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Low frequency ultrasound treatment of palm oil mill effluent for solubilization of organic matter

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ABSTRACT

Palm oil is one of the most widely consumed edible oils in the world. Its production requires a huge amount of water and subsequently generates a large volume of wastewater called palm oil mill effluent (POME). POME contains large amount of slowly degradable compounds, which have an undesirable impact on the efficiency of biological treatment processes. Ultrasound treatment assists organic compounds disintegration and improves their solubility. Hence, this study investigates the effect of low frequency ultrasound treatment on solubilization of POME organic matter. Possible mechanisms of ultrasonic treatment are also discussed. Ultrasonication was applied at low frequency (20 kHz) with various ultrasonication amplitudes and durations. The results indicated that the energy used for ultrasonication strongly influences the physical and chemical characteristics of POME. Based on the optimum specific energy of 25,136 J/g total solids, maximum particle size reduction was 77%, soluble chemical oxygen demand (SCOD) increased by 20% and degree of solubilization achieved was 15.5%.

Keywords: Disintegration; Organic matter; Palm oil mill effluent; Solubilization; Ultrasound

1. Introduction

Palm oil industry is one of the major Malaysian industries that has grown rapidly over the years. As shown in Table 1, fresh POME is an acidic brownish colloidal suspension and is regarded as a high strength wastewater.

POME is commonly treated by biological treatment using anaerobic and aerobic systems. Anaerobic digestion is the degradation of organic matter, in the absence of molecular oxygen, via enzymatic and bacterial conversion to produce biogas as a renewable energy source. However, biological hydrolysis of the particulate organics in POME, which is the first step in anaerobic degradation, has been considered to be the rate limiting step that slows down the overall treatment process.

Considering the organically rich nature of POME which consists of refractory and slowly degradable soluble compounds, solids and long chain fatty acids, ultrasonication can theoretically convert them into simpler compounds. Ultrasound is recognized for its mechanical and chemical effects capable of improving the hydrolysis of organic matter for anaerobic digestion [2].

Upon disintegration, organic compounds are transferred from solid to aqueous phase due to enhanced solubility. Several reports have demonstrated effective disintegration of sludge by ultrasound pretreatment to improve anaerobic digestion and increase methane yield [3,4]. Thus, ultrasound disintegration is a promising method to enhance rate of anaerobic digestion and improve production of biogas.

Ultrasonication can significantly modify the structure of materials present in a liquid system through physical and

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Table 1
Characteristics of raw POME [1]

Parameters	Values
pH	4.05
Biochemical oxygen demand, mg/L	22,700
Chemical oxygen demand (COD), mg/L	44,300
Soluble COD (SCOD), mg/L	17,140
Total suspended solids (TSS), mg/L	19,780
Oil and grease (O&G), mg/L	4,850

chemical reactions. Sound waves propagate through the liquid with given intensity (amplitude) and frequency. When an ultrasonic wave passes through a liquid medium, it generates cavitation bubbles which are subsequently compressed and expanded by the passing sound waves until a critical point is reached where they eventually implode; producing local extreme conditions of temperature and pressure [5,6]. This process by which the bubbles form, grow and undergo violent collapse is known as acoustic cavitation. The acoustic cavitation phenomenon is an adiabatic process that causes a massive build-up of energy inside the bubbles; resulting in extremely high temperatures and pressures. In a liquid–solid system, the local high temperature and pressure of the 'shock waves' attack solid components in the liquid medium mechanically. These forces in the liquid can seriously affect the structure of organic matrices; creating strong shear forces that can change the surface morphology, composition and chemical activity. The mechanical shear forces by jet streams during cavitation bubble implosion disintegrate the solid components, and increase contact between the reaction medium and the substrates to improve downstream processing rate. Besides physical effects, ultrasonic disintegration mechanisms are expected to include oxidizing effects by free radicals especially hydroxyl radicals, thermal decomposition of volatile hydrophobic substances, temperature rise, pyrolysis and combustion [7]. During cavitation, pyrolysis occurs due to the very high local temperature. Suslick et al. [8] reported that these temperatures are in the range of ~1,900 K at the interfacial region and ~5,000 K at the cavity region. Pyrolysis occurs inside and near the interface of the cavity and the surrounding liquid medium at the time of bubbles collapse where hydroxyl radicals form inside the cavity during ultrasound.

Frequency and intensity of ultrasonic waves can increase bubbles oscillation and lead to generation of larger bubbles with higher accumulated energy before implosion. In general, low frequency ultrasound (20–100 kHz) forms larger bubbles whereas medium frequency/sonochemical-effect ultrasound (300–1,000 kHz) generates smaller bubbles. If energy provided to the medium is inadequate, the bubbles will only oscillate and will not implode or damage the structure of particles. Although the bubbles will not implode, their oscillation and the consequent mixing may still be able to enhance contact between the medium and the substrate. Hence, low frequencies are usually applied in ultrasound pretreatment to disintegrate particles [9,10]. Whereas, medium frequency ultrasound is usually applied in environmental remediation processes to degrade pollutants via thermal decomposition reactions or oxidation destruction of target contaminants via free radical reactions [11].

Ultrasound pretreatment disintegrates organic solids to make them more readily available to microbes during anaerobic digestion [12]. It transforms large organic structures into shorter chain compounds that would be more readily hydrolyzed. As well as enhancement of anaerobic digestion [13], ultrasound has also been used as a polishing step after biological treatment [14].

Cavitation helps to accelerate the slow step of hydrolysis during anaerobic digestion and fermentation processes [15]. The use of ultrasound has also spread to treatment of wastewaters consisting of recalcitrant organic pollutants [16]. To date, there is limited information on the effects of ultrasonication on organics solubilization in POME. Improvement of organics solubilization of POME could accelerate subsequent biological treatment, and thus could reduce the overall treatment footprint. Therefore, the present study aims to address this gap of knowledge by investigating the effects of different ultrasonication dosages and durations on the solubilization of organics in POME and to determine the optimum specific energy for effective degradation of the organics. The physical and chemical properties examined in this study included particle size profile, temperature profile, microscopic observation and chemical oxygen demand (COD) solubilization. Possible mechanisms by which ultrasonic treatment alters these POME characteristics are also discussed.

2. Materials and methods

2.1. Sample collection

POME was obtained from a local palm oil mill in Perak, Malaysia. Fresh POME collected from the mill was stored at 4°C in a closed plastic container before the experiments. The characteristics of POME were determined in triplicate according to the procedures given in APHA Standard Methods [17].

2.2. Ultrasonic treatment

The ultrasonic unit was a Cole-Parmer 500-W Ultrasonic processor. The instrument was equipped with a 13 mm titanium transducer with an operating frequency of 20 kHz and supplied power of 500 W. Frequency is the number of occurrences of a repeating event per second. Hence, 20 kHz frequency means that the ultrasonic probe completes 20,000 vibration cycles per second. Batch experiments were carried out at room temperature (25°C ± 3°C) with 100 mL samples in a 150 mL glass beaker. The ultrasonic and temperature probes were submerged into the beaker to 2 cm above the bottom. Different ultrasonic amplitudes ranging from 20% to 50% were applied for various ultrasonication durations from 10 to 1,800 s. Ultrasonication amplitude is the strength of ultrasonication power applied to the sample which can be adjusted from the ultrasonic processor control panel. Ultrasonication power (W) is the measure of energy per unit of time that is conveyed from ultrasonic processor to the sample. The ultrasonication power cannot be set directly due to several operating parameters including amplitude, sample viscosity and horn immersion depth. Ultrasonication intensity is a function of the power input and the ultrasonic probe surface area; common unit is W/cm^2. For a given power input, the larger

the ultrasonic probe surface area, the lower the ultrasonication intensity. The density of ultrasonication is the amount of delivered power per unit of sample volume; common unit is W/mL. For a constant volume mode reaction (batch mode), ultrasonication intensity and ultrasonication power are directly related and can both be used to represent cavitation strength and the effectiveness of sonication. Specific energy (E_s) was calculated based on Eq. (1) to determine the optimum sonication condition. In this study, the effect of E_s on qualitative and quantitative parameters is also discussed.

$$E_s = \frac{(P \times t)}{(V \times \mathrm{TS}_0)} \quad (1)$$

where E_s is the specific energy (J/g total solids[TS]), P is the ultrasonication power (W), t is the ultrasonication time (s), V is the sample volume (L) and TS_0 is the initial TS concentration (g/L).

2.3. Analytical methods

2.3.1. Particle size and particle size distribution

Particle sizes of the POME samples were analyzed in triplicates using a Malvern Mastersizer Hydro 2000 MU laser diffractometer. 3 mL samples were injected into the machine. The volume weighted mean particle size was determined and the results of particle size distribution (PSD) were expressed according to their particle size range: d10, d50 and d90. For instance, d90 is defined as the cut-off diameter at which 90% of particles have diameters equal to or smaller than the value of d90.

2.3.2. Microscopic examination

The microscopic image evaluation of samples before and after disintegration could illustrate qualitative information of sample disintegration such as structural change in flocs and disappearance of filaments. 0.5 mL samples were spread evenly onto a microscope slide and structure of the sample solids were observed using a Carl Zeiss Trinocular Microscope fitted with a CCD camera AxioCam ERC 5s with 100× resolution. The images were acquired by ZEN Lite software.

2.3.3. Chemical oxygen demand and soluble chemical oxygen demand

COD was measured for the well-mixed POME and for the supernatant according to APHA 5220D Closed Reflux Method. COD solubilization represents the transfer of COD from the particulate fraction to the soluble fraction. Centrifuged (2,500 rpm for 10 min) POME supernatant was filtered through 0.45 μm cellulose acetate membrane filter prior to soluble chemical oxygen demand (SCOD) determination. Investigation of SCOD is necessary for understanding sonication-induced mass transfer effects on the POME samples.

COD solubilization efficiency was represented by the degree of solubilization, which was calculated using Eq. (2). Where SCOD_o is the soluble COD of the sample in mg/L before ultrasonication, SCOD is the soluble COD in mg/L after ultrasonication and COD is the total COD in mg/L before ultrasonication.

$$\text{COD solubilization} = \frac{\mathrm{SCOD} - \mathrm{SCOD}_o}{\mathrm{COD} - \mathrm{SCOD}} \times 100\% \quad (2)$$

3. Results and discussions

3.1. Characterization of POME

POME is a high-strength wastewater with high COD, total suspended solids (TSS) and oil and grease. The characteristics of POME used in the study are shown in Table 2. In general, POME characteristics are strongly affected by the crop season and the production quantity of crude palm oil [18]. The high amounts of TS and TSS in POME originate from insoluble organic substances being washed out during the production process. The organic substances including residue of fruits' carp, lignocellulosic compounds from fruit bunch and residue oil from crude palm oil processes. The BOD/COD ratio of POME is more than 0.4; indicates a good possibility that the organic matter is biodegradable and can be treated with a biological treatment system [19].

3.2. Effect of ultrasound on particle size and PSD

Previous studies have shown ultrasound treatment to be an effective technique that reduces particle size of sludge through the generation of intense shear forces [9]. The efficacy of size reduction depends on different factors such as ultrasonication density, ultrasonication intensity, ultrasonication power, sample characteristics and sample volume [13].

In this study, the disrupting effect of the ultrasound treatment was evident when sonication was performed for the first 10 s; the particle size of POME decreased drastically from 292 μm to 65–168 μm yielding 42%–78% particle size reduction. As shown in Fig. 1 and Table 3, higher ultrasonication power (40% and 50% ultrasonication amplitudes) can reduce the size of the particles in a shorter time compared with lower ultrasonication power. The ultrasonication amplitude is an important factor to determine the ultrasonication

Table 2
Characteristics of raw POME in this study (mean ± SD)

Parameters	POME mean value
Temperature, °C	80 ± 1
pH	5.03 ± 0.5
Biochemical oxygen demand	18,800 ± 2,230
Chemical oxygen demand (COD)	46,900 ± 4,550
Soluble COD (SCOD)	7,150 ± 330
Total solids (TS)	43,180 ± 3,300
Total suspended solids (TSS)	21,730 ± 2,300
Oil and grease (O&G)	7,683 ± 650

All the parameters listed are in mg/L, except for pH which has no unit.

density and intensity. This result is in agreement with other studies that show higher ultrasonication intensity is better than prolonged ultrasonication time in terms of disintegration of sludge flocs [20]. Regardless of ultrasonication power, the mean POME particle size was reduced to the smallest size of 65–70 μm at ultrasonication duration of 1,800 s, resulting in mean particle size reduction of roughly 76%–78%. The maximum size reduction achieved was 77.7% for the samples sonicated at 40% amplitude for 600 s and 50% amplitude for 1,800 s.

Considering energy consumption for the sonication process, the optimum sonication parameters based on the highest percentage of particle size reduction with the minimum E_s applied is presented in Table 3. Sonication at 40% amplitude for 600 s was the most energy efficient. The sample received sufficient ultrasonication intensity to break down most of the particles to the smallest size. Tiehm et al. [21] suggested that the hydro-mechanical shear force produced by ultrasonication cavitation, which occurs under sufficient ultrasonication intensity was the predominant factor in sludge disintegration. In this case, E_s of 25,136 J/g TS was required for size reduction of 77.7%. Sonication at 50% amplitude also produced the same level of size reduction, albeit at 25% higher specific energy, that is, 31,420 J/g TS.

The size distribution of both sonicated and unsonicated POME samples was determined in terms of cut-off diameters d10, d50 and d90 (Fig. 2). All POME particles observed were smaller than 700 μm in diameter. The d90 of untreated POME was 633.96 μm, and its median diameter was 240.83 μm. With the increase in specific energy of ultrasonication from 209 to 25,136 J/g TS, the d90 dropped from 504.56 to 202.81 μm and the median diameter decreased from 70.48 to 10.94 μm.

Similar to d90, the other PSD parameters, d10 and d50, also decreased with increasing E_s. The extent of the reduction, however, was slightly different for each case. Table 4 presents the different cut-off diameters of sonicated and unsonicated POME. It was found that the unsonicated POME particle sizes ranged from 37 to 650 μm. Ultrasonication at 25,136 J/kg TS reduced d10, d50, d90 by 99%, 95% and 68%, respectively. Particle size reduction remained almost constant for higher E_s (>25,136 J/g TS). It is expected that the reduction in size of suspended solids will increase the disintegration rate and the biodegradability of the organic solids will be improved.

3.3. Effect of ultrasound on temperature

Ultrasonication treatment leads to change in physicochemical properties of POME including

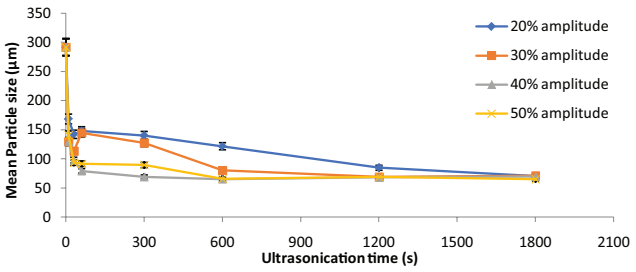


Fig. 1. Effect of ultrasonication amplitude on mean particle size.

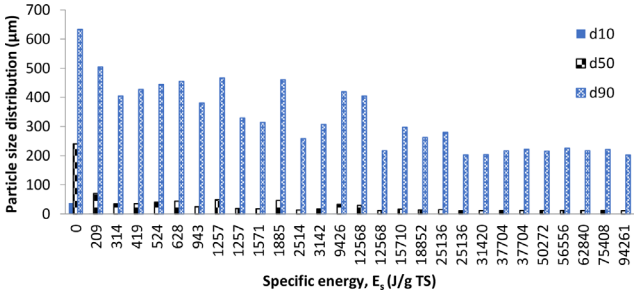


Fig. 2. Influence of specific energy on particle size distribution.

Table 3
Highest % mean particle size reduction from ultrasonication

Ultrasonication amplitude (%)	Ultrasonication duration (s)	Specific energy (J/g TS)	Mean particle size (μm)	Mean particle size reduction (%)
20	1,800	37,704	70.022	76.0
30	1,200	37,704	68.761	76.4
40	600	25,136	65.194	77.7
50	600	31,420	65.422	77.6
50	1,800	94,260	65.047	77.7

Table 4
Cut-off diameter of POME at different specific energy

Cut-off diameter of POME	Particle size (μm) for unsonicated and sonicated POME		
	Unsonicated, E_s = 0 J/g TS	Sonicated, E_s = 209 J/g TS	Sonicated, E_s = 25,136 J/g TS
d10	37.004	1.253	0.438
d50	240.830	70.418	10.935
d90	633.960	504.555	202.81

temperature. During sonication, the heat produced by ultrasonication is accompanied by acoustic cavitation and agitation; cavitational collapse produces intense local heating at the liquid–gas interface. Thus, the effects of ultrasonic energy and duration of ultrasonication on POME temperature were observed in this study. Previous studies have shown that prolonged sonication can raise sludge temperature by 18°C–37°C [22]. From Fig. 3, temperature was observed to rise by 17°C–58°C for POME samples sonicated for 10 min or more. The increase in temperature could occur as a result of specific absorption of acoustic energy by organic substances [15].

Fig. 3 shows that the temperature steadily increased with sonication time and amplitude until it reached a maximum value indicating equilibrium. Higher temperature was associated with higher sonication amplitude for the same treatment time. For example, at 30% ultrasonication amplitude, the change in temperature after 10 min of treatment was 43°C, while greater increase in temperatures was observed as 49°C at 40% ultrasonication amplitude and 55°C at 50% ultrasonication amplitude. The temperature increase might be attributed to three effects caused by ultrasonication: the loss of ultrasound energy into the POME by attenuation and absorption, the wild thermo-locomotion of bubbles agitation, and the thermal implosions of cavitation bubbles. Since more energy was dissipated to the sample with increased sonication, the rising curve was nearly proportional to the sonication time.

Temperature plays a significant role in bioconversion of organic matter during anaerobic digestion. It has a significant contribution in improving the sample quality and offers a reliable environment for the anaerobic bacteria. Ultrasonication could benefit anaerobic digestion in terms of energy saving in maintaining digester temperature where pretreatment is conducted on site.

3.4. Effect of ultrasound on microscopic image of POME

POME is a mixture of water, oil and natural sediments (solid particles and fibres). The sediments are irregular in form and have a wide range of sizes because of the way palm oil fruits are processed in factories. POME sediments are residuals from fresh fruits and fresh bunches (including fibres) that passed through the processing during palm oil extraction.

POME fibres have different shapes such as simple fibre, bunched fibre and flake fibre. The fibres in natural sediments of POME may either be partially embedded in the particles or may stand alone as shown in Fig. 4(a); approximate length

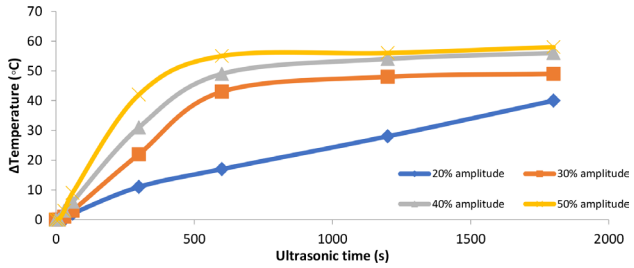
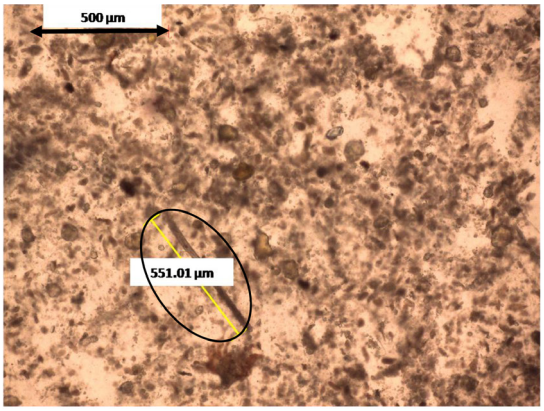
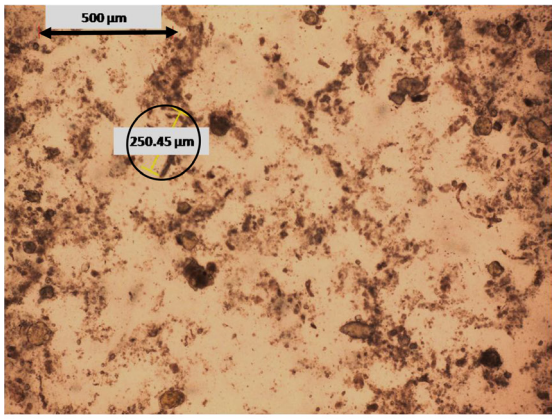


Fig. 3. Temperature profile with different ultrasonication time and amplitude.

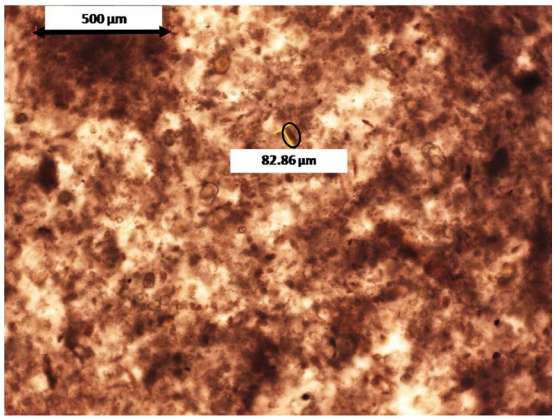
of 551 μm. According to Alrawi et al. [23], the larger strands had approximate diameter of 185 μm and the smaller ones were about 63 μm in diameter with lengths ranging from 700 to 7,900 μm. The length of fibre observed after sonication with E_s 2,514 J/g TS was 250.45 μm while with prolonged sonication up to 300 s, it was 82.86 μm which is about 85.1% in length reduction after ultrasonication treatment (Figs. 4(b) and (c)).



(a) Raw POME (unsonicated); E_s = 0 kJ/kg TS



(b) Sonicated at 40% 60s; E_s =2514 J/g TS



(c) Sonicated at 40% 300 s; E_s = 12568 J/g TS

Fig. 4. Microphotograph of POME particles before (a) and after ultrasound treatment ((b) and (c)). (a) Raw POME (unsonicated); E_s = 0 kJ/kg TS, (b) sonicated at 40% 60 s; E_s = 2,514 J/g TS, (c) sonicated at 40% 300 s; E_s = 12,568 J/g TS.

The most intuitionist evaluation of the disintegration effects of ultrasound can be obtained by microscopic analysis, which provides a visual appraisal of the pretreatment. Fig. 4 depicts the morphology of POME particles before and after ultrasound treatment at various sonication times at 10×.

These images reveal that the degree of POME particles disintegration was a function of the sonication time. With the increase in sonication time, the particle breakage for sonicated POME became more and more apparent, indicating that longer sonication time could cause greater particle disruption. For example, after 60 s (Fig. 4(b)), although the structure became somewhat looser and floc size became smaller, the architecture of flocs was basically the same as the unsonicated POME. The effects were more obvious after 300 s of sonication (Fig. 4(c)), when particle breakage became more apparent; the structure and the particle clusters were dispersed and loosened. It is predicted that shockwaves and high-speed microjets of ultrasonic cavitation impact loosened and destroyed the chemical linkages in the lignocellulosic structure [24].

3.5. Effect of ultrasound on SCOD and SCOD solubilization

SCOD has been used by many researchers as an indication of the amount of soluble organic matter in sludge. Therefore, SCOD was also selected as an indicator in this study to evaluate the particle disintegration efficiency. The increase in SCOD with sonication time, shown in Fig. 5, indicates that sonication treatment converted a portion of complex total chemical oxygen demand (TCOD) into soluble form (SCOD). As presented in Figs. 5 and 6, the SCOD increased by 20% after sonication for 10 min at 40% ultrasonic amplitude which is equivalent to E_s of 25,136 J/g TS and the SCOD solubilization achieved was 15.5%. The trend was consistent with the results of particle size reduction, indicating increase in disintegration efficiency with sonication time and amplitude. Simonetti et al. [25] investigated the effect of low frequency ultrasound treatment for waste activated sludge samples. Their results showed that SCOD increased by 80%–94% with sonication time of 5–20 min.

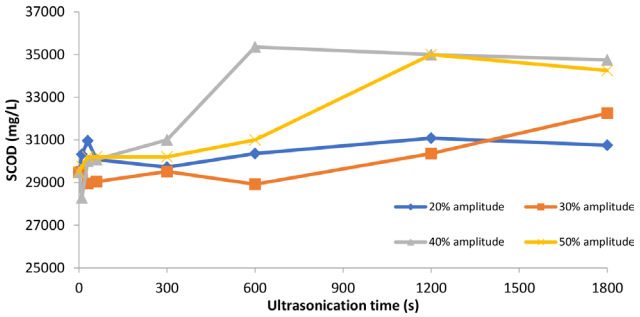


Fig. 5. Effect of ultrasonication parameters on the solubilization of POME in terms of SCOD.

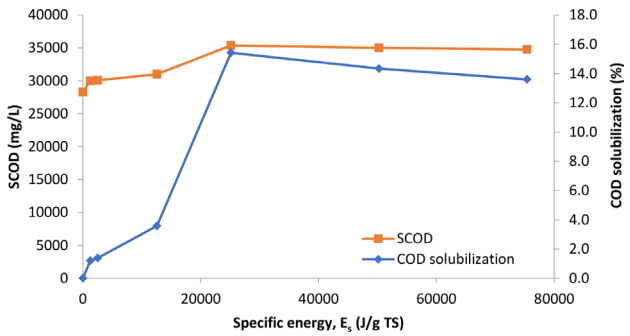


Fig. 6. Disintegration of POME using various sonication energies.

Two possible hypotheses can be put forward to explain the increase in soluble COD due to sonication treatment. The first is the de-agglomeration and breakage of larger particle structure into smaller size particles. This hypothesis was supported by microscopic appraisal and particle size analysis. In a study on the effect of sonication on sludge disruption, Show et al. [26] reported that the mean particle size decreased from ~50 to ~10 μm for municipal sludge sonicated at 0.52 W/mL. Chu et al. [27] observed the changes in ultrasonically treated waste activated sludge and reported that at 0.33 W/mL, the floc size decreased from 98.9 to 4 μm after 120 min of sonication. Another reason is related to the temperature increase after sonication, whereby the high temperature could enhance the solubility of organics. Increasing the solubilization of the complex organics means more volatile solids become readily amenable to further decomposition [28]. The SCOD results show that ultrasonication could transform TCOD of the particles into the soluble form, which could be beneficial to anaerobic digestion. Anaerobic bacteria fed with sonicated POME could be provided with more nutrients, substrate and available reaction sites in the digestion process. Therefore, a better degradability of sonicated POME might be expected in view of soluble organics availability.

4. Conclusions

Ultrasound pretreatment, through its cavitation and sonochemical reaction mechanisms, was able to solubilize complex organic molecules present in raw POME. The results indicated that low frequency ultrasound treatment could improve the characteristics of POME by reducing the particle size and increasing the soluble COD. Higher ultrasonication power ruptured more particles and more severely disintegrated the sturcture. Higher ultrasonication power can reduce the size of the particles in a shorter time compared with lower power. The optimum reaction conditions achieved were: ultrasonication duration of 600 s and ultrasonication amplitude of 40% which translate to E_s of 25,136 J/g TS.

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