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ORIGINAL ARTICLE

Synergistic effect of ultrasonic and microwave pretreatment on improved biohydrogen generation from palm oil mill effluent

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Abstract

This study aims to investigate the effect of the ultrasonic, microwave, and combined ultrasonic-microwave pretreatment of palm oil mill effluent (POME) sludge before fermentation to analyze hydrogen (H_2) production and chemical oxygen demand (COD) removal efficiency in batch fermentation. Experimental results showed that the pretreatment of sludge (ultrasonication, microwave, and ultrasonication-microwave pretreatment) had positively influence the H_2 production and COD removal efficiency during fermentation as compared to control one (without any pretreatment). Combined ultrasonication-microwave pretreatment of POME sludge was shown to be more effective for increasing both H_2 production and COD removal from POME with the highest cumulative H_2 and COD removal efficiency of 4080 mL H_2/L-POME and 75.56%, respectively. The improvement observed for cumulative H_2 production and COD removal was 12.14% and 21.42%, respectively, compared to the control one. These observations concluded that the POME sludge pretreatment with ultrasonication-microwave irradiation could be an effective strategy for improved treatment of POME with simultaneous production of H_2.

Keywords Ultrasonication · Microwave · Pretreatment · Hydrogen · Palm oil mill effluent · Sludge

1 Introduction

The biological transformation of carbohydrate-rich agro-industrial waste to the low carbon energy biofuel has been considered a promising approach to produce a potential value-added product with the simultaneous treatment of waste [13, 21, 49]. Hydrogen (H_2) (the cleanest energy carrier with intrinsic high combustion calorific value of 143 MJ·Kg^{-1} than any other hydrocarbons) production by dark fermentative processes using palm oil mill effluent (POME) wastewater as a source of carbon has been studied by various researchers in recent years [20, 32, 48]. The maximum theoretical stoichiometric yield for fermentative H_2 production from glucose is 12 mol· H_2·mol^{-1}glucose [33]. However, this maximum H_2 yield is still limited by the selective conditions of the fermentative process which includes the pretreatment of biomass, types of inoculum (pure or mixed culture), type of reactors, operating parameters (pH, temperature, substrate type) [35, 50], etc.

Malaysia is the second largest contributor of crude palm oil in the world by exporting 1,687,558 t of palm oil in the period of Jan–Dec 2018 [4, 54]. Nevertheless, the production of 1 t of crude palm oil requires 6–8 t of water, and over 50% of the water ends up as toxic and higher organic containing wastewater [9, 31]. The presence of high organic matter content in POME marks it as the most polluting wastewater in the world but in parallel an ideal substrate for fermentative hydrogen production. A typical POME is physio-chemically characterized as chemical oxygen demand (COD): 45,000–65,000 mg/L; biological oxygen demand (BOD): 18,000–50,000 mg/L; pH: 3.53–4.52; and oil and grease: 0.72–1.03% [36, 38]. Although the raw POME is used as a source for biogas production, the complex composition of POME increases the processing time and limited the maximum H_2 productivity. In this scenario, the

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implementation of pretreated sludge before anaerobic digestion is considered as sustainable and efficient method to increase H_2 productivity [3]. Different pretreatment strategies (biological and physicochemical) have been employed for high-strength agro-industrial wastewater and sludges to increase fermentative H_2 production [27, 30]. Currently, biological and physio-chemical pretreatment processes employed for POME include deoiling [42], sedimentation [61], pre-hydrolysis [11], acid pre-treatment [28], alkali pretreatment [37], ozonation pretreatment [8], peroxidation pretreatment [25], microwave irradiation [45], coagulation [41], etc., and these are evidenced for successful increment in biogas productivities.

The development of sonochemistry prevails a promising pretreatment of industrial and domestic wastewater to increase biodegradability and reduces toxicity with ultrasonic energy [5, 44]. The accelerative effect of ultrasonication on increased digestibility by sludge disintegration, COD solubilization in anaerobic digestion, and process stabilization have been reported in various literatures [10, 56]. Besides, the electromagnetic radiations (oscillation frequency of 0.3 to 300 GHz) of the microwave have also been applied for the pretreatment processes [40]. Although the quantum of energy applied to microwave irradiation is not capable of breaking down chemical bonds, H_2 bonds can be broken [53]. These pretreatment processes disintegrate the particle sizes and help accelerate the hydrolysis of sludge solids which subsequently improves the H_2 production from complex cellulosic organic wastewaters. Nonetheless, studies have focused on pretreatment of POME before dark fermentative H_2 production, with some of them reporting enhanced H_2 production by following physio-chemical pretreatment methods [2, 24, 28, 37, 47]. As compared to untreated sludge, the pretreatment of sludges before fermentation has significantly increase the organic matter biodegradability. However, the pretreatment efficiency is highly influenced by the nature of pretreatment methods as well as the composition of sludge. Therefore, it is necessary to test the impact of various pretreatment strategies of sludge on H_2 production through anaerobic digestion. There is little information that exists on the best pretreatment method to produce H_2 by enriching H_2-producing microbes from POME sludge. Therefore, in the present study, the POME sludge was pretreated before fermentation by ultrasonication, microwave, and combined ultrasonic-microwave to assess its effect on hydrogen production and COD removal using POME as a substrate.

2 Material and methods

2.1 Substrate and inoculum

The POME sludge and wastewater were taken from an anaerobic pond (1 m below from the sludge-pond surface) and fresh outlet, respectively, from the Felda palm oil industry, Lepar Hill, Gambang, Pahang, Malaysia. The collected POME was initially filtered by passing through a laboratory sieve (aperture 0.15/R20 cm 100 mesh) to remove major solid and coarse aggregates. The collected material was preserved in a laboratory fridge at 4 °C before experimentation to decrease microbial acidification and degradation [51]. Before fermentation the POME was sterilized by autoclaving at 121 °C for 20 min to ensure the removal of indigenous microorganisms. The POME was analyzed for COD, biochemical oxygen demand (BOD), pH, volatile solid (VS), acetate and butyrate concentration, and ethanol concentrations prior and after treatment, as depicted in Tables 1 and 2.

2.2 Sludge pretreatment

Three pretreatment processes were applied to POME sludge which include ultrasonication, microwave, and ultrasonication-microwave pretreatment. These pretreated sludges were further used as seed inoculum for fermentative H_2 production from sterilized POME.

(a). Ultrasonication: Sonication of sludge was performed using a probe-type ultrasonic sonicator (Qsonica, USA). Ultrasonic irradiation was performed at 500 W at 20 kHz. The tip of the probe was placed in the center of the sludge medium. The applied amplitude and ultrasonication time were 75% and 25 min, respectively [1, 17].

(b). Microwave pretreatment: Seed sludge was irradiated with microwave using a laboratory microwave (Transform MW680, Thailand). Microwave irradiation was applied to the sludge for 40 min at 160 °C as suggested in the literature [39].

2.3 Experimental setup

The experimentations were carried out in a series of 1 L scott bottles which served as batch reactors in the present study.

Table 1 Characteristics of palm oil mill effluent

Characteristics (mg/L)	Raw POME
COD	72,500±130
BOD	64,440±110
pH	5.2±0.2
VS	14,300±55
*Acetate	1.40±0.13
*Butyrate	2.7±0.21
*Ethanol	0.43±0.03

*Unit in g/L

COD chemical oxygen demand, *BOD* biological oxygen demand, *VS* volatile solid

Table 2 COD mass balance for fermentations

Parameters	Control	Ultrasonic pretreated	Microwave pretreated	Ultrasonic-microwave pretreated
$COD_{initial}$ (mg/L)	72,500	72,500	72,500	72,500
COD_{final} (mg/L)	32,150	28,130	21,150	17,980
COD removal (%)	55.65	61.27	70.82	75.20
H_2 yield (mol)	0.151	0.165	0.156	0.183
[a]H_2 mg COD/L	2416	2640	2496	2928
Acetate (mg COD/L)	1432	1542	1751	1771
Butyrate (mg COD/L)	2891	3984	3146	3978
Ethanol (mg COD/L)	1280	1970	1410	1780
VS (mg/L)	3600	2450	2450	2115
[b]COD mass balance (%)	83.97	82.64	84.47	82.66

[a] Calculated based on 16 g COD/mol H_2

[b] COD mass balance (%) = 100*[Total SMPs (mg COD) + VSS (mg COD) + H_2 (mg COD)]/[$COD_{initial}$ (mg COD)]

Seed inoculum of 30% v/v (210 mL) was added to each rector obtaining the final working volume of 700 mL. The pretreated sludge samples in different scott bottles were subjected to the aforementioned pretreatments. Before inoculation of seed sludge to the POME, 0.50% glucose along with 0.20% NH_4HCO_3 was added to sterilized POME for initial bacterial acclimatization [34]. The run was conducted at 35 ± 2 °C and initial pH of 5.50 (adjusted by adding 1 N NaOH), which is reported to be favorable for H_2 production [37]. The headspace of reactors was flushed with nitrogen gas (99.60% purity) to reach anaerobic conditions and sealed properly. The entire experiment lasted for 225 h and was performed on duplicates to get reproducibility of the results.

2.4 Analytical

The biogas production rate was measured every 24 h of interval using water displacement through a graduated cylinder filled with acidic water to reduce the dissolution of gas components [51]. The composition of produced gas was analyzed using a gas chromatograph (GCMS-QP, 2010; Shimadzu, Japan), equipped with the thermal conductive detector (GC-TCD) and a 0.2 m × 3 mm diameter Porapak Q stainless steel column. The operational parameters of GC were set according to our previous literature [33]. Nitrogen gas at a flow rate of 3.50 mL/min was used as a carrier gas. The soluble metabolites including volatile fatty acids (VFAs) were analyzed using high-performance liquid chromatography (Agilent 1200) equipped with a C-18 column and refractive index detector (RID). KH_2PO_4 with 0.05 M was used for the mobile phase with a flow rate of 0.50 mL/min [23]. The sample volume of 15 μL was injected into the column at a constant temperature of 55 °C. Besides, COD, BOD, and VS analysis was carried out by following methods recommended by APHA standard methods [43]. The H_2 yield was calculated by using the total volume of hydrogen produced in each batch of experiments over the amount of COD consumed from the POME.

3 Results and discussion

The H_2 production test was carried out using ultrasonic, microwave, and ultrasonic-microwave pretreated sludge and POME as substrate. The H_2 production rate is an index to evaluate the H_2 production performance by biological means [58]. Upon the comparison of effects of ultrasonication irradiation and microwave pretreatment POME with control (without pretreatment), it can be observed from Fig. 1 that differences in H_2 production rate for both pretreatment are apparent. It can be observed that pretreatment of sludge brings a positive effect on biodegradability which is confirmed by improved H_2 production rate compared with the control one. The marked increase of 29 mLH_2/h (control: 26 mLH_2/h) was observed when sludge was subsequently pretreated with ultrasonication-microwave, and the highest COD reduction of 75.56% is apparent for the highest H_2 rate at this scenario. On contrary, the ultrasonicated sludge had the lowest H_2 production rate of 24 mLH_2/h. The microwave pretreated sludge showed the maximum H_2 production rate of 26 mLH_2/h. These variations in production rate can be explained by the fact that the physical pretreatment of sludge before anaerobic digestion increases the sludge solubility as well as activates acidogenic bacteria more efficiently [44]. These observations are consistent with the earlier reports, where the combined approach of microwave-ultrasonic has increase the performance of anaerobic digestion significantly [46].

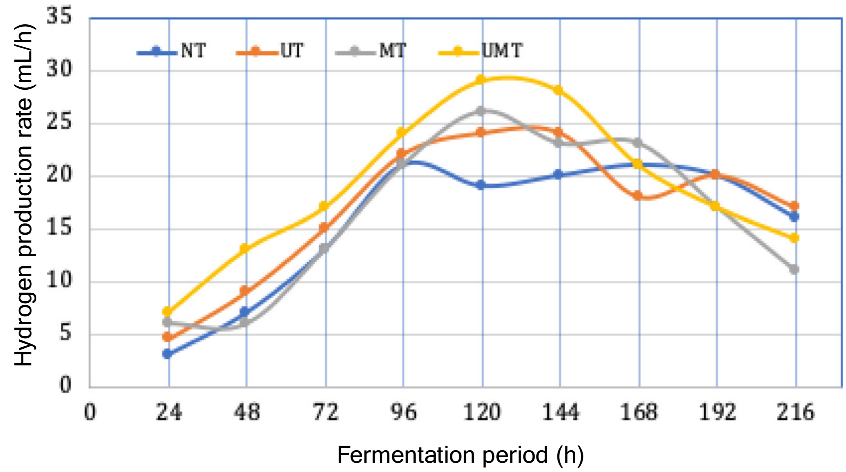

Fig. 1 Hydrogen production rate profile at different pretreatment (NT, nonpretreated (control); UT, ultrasonication pretreated; MT, microwave pretreatment; UMT, combined ultrasonication-microwave pretreatment)

The biological H_2 production is accompanied with the formation of VFAs (acetate, butyrate, propionate) in the fermentation period [7]. The fermentative microbes hydrolyze and ferment carbohydrates, proteins, and lipids to VFAs, which are further converted into acetate/butyrate and H_2 by acetogenic bacteria. In this study, the effect of sludge pretreatments on the pattern of VFA production can be seen in Fig. 2. Initially, the observed concentrations of acetate (at 24 h) in control, ultrasonicated, microwave, and ultrasonic-microwave pretreated were 0.32, 0.41, 0.41, and 0.31 g/L POME, respectively. The observed concentrations after 216 h of fermentation for the same were 14.36, 15.42, 17.59, and 17.77 g/L. Similar trend is observed for the butyrate concentrations, during the fermentation period at 24 h, were 1.42, 0.79, 1.24, and 1.43 g/L which increases after 216 h of the fermentation as 28.91, 39.84, 31.5, and 39.78 g/L for control, ultrasonicated, microwave and ultrasonic-microwave pretreated, respectively. These results suggest that pretreatment of sludge influences VFA production, which results in an improvement in H_2 production. Throughout the fermentation period, butyrate concentrations were dominated over the acetate in all sets of experiments. This suggests that the microbial consortium in sludge has followed the butyrate type of fermentative H_2 production as illustrated in Eq. (1). Ismail et al. demonstrated a similar pattern of VFA production profile in the study of H_2 production with anaerobic sludge from POME [18]. In another study, Sompong et al. reported similar results where the butyrate production dominated over the other metabolites during H_2 production from POME seeded with thermophilic microflora [52]. Besides being less significant than acetate and

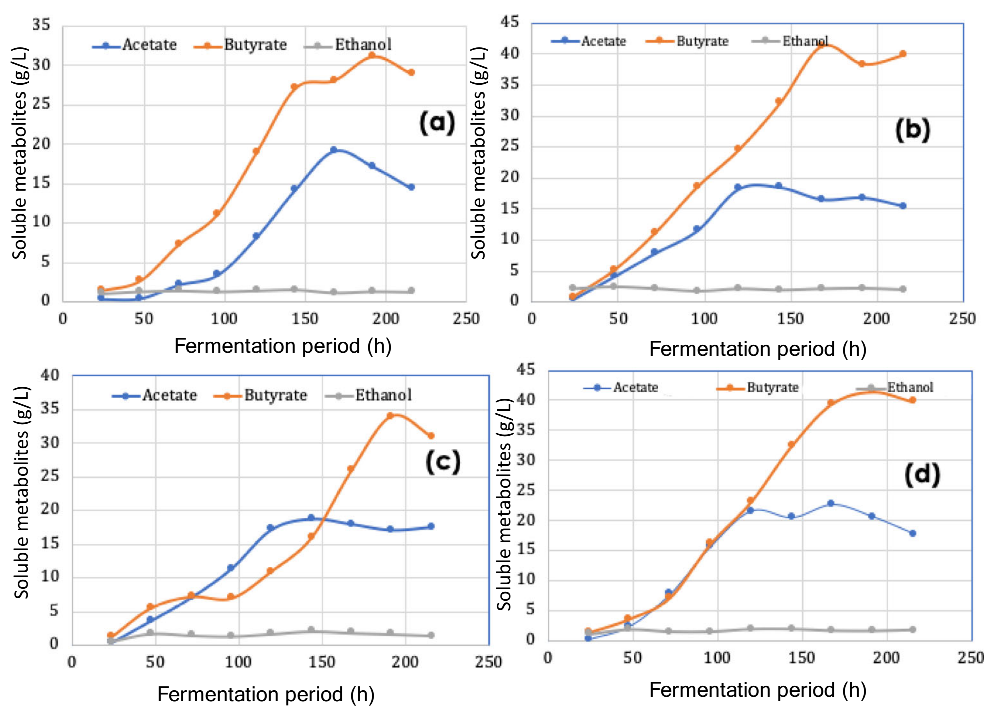

Fig. 2 Effect of pretreatment on soluble metabolite production during fermentation period (**a**) nonpretreated (control), (**b**) ultrasonicated, (**c**) microwave pretreated, and (**d**) ultrasonic-microwave pretreated

butyrate, ethanol in each fermentative reactor was observed, e.g., in control it ranged between 1.10 and 1.28 g/L, while using pretreated sludge in POME showed in the range 0.49 to 1.97 g/L which is also considerable.

$$C_6\ H_{12}O_6 \rightarrow CH_3\ CH_2\ CH_2COO^- + 2HCO_3^- + 3H \qquad (1)$$

The differences in pretreated sludge effects on a physical-chemical characteristic of POME after treatment, especially in terms of COD, BOD, pH, VS, acetate, butyrate, and ethanol dissolved in POME, indicate the microbial activities during the fermentation [12]. COD removal concentrations observed for control, ultrasonic pretreated, ultrasonic pretreated, and ultrasonic-microwave pretreated were 32,150 ± 70, 28,130 ± 110, 21,150 ± 130, and 17,980 ± 80 mg/L, respectively. BOD observation followed similar trends where it observed 29,150 ± 40, 19,500 ± 65, 17,250 ± 110, and 13,250 ± 70 mg/L for control, ultrasonic pretreated, and ultrasonic-microwave pretreated, respectively.

The higher BOD consumption and soluble metabolite production by ultrasonic-microwave pretreated sludge could be attributed to the selective microorganism selection by sludge pretreatment that properly adapted to the substrate and fermentative operational conditions. Metabolites such as ethanol production during fermentation were observed as 1.28 ± 0.20, 1.97 ± 0.25, 1.41 ± 0.22, and 1.78 ± 0.20 g/L for control, ultrasonic pretreated, ultrasonic-microwave pretreated, respectively. Despite this, the changes in pH of the fermentative medium reflect the efficiency of fermentative microbes. The final pH observed for ultrasonic pretreated, ultrasonic-microwave pretreated were 4.01 ± 0.30, 4.17 ± 0.25, and 3.97 ± 0.40, respectively, where the control account for final pH was 4.37 ± 0.35. This drop of pH in the medium after fermentation could be attributed to the accumulation of the metabolite which was produced during operations. In order to provide more explanation about sludge pretreatment effect on H_2 production and organic matter consumption, COD removal percentage was observed for each set of experimentation as illustrated in Fig. 3. It showed that the percentage of COD removal is influenced by the pretreatment strategies employed on sludges for H_2 production. Ultrasonic pretreatment has recorded 61% of COD reduction, compared to control one which was accounted for 55.65%. The microwave pretreated sludge has removed 70% of COD from POME, whereas the subsequent ultrasonic-microwave treated sludge resulted maximum COD removal percentage of 75.56. Results showed that the pretreatment of sludge can increase the COD consumption up to 15%. Grady et al. [16] has suggested that COD can be used as a parameter for organic waste conversion measurement in an anaerobic process related to biogas production [16]. To analyze the H_2 production during experimentations, the calculations of COD mass balance in the form of measurable parameters was undertaken for the organic matter fluxes (before the experiment and after experiments). Acetate and butyrate were predominated in the fermentative medium, while ethanol was also observed in the 1.20 to 1.70 g/L. The maximum H_2 yield of 0.18 mol.H_2 was observed when ultrasonic-microwave pretreated sludge was used as fermentative inoculum. The calculations of COD mass balance were based on experimental data which includes the presence of soluble metabolites, VS and H_2 (as 1 mol of H_2 at STP represents COD mass of 16 g COD/mol H_2) ([55]; G. [22]). As shown in Table 2, it was found that the COD mass balance was lower than 100% (w/w), i.e., in the range of 82 to 84%. This closure mass balance emphasizes data reliability.

The combined ultrasonic-microwave pretreatment was shown to be the most effective pretreatment enhancing H_2 production from POME which appeared to coincide with enhanced cumulative H_2 production. The cumulative H_2 production of each batch fermentation was 3360, 3684, 3504 and 4080 mL for control, ultrasonicated, microwave and combined ultrasonic-microwave pretreated sludge, respectively (Fig. 3).

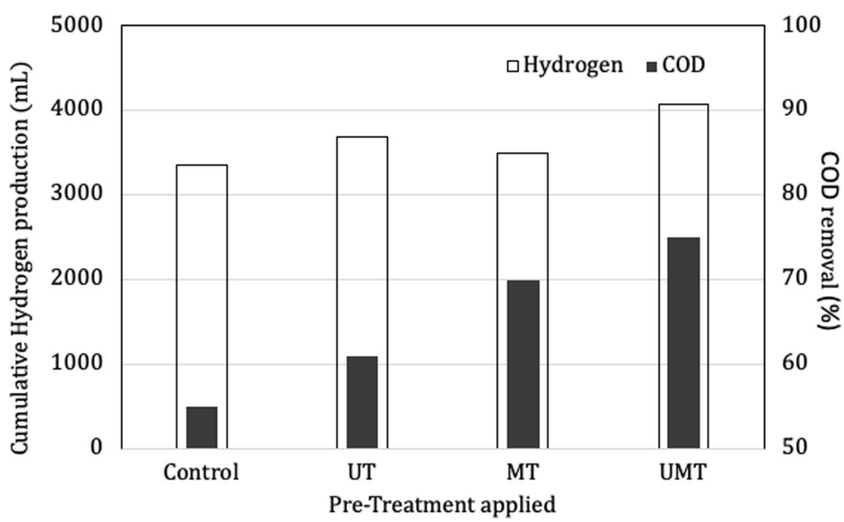

Fig. 3 Cumulative hydrogen production and COD reduction profile (NT, nonpretreated (control); UT, ultrasonication pretreated; MT, microwave pretreatment; UMT, combined ultrasonication-microwave pretreatment)

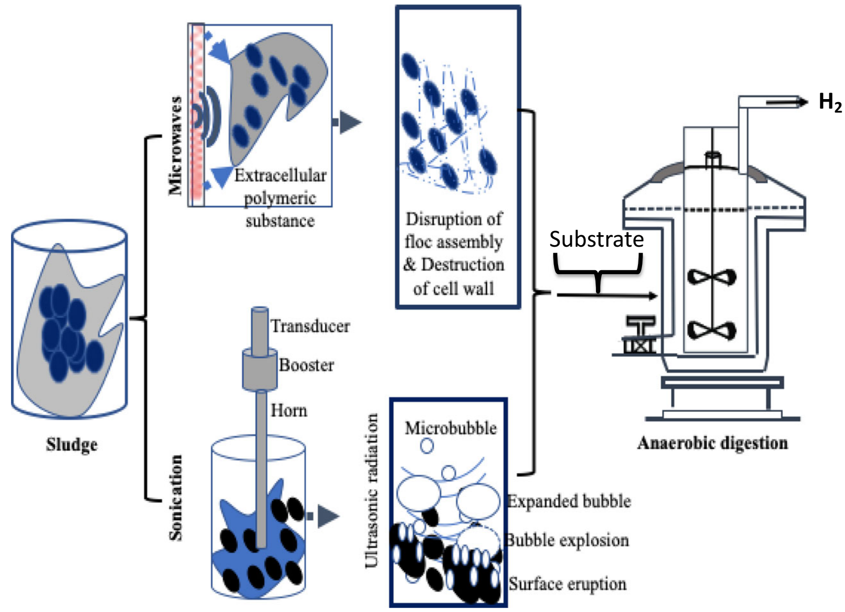

Fig. 4 Possible impact of sludge pretreatment on biohydrogen production

The combined ultrasonic-microwave pretreated improved the cumulative H_2 by 13% compared to control. These results suggest that pretreatment of sludge could be an important strategy to accelerate anaerobic biodegradability and disintegration of lignocellulosic biomasses, which is considered as one rate-limiting step and augments the fermentative H_2 production (Fig. 4) [26, 29]. The observed results are quite consistent with those in H_2 production from industrial wastewater, in which the substrate was pretreated ultrasonically followed by microwave and reported the improved H_2 productivity [6]. Elbeshbishy et al. demonstrated a similar pattern in a the study where they concluded that the ultrasonication pretreatment promotes the release of carbohydrate and protein into the liquid phase of the substrate, which causes increases in H_2 productivity [14]. In another study, a synergistic effect of ultrasonication pretreatment on biodegradability on dairy wastewater has been reported and observed a twofold increase in H_2 production in comparison with a controlled one [15]. Similarly, microwave irradiation pretreatment of wastewater is advisable as it was established that the microwave pretreatment causes polarized macromolecules alignment with the poles of the electromagnetic field, which results in the disruption of existing H_2 bonds and simultaneously increases the substrate availability [57]. Microwave pretreatment comprises the thermal and the non-thermal effect, which has been used for microbial disruption of waste activated sludge and reported the improved solubilization of waste and enhanced H_2 production [60]. Similar to observed results in the present study, the subsequent mechanical and ultrasonic pretreatments have reported enhanced hydrolysis, biodegradability, and biogas production when household organic waste was subject as a substrate for the anaerobic digestion [19, 59]. The subsequent ultrasonication and microwave pretreatment of POME sludge before fermentation increases the solubilization of organic waste which helps to improve the H_2 productivity. Even though fermentative H_2 production can be achieved without any pretreatment of sludge or wastewater, the application of ultrasonication and microwave treatment (thermal treatment) resulted in a considerable enhancement in H_2 productivity and COD reduction. Besides, the coupling of ultrasonication pretreatment followed with microwave could be a more effective approach to restrain H_2 production. Overall, results entail the application of pretreatment coupled with anaerobic digestion before the wastewater gets discharged to the water bodies to reduce its negative impact on the environment.

4 Conclusion

The pretreatment of POME before anaerobic digestion is advantageous for the maximum conversion of complex organic waste into H_2. Sonication, microwave, and coupling of sonication-microwave pretreatment employed for seed sludge have shown a considerable impact on cumulative H_2 production and COD reduction from POME wastewater. The improvement observed for cumulative H_2 production and COD removal was 12.14% and 21.42%, respectively, compared to the control one. The highest cumulative H_2 production and COD removal were observed when the ultrasonication and microwave pretreatment were applied together which suggested that the combined pretreatment approach gave better treatment performance of POME compared to ultrasonic and microwave pretreatment applied alone to sludge. Overall, sonication coupled with microwave pretreatment of sludge inoculum was more effective than the control one to neutralize the organic content of POME wastewater.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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