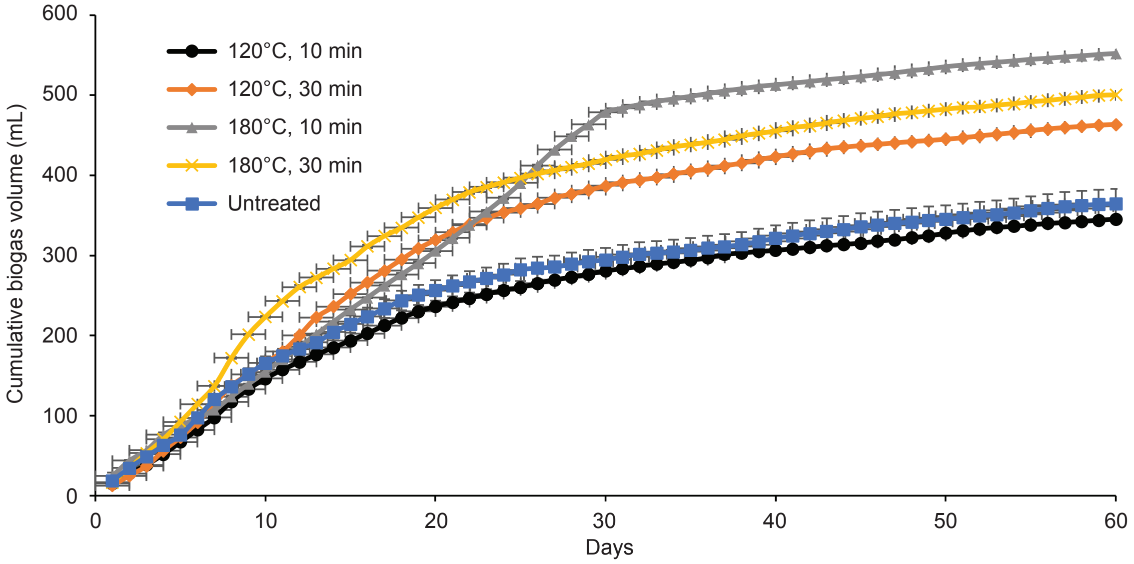

Figure 3. Cumulative biogas production of co-digestion of SCW pre-treated and untreated EFB with POME.

It can be seen from the data in *Table 4* that pre-treated EFB produced the highest biogas volume (547.27 mL), biogas yield (546.18 mL g^{-1} VS) and methane yield at 421.41 mL CH_4 g^{-1} VS, respectively. The results indicate that the aforementioned data for the pre-treated EFB were 32.0% higher than those for the untreated EFB. In addition, the methane content range of the pre-treated EFB was higher than that of the untreated EFB. Overall, the methane yield of SCW pre-treated EFB increased by 32.0% at 180°C compared to the untreated samples. In accordance with the present results, previous research has demonstrated that biogas production was only improved by 14.0% when rice straw was pre-treated to 180°C, while pre-treatment to 120°C led to a significant increment of biogas by 38.0% (Xiang *et al.*, 2021). In another study of hydrothermal SCW pre-treated acai processing waste and wheat straw, an increase in methane yield of 30.0% (Maciel-Silva *et al.*, 2019) and 32.0% (He *et al.*, 2022) was reported, respectively. These results further support an increase in the percentage of methane from 60.0%-85.0% in untreated to 92.0% in pre-treated. In another related study, methane production improved only by 63.4% upon pre-treatment of sludge at 180°C (Kim *et al.*, 2015). The percentage of methane after SCW pre-treatment observed in this study was higher (63.0%-92.0%) as compared to SCW pre-treated pineapple waste (75.0%-88.0%) reported by Aili Hamzah *et al.* (2022). Furthermore, SCW pre-treatment at a high temperature promotes biomass degradation during anaerobic digestion of EFB. This demonstrated that hydrothermal SCW pre-treatment increased the ability to fully utilise the EFB potential and increased biogas and methane yield.

Effluents Characteristics

A few parameters, including the initial and final effluent of the anaerobic digestion of SCW pre-treated EFB, were assessed to assure the stability of the anaerobic digestion process. *Table 5* summarises the pH, VS removal, and TAN at different pre-treatment conditions. The pH decreased slightly from its initial value of 7.0 to acidic. A decrease in pH in an anaerobic process can inhibit methanogens through rapid acidogenesis, thus contributing to low biogas production (Tepsour *et al.*, 2019; Wenjing *et al.*, 2019). None of the effluent pH values of the co-digestion of pre-treated EFB and POME differed significantly from the starting pH of 7.0. The final effluents can decrease to 6.78. Similarly, Tian *et al*. (2020) stated that the co-digestion of hydrothermal pre-treated waste recovered faster at 175°C when treated and achieved a suitable pH for methanogens, ranging from 6.8-7.2. The final TAN levels in the process ranged from 173.13 to 211.98 mg L^{-1}. In contrast, no TAN inhibition occurred in the present study since the maximum TAN did not exceed the inhibition level. Low biogas production often results from an increase in the TAN level above the threshold value, which inhibits the methanogenic activity. In a previous study involving hydrothermal SCW pre-treated sewage sludge, operating the pre-treatment under 175°C produced a lower TAN concentration and reduced TAN inhibition risk (Park *et al.*, 2021). The maximum VS removal from the co-digestion of SCW pre-treated EFB with POME was observed at 69.47%. This demonstrated that SCW pre-treatment aids in the degradation of organic matter into biogas by more than 50.00%. However, a prior study reported that fungal pre-treatment on EFB could achieve 60.00%-75.00% biodegradability (Suksong *et al.*, 2020). The lowest VS removal was reported in the untreated sample, for only 62.71% of VS removal. According to the higher VS removal reported by Gaballah *et al*. (2020), rape straw pre-treated with a steam explosion at 180°C combined with grinding resulted in 71.20% VS removal, followed by dilute acid and a steam explosion at 190°C with 70.60% VS removal.

TABLE 4. SUMMARY OF ANAEROBIC CO-DIGESTION PERFORMANCE OF SCW PRE-TREATED AND UNTREATED EFB WITH POME

Pre-treatment	Cumulative biogas volume	Cumulative biogas yield	Methane composition	Methane yield	Improvement
	(mL)	(mL g^{-1} VS)	(%)	(mL CH_4 g^{-1} VS)	(%)
120°C, 10 min	345.37 ± 35.69^{d}	299.96 ± 25.28^{c}	74-90	229.15 ± 17.22^{a}	-28
120°C, 30 min	463.70 ± 29.63^{c}	444.27 ± 36.14^{a}	72-90	$347.02 \pm 16.19^{a,c}$	8
180°C, 10 min	547.27 ± 20.73^{a}	546.18 ± 44.80^{b}	68-89	421.41 ± 15.85^{a}	32
180°C, 30 min	500.53 ± 9.49^{b}	$489.19 \pm 11.58^{a,b}$	63-92	$380.79 \pm 16.88^{a,b}$	19
Untreated	364.83 ± 5.62^{d}	323.7 ± 6.85^{c}	60-85	320.44 ± 11.32^{c}	0

Note: Data in the table are exhibited in the form of mean ± standard deviation. Different lowercase letters indicate significant differences at $p<0.05$ using Tukey test.

TABLE 5. SUMMARY OF INITIAL AND FINAL EFFLUENTS CHARACTERISTIC OF SCW PRE-TREATED AND UNTREATED EFB WITH POME

Pre-treatment	pH		TAN		VS		VS removal
			mg L^{-1}	mg L^{-1}	g VS L^{-1}	g VS L^{-1}	
	Initial	Final	Initial	Final	Initial	Final	
120°C, 10 min	7	6.96 ± 0.12^{a}	122.13 ± 9.28^{e}	194.13 ± 7.81^{b}	17	5.78 ± 0.22^{ab}	66.00 ± 1.29^{ab}
120°C, 30 min	7	7.00 ± 0.50^{a}	161.93 ± 5.15^{d}	173.13 ± 6.11^{d}	17	5.50 ± 0.58^{ab}	67.65 ± 3.35^{ab}
180°C, 10 min	7	6.98 ± 0.36^{a}	204.40 ± 6.86^{a}	192.73 ± 6.61^{c}	17	5.19 ± 0.61^{a}	69.47 ± 3.59^{ab}
180°C, 30 min	7	6.78 ± 0.12^{a}	197.80 ± 8.92^{b}	173.60 ± 8.51^{d}	17	5.71 ± 0.23^{ab}	66.41 ± 2.35^{ab}
Untreated	7	7.18 ± 0.39^{a}	190.43 ± 5.09^{c}	211.98 ± 5.11^{a}	17	6.34 ± 0.99^{b}	62.71 ± 5.82^{ab}

Note: Data in the table are exhibited in the form of mean ± standard deviation. Different lowercase letters indicate significant differences at $p<0.05$ using Tukey test.

Kinetic Study

It is assumed, using the modified Gompertz model (MGM) equation, that the rate at which biogas is produced in the digester equals a specific rate at which methanogenic bacteria grow. It can be seen in *Figure 4* that SCW pre-treated EFB at 180°C for 30 min produced faster biogas production compared to other pre-treatment conditions, while the slowest production can be seen at 120°C for 10 min and untreated. This finding suggests that the microbial growth rate is lower at low temperatures and shorter pre-treatment times. This is because lower temperatures result in the production of less fermentable sugar. High-temperature production of fermentable sugar enhances microbial growth and increases the availability of sucrose for conversion into methane. *Table 6* summarises the predicted parameters of MGM's equation. Despite having the maximum biogas production, there is an increase in SCW pre-treatment at 180°C for 30 min. Xiang *et al*. (2021) found that increasing the pre-treatment temperature from 120°C-180°C released more fermentable sugars that facilitate the anaerobic digestion process. Thus, an increase in the amount of fermentable sugar in the pre-treated sample increases the λ. These results reflect those of Hamzah *et al*. (2024), who also observed a similar trend. Also, the λ of the solid fraction hydrothermal SCW pre-treated rice straw increased from 3.49-5.25 days (Xiang *et al*., 2021), a slight increase in λ from 3.23-3.45 and 3.79 was observed by fungal pre-treatment of rice straw (Kainthola *et al*., 2019) and λ increased from 1 day to a maximum of 9.9 using acid and alkali pre-treatment for tobacco stalk (Zhang *et al*., 2020).

The maximum biogas production rate (μ) demonstrated an increasing trend as the SCW temperature and reaction time increased. The highest μ was observed at 180°C for 30 min with mL g^{-1} VS. day and the lowest μ at 120°C for 10 min with only 12.73 mL g^{-1} VS. day. An increase in the μ, from 12.33-14.75 mL g^{-1} VS. day, was observed when pre-treated rice straw was subjected to a temperature increase from 120°C-180°C (Xiang *et al*., 2021). The kinetic study is crucial in predicting, modelling, and monitoring the performance of a process under various conditions (Cao *et al*., 2020). The model assists in predicting kinetic parameters and elucidating the digestion process. The RMSE is a primary metric used to evaluate the effectiveness of a regression model and offers an assessment of the model's ability to accurately forecast the target value (accuracy) (Manogaran *et al*., 2023). It is widely acknowledged that a lower RMSE indicates superior model performance (Pečar *et al*., 2020). A lower RMSE signifies a more outstanding outcome of the model prediction. The RMSE values in the present study varied between 0.2416 and 0.8746. The coefficient of determination (R^2) values of the ratios for the pre-treated EFB were also higher in comparison with untreated materials. The R^2 of pre-treated EFB suggested good agreement between the experimental data and model simulation for all pre-treatments. The fact that the R^2 values were near to one further demonstrated that the kinetics of the MGM fit with the actual biogas generation. Previously, a good fit was observed using MGM for anaerobic co-digestion of chemically pre-treated agricultural waste and animal manure, reported at a range of 0.979-0.994 (Almomani & Bhosale, 2020), while hydrothermal SCW pre-treated rice

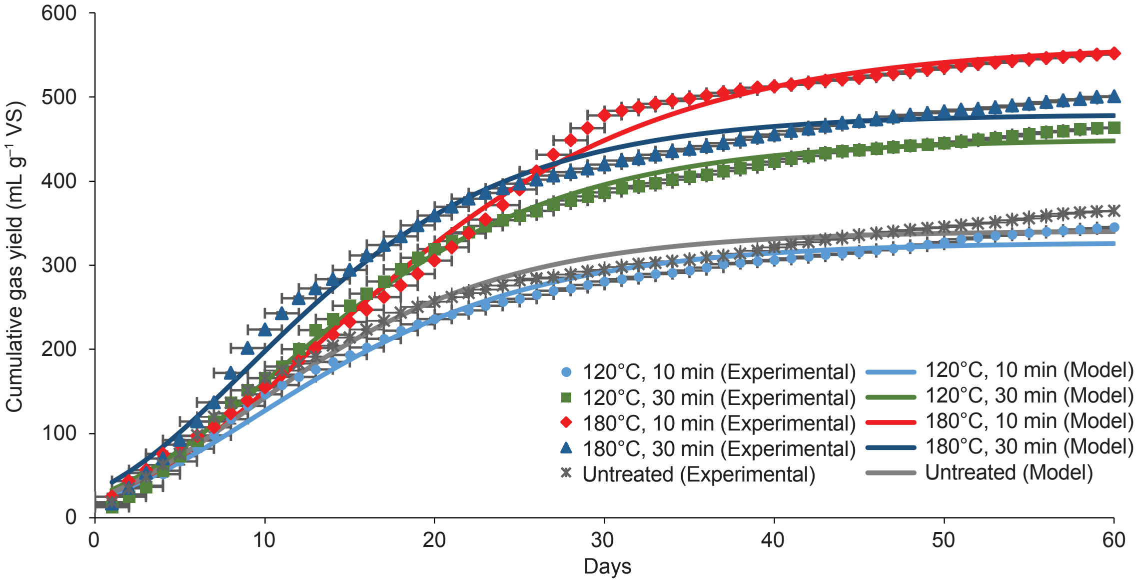

Figure 4. Cumulative biogas yield of co-digestion of SCW pre-treated EFB with POME.

TABLE 6. KINETIC PARAMETERS OF BIOGAS PRODUCTION DURING ANAEROBIC CO-DIGESTION - THE MODIFIED GOMPERTZ MODEL

Pre-treatment	120°C, 10 min	120°C, 30 min	180°C, 10 min	180°C, 30 min	Untreated
B_0 (mL g^{-1} VS)	327.37 ± 30.38^c	450.37 ± 28.97^b	562.28 ± 17.93^a	479.39 ± 7.40^b	341.04 ± 6.61^c
μ (mL g^{-1} VS day)	12.73 ± 1.79^c	$17.17 \pm 1.61^{a,b}$	18.25 ± 0.47^b	19.80 ± 0.25^b	$14.27 \pm 0.59^{a,c}$
λ (day)	0.00 ± 0.00^b	0.59 ± 0.40^b	1.82 ± 0.25^a	0.00 ± 0.11^b	0.00 ± 0.00^b
R^2	0.9874	0.9957	0.9953	0.9897	0.9805
RMSE	0.8567	0.7041	0.8724	0.2416	0.8746

Notes: B_0 - maximum biogas yield; μ - the maximum biogas production rate; λ - lag phase period; R^2 - coefficient of determination; RMSE - root mean square error. Data in the table are exhibited in the form of mean ± standard deviation. Different lowercase letters indicate significant differences at $p<0.05$ using Tukey test.

straw reported an increase in R^2 from 0.9944-0.9989 after being pre-treated at a SCW temperature of 150°C by using the same kinetic model (Wang *et al.*, 2018). The increase of R^2 values towards 1 and the gradual decrease of RMSE towards 0 imply a strong relationship between these models and the experimental results, suggesting a good fit (Hakimi *et al.*, 2023). Both the RMSE and the R^2 measure the degree to which a linear regression model accurately fits the experimental value. Thus, from this analysis, the MGM equation is suitable for the biogas prediction model because it has a low RMSE value and a good R^2 fit.

CONCLUSION

The impact of SCW pre-treatment process parameters on the methane production from co-digestion of EFB and POME was investigated in this study. The changes in the properties of EFB after pre-treatment depended on the SCW pre-treatment severity. Fermentable sugars such as glucose, xylose, mannose, cellobiose, and galactose were detected in the liquid fraction of SCW pre-treated EFB. High-temperature (180°C) SCW pre-treatment depicted better methane production than low-temperature pre-treatment. SCW pre-treatment at 180°C for 10 min achieved the maximum biogas volume of 547.27 mL, corresponding to 546.18 mL g^{-1} VS biogas yield and 421.41 mL CH_4 g^{-1} VS methane yields, respectively. Lignin reduction was observed in all pre-treated samples. Meanwhile, the SCW pre-treatment improved the VS removal and reduced the final TAN and pH of the anaerobic co-digestion process. Cumulative biogas production analysed by a modified Gompertz kinetic equation revealed that the longer λ observed at SCW pre-treatment produced the highest yield. This finding is likely to be related to the increase in the amount of fermentable sugar after the pre-treatments. This study demonstrated that SCW pre-treatment successfully increased methane production from the co-digestion of EFB and POME.

The SCW pre-treatment for a shorter time does not promote methane production. Though high-temperature (180°C) SCW pre-treatment depicted better methane production than low-temperature pre-treatment, a reduction in biogas production can be observed as the reaction time is prolonged to 30 min. The degradation of cellulose, hemicellulose, and lignin at SCW temperature, as well as the duration of the reaction, have an impact on the efficiency of biogas production, sugar yield, and the generation of inhibitory compounds. At elevated temperatures, there is an increased release of fermentable sugars, along with the potential presence of inhibitory compounds. The compounds exhibit toxicity toward the methanogens. Therefore, identification of the concentration of these compounds at high temperatures and longer reaction times is crucial. The integration of SCW pre-treatment can effectively remove lignin from lignocellulosic wastes, hence improving microbial hydrolysis. Selecting the SCW temperature and reaction time carefully is essential to maximising methane conversion and reducing inhibitor formation. Hence, there is still a need to investigate this pre-treatment method to achieve optimal methane production and the recovery of sugars while minimising the production of inhibitors.

ACKNOWLEDGEMENT

The authors are grateful for the research project under the Fundamental Research Grant Scheme (Ref. No: FRGS/1/2021/TK0/UPM/02/28) awarded by the Ministry of Higher Education Malaysia. The authors wish to acknowledge the support and technical facilities from the Faculty of Engineering, Universiti Putra Malaysia. The authors wish to thank Green Lagoon Technology Sdn. Bhd. for their kind assistance in raw material supply used in this study. The authors declare that they are free of any known conflicts of interest or direct personal connections that might have looked to have impacted the research presented in this study.

REFERENCES

Ahmad, F., Silva, E. L., & Varesche, M. B. A. (2018). Hydrothermal processing of biomass for anaerobic digestion – A review. *Renewable and Sustainable Energy Reviews*, *98*, 108–124. https://doi.org/10.1016/j.rser.2018.09.008

Aili Hamzah, A. F., Hamzah, M. H., Mazlan, N. I., Che Man, H., Jamali, N. S., Siajam, S. I., & Show, P. L. (2022). Optimization of subcritical water pre-treatment for biogas enhancement on co-digestion of pineapple waste and cow dung using the response surface methodology. *Waste Management*, *150*, 98–109. https://doi.org/10.1016/j.wasman.2022.06.042

Aili Hamzah, A. F., Hamzah, M. H., Che Man, H., Jamali, N. S., Siajam, S. I., & Show, P. L. (2023). Subcritical water pretreatment for anaerobic digestion enhancement: A review. *Pertanika Journal of Science & Technology*, *31*(2), 1011–1034. https://doi.org/10.47836/pjst.31.2.19

Akita, H., Yusoff, M. Z. M., & Fujimoto, S. (2021). Preparation of oil palm empty fruit bunch hydrolysate. *Fermentation*, *7*(2), 81. https://doi.org/10.3390/fermentation7020081

Almomani, F., & Bhosale, R. R. (2020). Enhancing the production of biogas through anaerobic co-digestion of agricultural waste and chemical pre-treatments. *Chemosphere*, *255*, 126805. https://doi.org/10.1016/j.chemosphere.2020.126805

Antwi, E., Engler, N., Nelles, M., & Schüch, A. (2019). Anaerobic digestion and the effect of hydrothermal pretreatment on the biogas yield of cocoa pods residues. *Waste Management*, *88*, 131–140. https://doi.org/10.1016/j.wasman.2019.03.034

American Public Health Association (APHA). (1998). *Standard methods for the examination of water and wastewater* (20th ed.)

Awoh, E. T., Kiplagat, J., Kimutai, S. K., & Mecha, A. C. (2023). Current trends in palm oil waste management: A comparative review of Cameroon and Malaysia. *Heliyon*, *9*(11), e21410. https://doi.org/10.1016/j.heliyon.2023.e21410

Cao, Z., Hülsemann, B., Wüst, D., Illi, L., Oechsner, H., & Kruse, A. (2020). Valorization of maize silage digestate from two-stage anaerobic digestion by hydrothermal carbonization. *Energy Conversion and Management*, *222*, 113218. https://doi.org/10.1016/j.enconman.2020.113218

Chan, Y. J., Lee, H. W., & Selvarajoo, A. (2021). Comparative study of the synergistic effect of decanter cake (DC) and empty fruit bunch (EFB) as the co-substrates in the anaerobic co-digestion (ACD) of palm oil mill effluent (POME). *Environmental Challenges*, *5*, 100257. https://doi.org/10.1016/j.envc.2021.100257

Chen, J., Wang, X., Zhang, B., Yang, Y., Song, Y., Zhang, F., Liu, B., Zhou, Y., Yi, Y., Shan, Y., & Lü, X. (2021). Integrating enzymatic hydrolysis into subcritical water pretreatment optimization for bioethanol production from wheat straw. *The Science of the Total Environment*, *770*, 145321. https://doi.org/10.1016/j.scitotenv.2021.145321

Dasgupta, A., & Chandel, M. K. (2019). Enhancement of biogas production from organic fraction of municipal solid waste using hydrothermal pretreatment. *Bioresource Technology Reports*, *7*, 100281. https://doi.org/10.1016/j.biteb.2019.100281

Dolah, R., Karnik, R., & Hamdan, H. (2021). A comprehensive review on biofuels from oil palm empty fruit bunch (EFB): Current status, potential, barriers and way forward. *Sustainability*, *13*(18), 10210. https://doi.org/10.3390/su131810210

Gaballah, E. S., Abomohra, A. E.-F., Xu, C., Elsayed, M., Abdelkader, T. K., Lin, J., & Yuan, Q. (2020). Enhancement of biogas production from rape straw using different co-pretreatment techniques and anaerobic co-digestion with cattle manure. *Bioresource Technology*, *309*, 123311. https://doi.org/10.1016/j.biortech.2020.12331

Hakimi, M., Manogaran, M. D., Shamsuddin, R., Johari, S. A. M., Hassan, M. A. M., & Soehartanto, T. (2023). Co-anaerobic digestion of sawdust and chicken manure with plant herbs: Biogas generation and kinetic study. *Heliyon*, *9*(6), e17096. https://doi.org/10.1016/j.heliyon.2023.e17096

Hamzah, A. F. A., Hamzah, M. H., Man, H. C., Jamali, N. S., Siajam, S. I., & Show, P. L. (2024). Biogas production through mono- and co-digestion of pineapple waste and cow dung at different substrate ratios. *BioEnergy Research*, *17*(2), 1179–1190. https://doi.org/10.1007/s12155-022-10478-2

He, C., Hu, J., Shen, F., Huang, M., Zhao, L., Zou, J., Tian, D., Jiang, Q., & Zeng, Y. (2022). Tuning hydrothermal pretreatment severity of wheat straw to match energy application scenarios. *Industrial Crops and Products*, *176*, 114326. https://doi.org/10.1016/j.indcrop.2021.114326

Jomnonkhaow, U., Sittijunda, S., & Reungsang, A. (2022). Assessment of organosolv, hydrothermal, and combined organosolv and hydrothermal with enzymatic pretreatment to increase the production of biogas from Napier grass and Napier silage. *Renewable Energy*, *181*, 1237–1249. https://doi.org/10.1016/j.renene.2021.09.099

Kainthola, J., Kalamdhad, A. S., Goud, V. V., & Goel, R. (2019). Fungal pretreatment and associated kinetics of rice straw hydrolysis to accelerate methane yield from anaerobic digestion. *Bioresource Technology*, *286*, 121368. https://doi.org/10.1016/j.biortech.2019.121368

Kim, D., Lee, K., & Park, K. Y. (2015). Enhancement of biogas production from anaerobic digestion of waste activated sludge by hydrothermal pre-treatment. *International Biodeterioration & Biodegradation*, *101*, 42–46. https://doi.org/10.1016/j.ibiod.2015.03.025

Kurnin, N. A. A., Ismail, M. H. S., Yoshida, H., & Izhar, S. (2016). Recovery of palm oil and valuable material from oil palm empty fruit bunch by sub-critical water. *Journal of Oleo Science*, *65*(4), 283–289. https://doi.org/10.5650/jos.ess15209

Lee, J., & Park, K. Y. (2020). Impact of hydrothermal pretreatment on anaerobic digestion efficiency for lignocellulosic biomass: Influence of pretreatment temperature on the formation of biomass-degrading byproducts. *Chemosphere*, *256*, 127116. https://doi.org/10.1016/j.chemosphere.2020.127116

Liew, Z. K., Chan, Y. J., Ho, Z. T., Yip, Y. H., Teng, M. C., Ameer Abbas, B. A. I. T., Chong, S., Show, P. L., & Chew, C. L. (2021). Biogas production enhancement by co-digestion of empty fruit bunch (EFB) with palm oil mill effluent (POME): Performance and kinetic evaluation. *Renewable Energy*, *179*, 766–777. https://doi.org/10.1016/j.renene.2021.07.073

Lim, Y. F., Chan, Y. J., Hue, F. S., Ng, S. C., & Hashma, H. (2021). Anaerobic co-digestion of palm oil mill effluent (POME) with decanter cake (DC): Effect of mixing ratio and kinetic study. *Bioresource Technology Reports*, *15*, 100736. https://doi.org/10.1016/j.biteb.2021.100736

Maciel-Silva, F. W., Mussatto, S. I., & Forster-Carneiro, T. (2019). Integration of subcritical water pretreatment and anaerobic digestion technologies for valorization of açai processing industries residues. *Journal of Cleaner Production*, *228*, 1131–1142. https://doi.org/10.1016/j.jclepro.2019.04.362

Mahmod, S. S., Jahim, J. M., Abdul, P. M., Luthfi, A. A. I., & Takriff, M. S. (2021). Techno-economic analysis of two-stage anaerobic system for biohydrogen and biomethane production from palm oil mill effluent. *Journal of Environmental Chemical Engineering*, *9*(4), 105679. https://doi.org/10.1016/j.jece.2021.105679

Mamimin, C., Chanthong, S., Leamdum, C., O-Thong, S., & Prasertsan, P. (2021). Improvement of empty palm fruit bunches biodegradability and biogas production by integrating the straw mushroom cultivation as a pretreatment in the solid-state anaerobic digestion. *Bioresource Technology*, *319*, 124227. https://doi.org/10.1016/j.biortech.2020.124227

Manogaran, M. D., Hakimi, M., Ahmad, M. H. N. B., Shamsuddin, R., Lim, J. W., Hassan, M. A. M., & Sahrin, N. T. (2023). Effect of temperature on co-anaerobic digestion of chicken manure and empty fruit bunch: A kinetic parametric study. *Sustainability*, *15*(7), 5813. https://doi.org/10.3390/su15075813

Park, M., Kim, N., Jung, S., Jeong, T., & Park, D. (2021). Optimization and comparison of methane production and residual characteristics in mesophilic anaerobic digestion of sewage sludge by hydrothermal treatment. *Chemosphere*, *264*, 128516. https://doi.org/10.1016/j.chemosphere.2020.128516

Pečar, D., Pohleven, F., & Goršek, A. (2020). Kinetics of methane production during anaerobic fermentation of chicken manure with sawdust and fungi pre-treated wheat straw. *Waste Management*, *102*, 170–178. https://doi.org/10.1016/j.wasman.2019.10.046

Phuttaro, C., Sawatdeenarunat, C., Surendra, K., Boonsawang, P., Chaiprapat, S., & Khanal, S. K. (2019). Anaerobic digestion of hydrothermally-pretreated lignocellulosic biomass: Influence of pretreatment temperatures, inhibitors and soluble organics on methane yield. *Bioresource Technology*, *284*, 128–138. https://doi.org/10.1016/j.biortech.2019.03.114

Saritpongteeraka, K., Natisupacheevin, K., Tan, C., Rehman, S., Charnnok, B., Vaurs, L. P., Leu, S., & Chaiprapat, S. (2021). Comparative assessment between hydrothermal treatment and anaerobic digestion as fuel pretreatment for industrial conversion of oil palm empty fruit bunch to methane and electricity - A preparation study to full scale. *Fuel*, *310*, 122479. https://doi.org/10.1016/j.fuel.2021.122479

Sarker, T. R., Pattnaik, F., Nanda, S., Dalai, A. K., Meda, V., & Naik, S. (2021). Hydrothermal pretreatment technologies for lignocellulosic biomass: A review of steam explosion and subcritical water hydrolysis. *Chemosphere*, *284*, 131372. https://doi.org/10.1016/j.chemosphere.2021.131372

Simanungkalit, S. P., Mansur, D., Nurhakim, B., Agustin, A., Rinaldi, N., Muryanto, N., & Fitriady, M. A. (2017). Hydrothermal pretreatment of palm oil empty fruit bunch. *AIP Conference Proceedings*, *1803*, 020011. https://doi.org/10.1063/1.4973138

Sitthikitpanya, S., Reungsang, A., & Prasertsan, P. (2018). Two-stage thermophilic bio-hydrogen and methane production from lime-pretreated oil palm trunk by simultaneous saccharification and fermentation. *International Journal of Hydrogen Energy*, *43*(9), 4284–4293. https://doi.org/10.1016/j.ijhydene.2018.01.063

Suksong, W., Wongfaed, N., Sangsri, B., Kongjan, P., Prasertsan, P., Podmirseg, S. M., Insam, H., & O-Thong, S. (2020). Enhanced solid-state biomethanisation of oil palm empty fruit bunches following fungal pretreatment. *Industrial Crops and Products*, *145*, 112099. https://doi.org/10.1016/j.indcrop.2020.112099

Sulaiman, N. S., Sintang, M. D., Mantihal, S., Zaini, H. M., Munsu, E., Mamat, H., Kanagaratnam, S., Jahurul, M. H. A., & Pindi, W. (2022). Balancing functional and health benefits of food products formulated with palm oil as oil sources. *Heliyon*, *8*(10), e11041. https://doi.org/10.1016/j.heliyon.2022.e11041

Tan, Y. D., & Lim, J. S. (2019). Feasibility of palm oil mill effluent elimination towards sustainable Malaysian palm oil industry. *Renewable and Sustainable Energy Reviews*, *111*, 507–522. https://doi.org/10.1016/j.rser.2019.05.043

Technical Association of the Pulp and Paper Industry. (TAPPI). (1950). *T.A.P.P.I. standards: Testing methods, recommended practices, specifications of the Technical Association of the Pulp and Paper Industry*.

Tepsour, M., Usmanbaha, N., Rattanaya, T., Jariyaboon, R., O-Thong, S., Prasertsan, P., & Kongjan, P. (2019). Biogas production from oil palm empty fruit bunches and palm oil decanter cake using solid-state anaerobic co-digestion. *Energies*, *12*(22), 4368. https://doi.org/10.3390/en12224368

Tian, W., Chen, Y., Shen, Y., Zhong, C., Gao, M., Shi, D., He, Q., & Gu, L. (2020). Effects of hydrothermal pretreatment on the mono- and co-digestion of waste activated sludge and wheat straw. *The Science of the Total Environment*, *732*, 139312. https://doi.org/10.1016/j.scitotenv.2020.139312

Vakalis, S., Georgiou, A., Moustakas, K., & Fountoulakis, M. (2022). Assessing the effect of hydrothermal treatment on the volatile solids content and the biomethane potential of common reed (*phragmites australis*). *Bioresource Technology Reports*, *17*, 100923. https://doi.org/10.1016/j.biteb.2021.100923

Wang, D., Shen, F., Yang, G., Zhang, Y., Deng, S., Zhang, J., Zeng, Y., Luo, T., & Mei, Z. (2018). Can hydrothermal pretreatment improve anaerobic digestion for biogas from lignocellulosic biomass? *Bioresource Technology*, *249*, 117–124. https://doi.org/10.1016/j.biortech.2017.09.197

Wenjing, L., Chao, P., Lama, A., Xindi, F., Rong, Y., & Dhar, B. R. (2019). Effect of pre-treatments on biological methane potential of dewatered sewage sludge under dry anaerobic digestion. *Ultrasonics Sonochemistry*, *52*, 224–231. https://doi.org/10.1016/j.ultsonch.2018.11.022

Xiang, C., Tian, D., Hu, J., Huang, M., Shen, F., Zhang, Y., Yang, G., Zeng, Y., & Deng, S. (2021). Why can hydrothermally pretreating lignocellulose in low severities improve anaerobic digestion performances? *The Science of the Total Environment*, *752*, 141929. https://doi.org/10.1016/j.scitotenv.2020.141929

Zerback, T., Schumacher, B., Weinrich, S., Hülsemann, B., & Nelles, M. (2022). Hydrothermal pretreatment of wheat straw – Evaluating the effect of substrate disintegration on the digestibility in anaerobic digestion. *Processes*, *10*(6), 1048. https://doi.org/10.3390/pr10061048

Zhang, H., Wang, L., Dai, Z., Zhang, R., Chen, C., & Liu, G. (2019). Effect of organic loading, feed-to-inoculum ratio, and pretreatment on the anaerobic digestion of tobacco stalks. *Bioresource Technology*, *298*, 122474. https://doi.org/10.1016/j.biortech.2019.122474