

1 Applicability of various 2 pretreatment techniques to 3 enhance the anaerobic digestion 4 of Palm Oil Mill Effluent (POME): 5 A Review 6

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14

15 Abstract

16

17 Palm Oil Mill Effluent (POME) is a potential source of renewable energy. With the
18 intensified energy demands in various industries, if the biogas production from POME
19 treatment can be sustained, the palm oil industry can be made more environmentally and
20 economically sustainable. The current POME treatment process is ineffective in removing
21 residual oil and creates a large amount of greenhouse gas emissions. Furthermore, the
22 limitation of the treatment process is the instability of the anaerobic digestion stage, which is
23 affected by various operating parameters. Subsequently, the chemical and physical
24 characteristics of POME vary depending on different factors, such as the harvest season and
25 the efficiency of the palm oil extraction process. Therefore, having an effective pretreatment
26 system can radically reduce the load on the anaerobic digesters, sustain biogas production,
27 and enhance the treated effluent quality so that it can conform to the stringent environmental
28 standards. The focus of this paper is to review the pretreatment methods (treatments
29 undertaken prior to anaerobic digestion, which contributes to enhancing the hydrolysis step).

30A comparison of various types of sludge will be carried out to allow a thorough
31understanding of the impacts of these pretreatment methods and the suitability of their
32implementation for POME treatment. It was established that thermal pretreatment has bene
33proven to enhance solubilization, biogas production and the quality of the treated effluent.
34The increase in biogas production can easily compensate for the energy added to the system.

35**Keywords:** Palm Oil Mill Effluent, Anaerobic digestion, Pre-treatment techniques,
36solubilization, Biogas production, sludge pretreatment.

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115 1. Introduction

116

117 Oil palm tree plantations cover approximately 3 million hectares of cultivated area in
118 Malaysia - a 70-fold increase since the 1960s [1]. It was predicted that the total world
119 demand for palm oil in 2015 was 64.5 million tons and will increase to up to 95.7 million
120 tons by 2025, showing that palm oil industries will unceasingly grow to satisfy the world
121 demand [1]. The pronounced environmental encumbrance that comes with the production of
122 Crude Palm Oil (CPO) is Palm Oil Mill Effluent (POME), which is a waste product. Each
123 tonne of CPO production produces approximately 2.5–3.0 m³ of POME [2]. POME is a thick
124 brownish colloidal suspension with a pH of approximately 4.0-5.0; it is produced from the
125 mixture of sterilizer condensate, separator sludge, and hydrocyclone waste in a 9:15:1 ratio,
126 respectively [3]. It is discharged at a temperature of 80-90°C. POME is wastewater
127 comprised of 95%–96% water, 0.6%–0.7% oil and 4%–5% total solids, including 2%–4%
128 suspended solids as well as cellulosic material, fat, oil and grease. These characteristics of
129 POME are dependent on the efficiency of the operation and process control of the mill. Along
130 with a foul smell, POME is made up of organic materials, such as lignin, carotene, phenolic,
131 and pectin, which are of 4700 ppm, 8 ppm, 5800 ppm, and 3400 ppm, respectively [4].
132 POME can cause pollution due to the high amount of easily degradable organic matter, which
133 refers to the contents of high COD and BOD that have the values of 50,000 ppm and 25,000
134 ppm, respectively [4,5]. POME contains 6000 mg/L of oil and grease, 59,530 mg/L of
135 suspended solids and 750 mg/L of nitrogen. Consequently, POME can cause water pollution.
136 POME discharge contains microorganisms that will compete with the aquatic life mainly on
137 the uptake of oxygen. This occurrence will ultimately cause the aquatic life to have less
138 oxygen, slowly hindering the growth of the marine life and, eventually, causing the extinction
139 of the latter [4–6].

140The current POME treatment process is inefficient in terms of the residual oil separation in
141the waste stream and produced a large amount of greenhouse gas emissions owing to the
142facultative ponds. After the POME has cooled down to the adequate temperature for
143mesophilic conditions in the oil removal pit, also known as the cooling ponds, it is sent
144directly to the anaerobic digestion step. However, often, the treated effluent quality is poor
145and does not conform to the strict environmental standards.

146 Anaerobic digestion (AD) is commonly employed to treat POME since it is
147environmentally friendly and is the most cost-effective method [7]. However, AD is also the
148restrictive step in the POME treatment process, as hydrolysis is the rate-limiting step of AD;
149the latter results in the organic matter only partially putrefying. AD is a tedious and unstable
150process, as it is dependent on various types of operating parameters, which are the pH of the
151system, the temperature, AD inhibitors, and the retention time [8,9]. Therefore, for AD to be
152effective, it must be aptly controlled and monitored, as it can produce a considerable amount
153of biogas and fundamentally enhance the treatment quality of the effluent. Consequently,
154POME has a stringent effluent quality standard to which it must conform. It was observed
155from several studies [9–11] that POME is not only able to be effectively treated, but it can
156also produce valuable by-products, such as methane, and the treated sludge can be used as
157fertilizer. Other gaseous emissions from AD can thus be treated in a simplified manner [12].
158Therefore, it is essential to resolve the issues pertaining to the AD of POME to ensure that the
159treatment process operates optimally and that the biogas production is enhanced and
160sustained.

161 The current POME treatment research focuses on the polishing techniques, which are
162known as tertiary treatments. The polishing technologies cater to the treatment of POME after
163AD to further improve the treated effluent quality. However, the polishing treatment does not
164address the foremost limitations of the current treatment process, which are the extensive

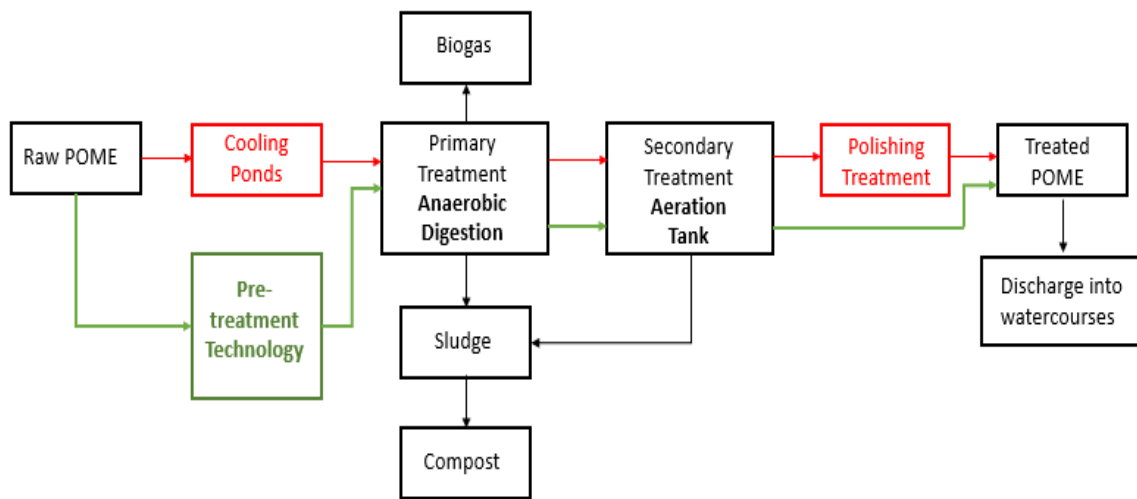
165greenhouse gas emission from the cooling ponds and the facultative ponding system, poor
166treated effluent quality and the substantial land use of the ponding system. Furthermore,
167removal of residual oil in the waste stream is ineffective and further hinders AD due to scum
168formation and other inhibitory factors [4,13].

169 On the other hand, pretreatment techniques used on the effluent before AD are not only
170able to remedy the latter but can also enhance the anaerobic digesters' performance and boost
171the biogas production, which can be captured and used as a renewable energy source.
172Therefore, identifying suitable pretreatment techniques for the treatment of POME can
173potentially assist in ensuring a stable AD operation along with sustainable biogas production
174[14]. Nonetheless, studies on the pretreatment of POME prior to AD are limited; none of
175these techniques have been implemented on a full-scale plant, and most are still at research
176stages [5]. Due to the scarcity of research on this topic, the main aim of this paper is to
177investigate the practicability of the pretreatment technologies that can be implemented in the
178traditional POME treatment process. The article will attempt to review the principles of AD,
179the sludge characteristics and the potential pretreatment techniques that will assist in ensuring
180a stable anaerobic digester operation with an increase in the production of biogas despite the
181different conditions associated with the POME treatment. The current treatment methods
182tested on POME at laboratory scale will be analysed, and the different promising
183pretreatment techniques will be thoroughly evaluated.

184 2. General Scheme of Palm Oil Mill Effluent treatment 185

186 Figure 1 shows the process flow of the conversion of raw POME into treated POME. The
187physical and chemical characteristics of raw POME are continually changing since many
188factors affect the content and the behaviour of POME, for example, the physiochemical
189characteristics alter from the high crop season to the low crop season, and the efficiency of

190the palm oil extraction process determines the remaining amount of oil and grease in the
 191POME. In the current process scheme, raw POME is directed to a cooling pond where the
 192residual oil in the POME is scraped off the surface of the pond to recover any CPO lost into
 193the waste stream. Since there is no treatment of POME before AD in the current scheme, the
 194primary treatment of POME is AD. Without proper controls, the AD cannot perform
 195optimally, the factors affecting AD will be further discussed in Section 3. The effluent from
 196the AD is then transferred into an aeration tank where the air is continuously pumped.
 197Aerobic treatment post AD is known to improve the treatment quality of the POME by
 198further removing the total Chemical Oxygen Demand (tCOD), BOD, Volatile Suspended
 199Solids (VSS) and Total Suspended Solids (TSS) [15]. The Aerobic treatment is the most
 200satisfactory secondary treatment since an ample and evenly distributed oxygen supply in an
 201aeration system results in rapid, economically viable, and effective effluent treatment.



202

Legend

- Conventional Process
- Proposed Process
- Unchanged streams
- Processes Eliminated
- Process Introduced

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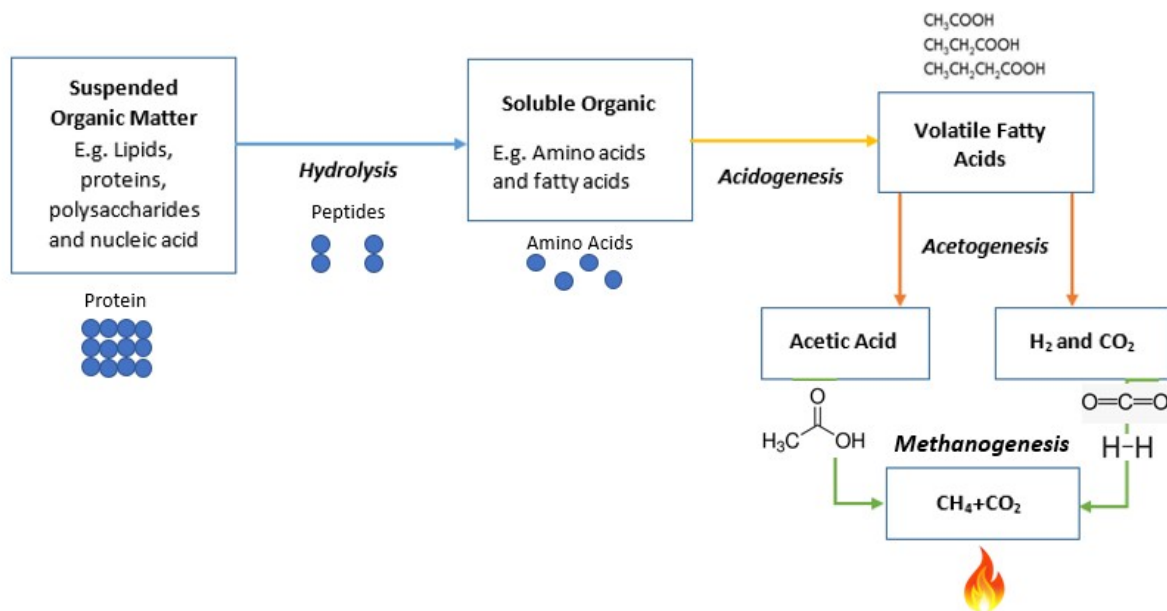
204 *Figure 1: The Conventional and Proposed process flow diagram for POME*
205 *treatment*
206

207 Nevertheless, the issue with the current process scheme is that the raw POME
208 discharged at 80-90°C is sent to the cooling pond where a considerable amount of heat is lost
209 (temperature drops to 60°C), POME is allowed to further cool down to reach an adequate
210 mesophilic temperature and the process of oil extraction from the cooling pond is highly
211 ineffective. Subsequently, if the cooling pond was eliminated, and raw POME was treated by
212 introducing a pretreatment technology, as illustrated by the green section in Figure 1, the
213 treated POME would then be sent to the anaerobic digester at a higher temperature. These
214 alterations in the AD process would be significantly enhanced, and the amount of biogas
215 produced would upsurge, all while drastically reducing the greenhouse gas emissions to the
216 atmosphere, as the biogas produced will be captured and used as a source of energy. This
217 alteration in the process will not only improve the treatment quality of the POME but will
218 prevent heat loss that can be essential to improve the hydrolysis step in AD [3,16,17]. Other
219 factors that are of importance are the biogas quality and the final gaseous emission, which are
220 primarily dependent on the anaerobic biogas system, implying that the operation and process
221 control of AD must be top notch [18]. The polishing treatment can subsequently be
222 eliminated since the enhanced AD process can achieve a better-treated effluent quality. One
223 of the objectives of this paper is to find the best performing pretreatment technique that can
224 be coupled with the anaerobic digesters to ameliorate the sustainability of the POME
225 treatment process.

226 3. Anaerobic Digestion 227

228 Anaerobic digestion (AD) is commonly used in water treatment industries since it is
229 environmentally friendly and is the most cost-effective practice [19]. Some advantages of AD

230are as follows: first, AD operates in the absence of oxygen, thus eliminating the high energy
 231cost associated with the extensive supply of oxygen; second, it reduces the volatile solids
 232content; third, it enables energy recovery due to the production of methane; and finally, it
 233decimates most of the pathogens present in sludge as well as limits the stench concomitant
 234with the remaining putrid substance. However, AD is also the restrictive stage of the
 235treatment process since the organic matter only partially putrefies due to its rate-limiting step,
 236which is hydrolysis. The AD performance is unstable; thus, it has to be appropriately
 237controlled [20]. Many essential parameters are required to be monitor and managed during
 238the AD of POME; these are the pH of the system, temperature, solid and hydraulic retention
 239time, organic loading rate, solid loading and the substrate oil content [19,20]. Figure 2 shows
 240the different processes that occur in AD.



241

242 *Figure 2: The stages of Anaerobic Digestion*
 243

244 In the AD of POME, a chain of reactions is involved, explicitly, hydrolysis,
 245acidogenesis, acetogenesis, and methanogenesis. Hydrolysis is a reaction in which the
 246particulate in organic compounds or complex molecules, such as carbohydrates, proteins, and
 247lipids, are broken down into simpler molecules, e.g., sugar and amino acid, via the activities

248of extracellular enzymes from hydrolytic microorganisms. In acidogenesis, the acidogenic
249bacteria further breaks down the hydrolysed molecules into organic acids and intermediates,
250which will form acetic acid, carbon dioxide, and hydrogen, as well as inorganic compounds,
251such as ammonia and hydrogen sulfide. Lastly, in methanogenesis, the acids are permuted to
252methane gas by methanogenesis bacteria.

253 Despite the many advantages of AD, there are also a few limitations to it [21–23]; if
254not well-controlled, the AD process can be inefficient and the biodegradability of the
255substrate may be adversely affected. The rate of AD is relatively slow, the substantial
256capacity of sludge and the costly reactors are often not compensated by the amount of biogas
257produced; however, the savings from the electrical energy for aeration and hygienisation are
258significant advantages [19].

259 The microbiology of AD is intricate; nevertheless, it is essential to grasp the
260understanding of how the microorganisms execute AD. AD is carried out by several bacterial
261groups that function under specific optimum environments. These bacteria groups are
262responsive to multiple process parameters, such as temperature, pH, retention time, the
263organic loading rate, and oil content of the substrate and may also be inhibited by these
264parameters. It is fundamental to systematically understand how these factors affect AD to be
265able to counteract the undesirable effects and regulate the needed conditions that favour a
266productive digestion process, which will, in turn, allow a continual production of biogas and
267better effluent quality.

268 3.1 Temperature

269 From many different studies [4,8,19,21,24,25], it was found that temperature plays a
270vital role in determining the rate at which the anaerobic degradation occurs, implying that the
271temperature of the system governs the rate of hydrolysis and methanogenesis. AD generally
272exhibits two optimal temperature ranges, as follows: a mesophilic condition, which operates
273at an optimal temperature of approximately 35 °C, and thermophilic, which operates at an

274optimal temperature of approximately 55 °C. Although mesophilic processes have extended
275retention times, their stability makes them beneficial. While a system operating at a higher
276temperature (thermophilic conditions) has numerous advantages in terms of higher
277biodegradability of organic compounds, enhanced rates of biological and chemical reactions
278as well as an increased elimination rate of pathogens, the application of higher temperatures
279may have a negative impact owing to the instability of the process, which adversely affects
280the microorganisms, thus causing fluctuations in the treatment performance. Moreover, there
281will be an upsurge in free ammonia, which can inhibit the function of the microorganism and
282an increase in pKa of the fatty acids will make the process even more inclined to inhibition
283[19,26].

284 3.2 pH

285 Meanwhile, the pH value of a digester's load is an essential parameter to consider
286since the pH governs the efficiency and stability of the digester. In an ideal AD process, most
287of the products of the metabolic stage are continually being converted into the next broken-
288down products without any substantial build-up of intermediary substances, such as fatty
289acids, which would cause the pH to drop. The ideal pH of AD depends on the adaptability of
290the bacteria. The methanogens are the most sensitive to pH and operate optimally at a short
291pH range of 6.5 to 7.2. Therefore, the AD process is often conducted at a pH range of 6.8 to
2927.6 to ensure an optimal environment for the methanogens to effectively generate methane.
293At a pH lower than 6.3 and higher than 7.8, the methane production rate may be adversely
294affected [8]. The alkalinity and pH of the system can be attuned using various chemicals,
295such as sodium bicarbonate, potassium bicarbonate, calcium carbonate (lime), calcium
296hydroxide (quicklime), and sodium nitrate [27].

297 Appels *et al.* (2008) noted that a volatile fatty acid (VFA) is an essential intermediate
298in the AD process. The VFA is broken down by proton-reducing acetogens alongside

299methanogenic bacteria feeding on hydrogen. However, it was noted that a concentration of
3006.7–9.0 mol/m³ VFA could be harmful to microorganisms, particularly to methanogens. The
301build-up in VFA occurs due to discrepancies in the AD process, which can be triggered by a
302fluctuation in the temperature, pH, organic loading, and harmful compounds. Consequently,
303the methanogens are unable to eliminate the volatile organic acids and hydrogen in due time
304as this takes place, and the acid concentration increases.

305Subsequently, the decrease in pH to an extremely acidic nature causes the hydrolysis to be
306inhibited. The latter occurs when there is an accumulation of insolubilized VFA. These can
307move spontaneously through the cell membrane, where they breakdown and produce a
308decrease in the pH, which causes interference in the homeostasis [28].

309 The occurrence of increasing VFA concentrations in a batch anaerobic reactor system
310has a different influence on the distinct metabolic stages of hydrolysis, acidogenesis, and
311biogas production, i.e., methanogenesis. The methanogens, as mentioned earlier, are highly
312sensitive to the pH, while the fermentative microorganisms are, to some extent, less sensitive
313and can operate in a broader range of pH, i.e., between 4.0 and 8.5. At a low pH, the main by-
314products formed are acetic and butyric acid, while at a higher pH of 8.0, mainly acetic and
315propionic acid are produced. Excess VFA (concentration higher than 7.85 mol/m³) tends to
316reduce the pH of the system, consequently hindering the AD [21,28,29]

317

318 3.3 Solid and hydraulic retention time

319 The solids retention time (SRT) is the typical amount of time during which the solids
320occupy the digester, while the hydraulic retention time (HRT) is the period of time that the
321liquid sludge is retained in the reactor. The ratio between SRT and HRT can be varied by
322separating the solids and the liquids downstream of the digester and recycling one of the
323separated streams. The SRT influences the subsequent phases of the AD process. A decrease
324in the SRT causes the degree of the reactions to decrease. The study of the impact of the

325retention time on the disintegration efficiency has mainly been undertaken at the laboratory
326scale. The correlation between gas production and retention time demonstrated that, first, the
327AD is unstable and inconsistent at an SRT shorter than 5 days, as the VFA concentration
328increases due to a loss of methanogenic bacteria. Second, at an SRT between 5–8 days, the
329VFA concentrations are still moderately elevated, as there is only partial disintegration of
330organic compounds, particularly lipids. Third, steady digestion is achieved after 8–10 days,
331during which time, lower VFA concentrations are observed, as the solubilization of lipids
332takes place during this period. Finally, at an SRT greater than 10 days, the sludge has
333substantially been solubilized. Based on the observations at these different stages, it can be
334established that the SRT is an important design and operating factor in the AD processes.
335[30,31]

336 The hydraulic retention time (HRT) operates differently in comparison to the SRT.
337The HRT has often been associated with the size of the digester. The shorter the HRT of a
338process, the smaller the size of the digester used, which is an advantageous aspect, as it
339reduces the capital cost of the system.

340 Additionally, a shorter HRT tends to induce an increase in the rate of biogas
341production but adversely affects the disintegration of organic matter, such as the total solids
342(TS) or the Biological Oxygen Demand (BOD). The latter affects the process stability
343negatively. The HRT can deviate from 5 to 34 days based on the different amount of solid
344waste digested [31,32].

345 3.4 Organic loading rate

346 The organic loading rate (OLR) is described as the quantity of organic matter, (i.e.,
347mainly the COD of the substrate or volatile solid), treated by a specific volume of the
348anaerobic digester within a particular timeframe. The OLR of a process is associated with the
349hydraulic retention time. A shorter hydraulic retention time will produce a higher OLR [4,33–

35035]. Increasing the OLR in a wastewater treatment system often causes the treatment
351efficiency to decrease. On the other hand, the biogas production rate increases typically with
352an increasing OLR up to a certain level, where an additional increase in the loading rate is
353detrimental to the methanogens [34]. When acetic acid is not converted to methane rapidly
354enough, the production of biogas worsens as the methanogenic activity is halted due to the
355accumulation of acid in the system, which, in turn, lowers the pH of the medium [36].

356 3.5 Solid Loading

357 The main difference between AD technologies for the treatment of municipal and
358industrial biodegradable wastes is the solids concentration of the process. Wet AD systems
359function at low total solids, which is usually less than 12-20% TS, while dry AD processes
360have high operating solid contents ranging from 20-40% TS [37]. The efficiencies of the wet
361and dry AD systems were measured in terms of digestate management, technical operation,
362economic performance, and energy balance. Wet AD plants have better energy balance and
363economic performance in comparison to dry AD plants.

364 Nevertheless, dry AD plants have many advantages, comprising of better adaptability
365in the type of feedstock used, shorter retention times, less usage of water, and more
366appropriate end product management [38]. The dry mesophilic AD (40°C) of the paunch
367reached high yields of 126.9 to 212.1 mL/g VS and a volatile solid reduction of 32.9 to
36855.5% under SRT of 40 days at an OLR of 2.0 to 2.7 g VS/L d, while the psychrophilic AD
369(22°C) of the paunch was observed to have a significantly longer treatment time of 100 days
370and a low OLR of 0.7 to 1.1 g VS/L d [39]. Riya *et al.* (2018) studied the semi-batch dry AD
371under steady loading and varying TS contents and observed that as the substrate TS content
372increases (27-42%), the reactor TS content also increases (19-31%). The biogas production
373was noted to be stable when the reactor TS content was 19-27%, but as the reactor's TS
374content exceeded 28%, the biogas production was inhibited. From the above studies, it can be

375concluded that the solid loading influences the SRT and the biogas production of an AD
376system and is a unique aspect to investigate in regard to the treatment of POME.

377 3.6 Substrate oil content

378Martín-González *et al.* (2010) study the evaluated fat, oil and grease waste from sewage
379treatment plants (STP-FOGW) as a co-substrate in the wet AD of the source collected an
380organic fraction of municipal solid waste (SC-OFMSW) under a mesophilic condition. Batch
381experiments of different co-digestion ratios displayed an enhancement in methane production
382related to the STP-FOGW addition. The biogas yield was observed to increase from 0.38 to
3830.55 L/g VS_{feed} when STP-FOGW was added to the reactor feed. Both the VS reduction and
384biogas methane content were found to be constant, and no inhibition due to long chain fatty
385acid (LCFA) build-up was observed. Martínez *et al.* (2011) studied the AD of high lipid
386content waste undertaken using sewage sludge as the primary substrate; no change in the
387specific methane production was noted with a fat, oil and grease (FOG) content of 0.2%
388(V/V). Nonetheless, an increase in FOG concentration of up to 1.8% was significantly
389detrimental to the performance of the process. When digesting fat from a milk-processing
390factory, the outcomes displayed an effective operation of the semi-continuous reactor
391operating with an HRT of 40 days, although a high inhibition was reported from the batch
392experiments executed.

393 Taking the factors mentioned above into consideration can allow continuous digestion in
394which the microorganisms have the necessary medium to continually breakdown complex
395matter into simpler molecules and to obtain the desired by-product, which is, in this case,
396methane-enriched biogas. Pre-treatment techniques can be implemented before AD to cater to
397the constricting features of the AD process. Therefore, it is essential to identify which
398parameters influence which stage of the process so that the most suitable pretreatment

399 techniques can be established. These techniques need to be able to not only enhance the AD
400 process, but they must also be technically, environmentally, and economically feasible.

401 4. Pretreatment techniques tested on various 402 substrates applicable to treating POME. 403

404 Since the studies of pretreatment techniques undertaken on POME are scarce
405 compared to the research conducted of the pretreatment techniques used on different types of
406 sludge, such as primary sludge, mixed sludge, waste activated sludge (WAS) and organic
407 fraction of municipal solid waste (OFMSW). This study discusses the pretreatment
408 techniques used on different types of sludge, which can assist in finding the appropriate
409 pretreatment techniques and the optimum conditions under which POME can efficiently be
410 treated to enhance solubilization and sustain biogas production.

411 4.1 Biological Pre-treatment 412

413 In general, the studies undertaken had a biological pretreatment condition of 70°C.
414 The effect of the biological pretreatment at 70°C for 7 days on both the activated and primary
415 sludge was investigated by Gavala et al. (2003); they observed that the activated sludge
416 produced 0.41 ml of CH₄/gVS_i with no significant increase of methane production, while the
417 methane production from primary sludge resulted in an 86% increase in methane production.
418 Similarly, Skiadas et al.'s (2005) study entailed the biological pretreatment condition (70°C,
419 2 days) on both activated and primary sludge; however, the latter's findings proved to be
420 contradictory to those of Gavala et al. (2003), who observed a 28% and 11% increase in
421 methane production in activated and primary sludge, respectively [44]. The contradictory
422 results may be due to the age of substrates (sludge) and the different types of biological
423 organisms used in the treatment studied. The effect of biological pretreatment on the
424 OFMSW using mature compost as a biological agent at a temperature of 55°C was conducted

425by Fdez.-Güelfo et al. (2011); they noted that the cumulative biogas and methane production
426increased to 190% and 141.6% of the original, untreated values, respectively. Finally, Yu et
427al. (2013) investigated the effect of 2 enzymes on WAS at 37°C for 28 h; they found that the
428sCOD increased by 78.2% and achieved a 20% improvement in methane production.

429 It can be established from the studies mentioned above that under different conditions
430and with varying types of substrates, the increment of methane production can range between
43111 to 141%, showing that biological pretreatment does enhance AD, specifically hydrolysis,
432since the methane production was increased in a shorter period [46]. Nevertheless, the
433economic feasibility of the technique has to be investigated more profoundly since the high-
434cost of commercial enzymatic preparation may make the pretreatment economically
435infeasible, which is the major drawback of this technique [47].

436 4.2 Chemical Pretreatment

437

438 4.2.1 Acid Pretreatment

439 The most common acids used for pretreatment are HCl and H_2SO_4 . Tyagi et al.
440(2011) reported that acid treatment to solubilize the WAS undertaken at the laboratory scale.
441They stated that the conditions to achieve sludge solubilization with an acid pretreatment are
442significantly rigorous in terms of the extreme pH values and longer reaction times; enhanced
443solubilization of WAS of approximately 50 to 60% was obtained by treating the sludge with a
444sulfuric acid of concentration ranging from 0.5–1.0 g acid/g TSS for a duration of 30 s and at
445ambient temperature. Nevertheless, this process appears impractical for the sludge
446solubilization at such high dosages of acid. Another issue with acid pretreatment is, since the
447pH levels are extreme, re-neutralization is necessary. Many studies compared the efficiency
448of both acidic and alkali treatments and concluded that the alkali treatment was more
449effective than the acid treatment.[22,48].

450 A study using free nitrous acid at a pH of 2 showed that the sCOD increased by 0.16
451mg COD/mg VS, and the highest methane yield (255 L CH_4 /kg VS added) was achieved
452within 15 days using a concentration of 2.13 mg N/L [49]. Another study undertaken at a pH
453of 2 stated that the VS removal increased by 1.7% compared to the untreated sample, the
454tCOD elimination was 2.7% higher than the untreated sample, and the biogas production was
45512% higher than the untreated sample. From the results of the above study, it can be
456distinguished that acid pretreatment slightly improves biodegradability. However, the
457significant disadvantages of this technique are the operational and maintenance cost
458associated with the latter due to extreme pH, which may lead to corrosion, and the additional
459step of re-neutralization.

460 4.2.2 Alkali Pretreatment

461 The most common alkalis used for chemical pretreatment are lime, NaOH, and KOH.
462Many studies presented that an alkali pretreatment is more efficient than an acid pretreatment.
463It was found that an alkali treatment is effective in sludge solubilization with the order of
464efficiency being NaOH > KOH > $Mg(OH)_2$ and $Ca(OH)_2$ [50]. López et al. (2008) also
465investigated the effect of quicklime as a pretreatment on the organic fraction of municipal
466solid waste (OFMSW) at room temperature for up to 6 h; they observed that the sCOD
467increased from 13,675 to 20,101 mg/l when the lime concentration was 70 mEq $Ca(OH)_2$ /L,
468which indicated 13% solubilization of OFMSW, the concentration of volatile dissolved solid
469was slightly augmented (5.8%), and the increase in the methane yield was up to 172.7%.
470They also observed that the optimum condition was 62.0 mEq $Ca(OH)_2$ /L for 6 h; however,
471at a longer period of time, the solubilization decreased. This phenomenon was related to the
472instability and formation of complex and undigestible compounds. An advantage of alkali
473pretreatment using quick lime is that the cost of quick lime is relatively low and can be
474adequate for full plant scale purposes [50–52]. Lin et al. (2009) investigated the effect of

475alkali pretreatment on pulp and paper sludge; they found that the sCOD concentration was 12
 476times higher than the control when the NaOH concentration was increased by 1.2%; the
 477concentration of VSS decreased from 6% to 19% after pretreatment; the settleability of the
 478pre-treated bioreactors were 56-162% higher than the control; the methane yield reached a
 479maximum of 88%.

480 To summarize the above findings, under appropriate alkali treatment conditions, WAS
 481becomes more biodegradable by altering its chemical structure. The latter induces swelling in
 482WAS, which provides a greater surface area for the microbial enzymatic activities. Alkali
 483pretreatment under favourable conditions can reduce the retention time of the digesters and
 484increase the methane production [53]. Since alkali pretreatment works with different types of
 485substrates, as mentioned above, it may be a viable option for the treatment of POME. Table 1
 486below shows the results of other studies on the alkali pretreatment performance.

487Table 1: Performance of Alkali pretreatment

Substrate	Treatment conditions	AD conditions	Solubilization	Gas production	References
Activated sludge	45 meq NaOH/L, 55°C, 240 min	Batch 20 days, 35°C	—	88% increase in CH_4 production	[54]
Activated sludge	20 meq NaOH/L, 35°C, 7-20 days	—	52% COD and VS removal	163% CH_4 production	[22]
Activated sludge	0.2 mol/L NaOH, 8 h	—	—	31% CH_4 production	[22]
Pulp and paper sludge	8 g NaOH/100 g TS sludge	Batch 42 days, 37°C	83% COD removal, 12 times more	83% CH_4 production, more methane	[53]

Activated sludge	1.65 g KOH/L, pH 10, 130°C, 60 min	Batch 24 days, 35°C	—	than control	yield 183.5% 30% biogas	[54]
Activated sludge	1.65 g KOH/L, pH 10, 130°C, 60 min	CSTR, HRT 20 days, 35°C	—		75% of CH_4 production	[54]
Activated sludge	NaOH	20-40°C, 0.5-24 h	45%	COD solubilized	112% of CH_4 production	[55]

488

489 4.2.3 Ozone pretreatment

490 This section analyses the effect of ozone pretreatment on different types of substrates.

491 It was reported that the methane production was 2.2 times higher compared to the untreated
 492 sludge following ozone treatment when treated with a dose of 0.1 g O_3 /g COD. A limited
 493 ozone treatment appears compatible with the subsequent digestion of biosolids and can be
 494 economically feasible. Bougrier *et al.* (2007) reported that ozonation increased the biogas
 495 production with the ozone dose of 0.15 g O_3 /g TS, which resulted in a considerable increase
 496 in the sCOD/tCOD ratio from 4% to 37%, while Zhang *et al.* (2009) observed that ozone
 497 pretreatment achieved the maximum upsurge in biogas production, which was 2.4 times
 498 higher than the untreated experiment. The implementation of ozonation into activated sludge
 499 does not prominently affect the effluent quality; however, the settling properties of the sludge
 500 are enhanced. Sludge ozonation may be cost-effective for WWTPs that have substantially
 501 high sludge disposal costs and operational problems, for example, sludge bulking and
 502 foaming [58]. The proposed ozone dose ranges from 0.03 to 0.05 g O_3 /g TSS, which is
 503 suitable to reach an equilibrium between the sludge reduction efficiency and cost [58].

504 To summarize, sludge ozonation is a well-proven technology and has been
 505 implemented at full scale. The latter can be of an economic advantage for larger plants with

506high disposal cost. Ozonation greatly improves the sludge dewaterability, settleability, and
507prevents bulking or foaming. However, a drawback associated with ozonation is that
508ozonation is a costly treatment, as it consumes a considerably large amount of energy and it
509has a high operating cost as well as a capital cost, as a great deal of energy is required to
510produce liquid oxygen[22].

511 4.2.4 Peroxidation pretreatment

512 There are several types of chemicals used for peroxidation pretreatment, namely,
513hydrogen peroxide, Fenton peroxidation, which is a mixture of hydrogen peroxide and Iron,
514dimethyldioxirane (DMDO), peroxymonosulphate (POMS), and Peracetic acid (PAA). Kim
515*et al.* (2009) reported that the combined alkaline hydrolysis and hydrogen peroxide oxidation
516causes the disintegration of sludge particles to be broken down into soluble organic
517compounds, which results in a tCOD and TS reduction along with the production of sCOD.
518They found that under the conditions of a pH of 11 and a 1.6 M H₂O₂ dose, the TS content
519decreased by 49% and the viscosity decreased by 69.1% compared to the initial untreated
520sludge, the sCOD/tCOD ratio increased from 0.02 for untreated raw sludge to 0.55, and the
521settleability also improved as the sludge volume index (SVI) declined from 67.6 ml/g to 62.9
522ml/g, which indicated a 7% enhancement in settleability.

523 Pre-treatment with peracetic acid (PAA) was found to solubilize the organic material
524efficiently. Through a pretreatment with 25 g PAA/kg DS, an increase in biogas production
525of 21% was attained [19]. Nevertheless, for dosages exceeding 40 g PAA/kg DS, the biogas
526production was less compared to the untreated experiment. This occurrence can be due to the
527inhibition of the anaerobic microorganisms by the increased VFA-concentrations. Neyens *et*
528*al.*(2003) reported that the addition of an amount of approximately 25 g H₂O₂/kg DS under
529the proposed conditions of pH, temperature, pressure, time and Fe²⁺ in combination with
530polyelectrolyte (Acrylamide/Adame-quate emulsion, 30% cationic, 100% branching) could

531be a favourable sludge treatment, achieving enhanced dewaterability, with a 30% reduction in
532the sludge volume compared to the untreated experiment. They noted a reduction in the
533capillary suction time (CST) value of approximately 40 s in contrast to the untreated sample
534[60].

535Another study showed that the result of the peroxidation treatment achieved a significant rise
536in the COD and BOD concentration in the sludge water, along with an upsurge in the
537BOD/COD ratio. Furthermore, a substantial increase in biogas production was observed. The
538increase in biogas production was measured to be 75% with Fenton, while with the
539peroxymonosulphate (POMS) treatment, the biogas production increased by nearly 2-fold,
540versus an even greater 2.5-fold for the dimethyldioxirane (DMDO) treatment. In another
541study, it was observed that the methane content in the biogas produced was consistently
542between 65 and 70% [61].

543 Some disadvantages linked to the use of Fenton oxidation are the safety hazards
544associated with the use of H₂O₂, the need to copiously lower the pH and the subsequent
545neutralization and the system is exceptionally corrosive due to low pH conditions and may, in
546turn, result in a relatively high operating and maintenance cost.

547

548 4.3 Thermal Pretreatment

549 4.3.1 Conventional Heating

550

551Thermal pretreatment has many benefits in regard to enhancing AD in general — the sCOD
552increases with an increased temperature and treatment time [62]. The COD solubilization was
553noted to be linearly correlated to a temperature treatment of 60 to 170°C (Bougrier et al.
5542008). Tyagi et al. (2011) stated that thermal pretreatment causes 40-60% solubilization of
555WAS and 20-35% solubilization of primary sludge. It was reported that the dry solids
556reduction was 55 and 35% for the WAS and primary sludge, respectively. At a treatment of

55790°C, it was observed that the VSS degradation increased to 36% and the methane production
558increased to 100% [22]. The sludge solubilization was reported to increase to 80% with 71%
559degradation of tCOD and a 59% increase in TS reduction. The thermal pretreatment enabled
560the HRT to decrease to 5 days. At an HRT of 2.9 days (at 175°C, 40 min), a 65% TSS
561reduction was observed. The table below shows the effect of thermal pretreatment obtained
562from different studies. Thermally pre-treated sludge before AD can enhance the methane
563production from 60 to 70% [22]. It was observed that the increase of methane production and
564sludge COD solubilization are linearly correlated up to a temperature of 190°C [54]. The
565increase in methane production is governed by the initial biodegradability of sludge. It was
566noted that thermal pretreatment has a better impact on less biodegradable sludge, for
567example, WAS showed a better methane yield after digestion than primary sludge. Thermally
568pre-treating the sludge at 170°C for 15 min results in a 92% higher biogas production
569[16,64,65]. However, a drawback to thermal pretreatment is the potent foul smell that is due
570to compounds, such as dimethyl disulfide, toluene, dimethyl trisulfide and 1,2,4-
571trimethylbenzene, which are present in the emitted gas. It is also known that some volatile
572organic compounds can be carcinogenic or toxic when inhaled; thus, the emitted gas from
573thermal treatment needs to be controlled [66].

574 Table 3 below shows the effect of thermal pretreatment at different temperatures and
575different sludge types on the dewaterability, the biogas production, and the digestibility of the
576mentioned sludge.

577Table 2: Performance of thermal pretreatment on different types of sludge

Substrate	Temperature/°C	Dewaterability	Biogas Production	Digestibility	References
Sewage	150	Improved at 180°C	—————	At a higher temperature, refractory compounds formation occurs	[55]

WAS	175	Improved at 175°C	60-70% increase in CH_4 production	Digestibility was improved	[55]
WAS	170	_____	_____	40-60% enhancement in solubilization	[22]
Primary	170	_____	_____	20-35% solubilization	[22]
WAS	170 for 6 min	_____	100% CH_4 production	30% VSS degradation HRT reduced to 5 days	[22]
WAS	121 for 30 min	_____	34% CH_4 production	30% VS reduction, 67.8% sCOD removal	[22]
Mixed Sludge	170 for 30 min	84% of the water quantity removed	74% increase in specific CH_4 production	_____	[64]
WAS	70	_____	28% increase in CH_4 production	Decreased the HRT from 15 to 13 days	[54]
WAS	70 for 9 hrs	_____	58% increase in biogas	_____	[54]
Primary	70 for 30 min	_____	48% CH_4 production	_____	[64]
WAS	170 for 30 min	_____	78% CH_4 production	_____	[64]
WAS	190 for 30 min	_____	23% CH_4 production	_____	[64]

578

579The thermal pretreatment technique is a promising and well-established technique since it has
580been implemented at full scale. There are several technologies based on the thermal
581pretreatment method used in industries, such as the CAMBI and BIOTHELYS technologies
582(*CambiTHP Technologies*, 2012). Subsequently, thermal pretreatment undeniably enhances

583the AD performance; it can be further investigated for the treatment of POME since POME is
584discharged at a high temperature of 80-90°C.

585 4.3.2 Microwave irradiation

586 Microwave irradiation (MI) was observed to efficiently solubilize the COD
587particulates of waste sludge with diameters less than 0.45 mm [68]. The sCOD/tCOD of
588waste activated sludge was noted to increase from 8% (control) to 18% after MI at a
589temperature of 72.5°C and from 6% (control) to 18% after MI at a temperature of 96°C.
590Increases in the sCOD/tCOD ratios of 19% and 21% were observed for WAS, irradiated to
59191°C and 100°C, respectively. Similarly, Eskicioglu et al. (2006) observed that the sCOD of
592MI pre-treated WAS at 96°C was 143% higher than the control. The variability in the
593sCOD/tCOD ratios of WAS pre-treated to similar temperatures is likely due to different
594sludge sources and extracellular polymeric substance characteristics. At 175°C, MI achieved
59554% of the ultimate chemical solubilization ratio (sCOD/tCOD) [68]. Yu *et al.* (2010) and
596Tyagi and Lo (2013) agreed that MI increases the solubilization of COD, thus demonstrating
597that the MI technique assisted in the disruption of intricate WAS floc structures and released
598extracellular and intracellular biopolymers into the medium. It was noted that the COD
599solubilization increased by 300% from an initial 8% after MI pretreatment of WAS with a TS
600content of 5.4% at 96°C. Furthermore, with a higher treatment temperature of 175°C on WAS
601with a TS content of 3%, the sCOD increased from 9% to 35%. This finding indicates that the
602increase in the sCOD rate is proportional to the increase in the temperature for the MI
603treatment.

604 The MI pretreatment had a good impact on methane production. Eskicioglu et al.
605(2006) observed 211% higher cumulative biogas production compared to the control at the
606end of 23 days of the mesophilic AD when MI pre-treated at 96°C. They observed that in a
607temperature range of 50–175°C with acclimatized inoculum, all pre-treated digesters

608 improved the cumulative biogas production and the pre-treated sludge irradiated at 1.2°C/min
609 to 175°C produced the highest cumulative biogas production, which was 31±6% higher than
610 the control after 18 days of digestion [68]. Tyagi and Lo's (2013) study resulted in a 64% and
611 79% enhancement in the tCOD removal and methane production, respectively. They also
612 noted a reduction in the SRT from 15 days to 8 days. The VS reduction of MI pre-treated
613 sludge was observed to be higher than that of conventional heating and was attained in a
614 lesser amount of time. Sun *et al.*, (2016) found that after 20 days of AD, the MI-Alkaline
615 Carbon Fibre process at an alkaline pH attained an increase in biogas production of 63.7% in
616 comparison to the untreated experiment.

617 It can be concluded that MI efficiently disintegrates the sludge by increasing the
618 readily decomposable component in the liquid phase of the sludge, which in due course
619 enhances the VS reduction, improves sludge dewaterability, biogas production all through
620 AD and radically reduces the SRT. MI can potentially produce environmentally safe
621 pathogen-free sludge, diminishing the exposure to pathogens and enabling the treated
622 substrate to be used for land applications (Tyagi and Lo 2013). MI is a technique that is worth
623 investigating for POME treatment.

624 4.4 Ultrasonic Pretreatment

625

626 Most researchers tend to evaluate the sludge disintegration efficiency with the sCOD
627 parameter. However, Pilli *et al.* (2011) state that there is more at stake than just the sCOD in
628 regard to ultrasonic pretreatment since sludge disintegration depends on several factors, such
629 as the sludge type, power supply, TS content, ultrasonic frequency, temperature, ultrasonic
630 density, sonication time and the sludge characteristics. Ultrasonication has no impact on the
631 tCOD; as an alternative, the sCOD/tCOD ratio is used to determine the solubilization of
632 organic matter from the solid to liquid state. There is a linear correlation between the degree

633of disintegration and the applied ultrasonic density [74]. The degree of disintegration
634increased more than 2-fold when the intensity was increased from 6 W/cm² to 18 W/cm².
635When higher mechanical shear forces are applied using higher intensities, they disrupt the
636cell walls of the microorganisms, which thus increases the solubilization of the COD. The
637latter, in turn, improves the degree of disintegration. The ultrasonication increases the
638concentration of COD, protein, and carbohydrates by disrupting the flocs and disintegrating
639the cell walls of the microorganisms that discharge the extracellular organic compounds
640confined in the bacterial flocs. The effect of the TS concentration on the degree of
641disintegration was studied; with an increase in the TS concentration of sludge, the
642solubilization of sCOD increased up to an optimum value, beyond which, the solubilization
643of sCOD declined due to the attenuation effect. The floc disintegration efficiency was
644unfavourably affected at a higher TS content (6%), an explanation for this observation is due
645to the disruption in the generation of cavitation bubbles at a higher solids concentration and
646attenuation in the sonication intensity in the aqueous medium. For instance, the sCOD is
647augmented from 1000 mg/L to 1800 mg/L, 4000 mg/L, 5800 mg/L and 3200 mg/L at the TS
648contents of 0.98%, 1.7%, 2.6% and 3.6% w/w, correspondingly. Pérez-Elvira *et al.* (2009)
649found that the solubilization is directly proportional to the energy applied, which is in
650agreement with Pilli *et al.*'s (2011) study. They also found analogously to [76], that for the
651same energy applied it is more efficient to increase the power rather than the sonication time.
652For instance, for a similar applied energy of 100 J/mL of sludge at 180 W for 10 s, the
653treatment gave 24% sCOD, while the 26 W for 60 s treatment gave a 7% sCOD.

654 It was observed that the particle size of the waste dictates the particle solubilization
655rate. Pilli *et al.*, (2011b) and Tyagi *et al.*, (2014) reported the impact of sonication on AD in
656continuous digesters and found that the biogas production rate increases for sonicated sludge
657with a lower HRT. For example, at a retention time of 2.5 days, the biodegradability

658improved by 60%, and the gas conversion efficiency improved by 40%. The biogas
659production rate was enhanced to the point where the retention time was less than 2.5 days.
660The VS reduction in the control experiment was 45.8%, and in the pre-treated experiment VS
661reduction was 50.3%, with a residence time of 22 days. The VS concentration in the effluent
662of the pre-treated sample was observed to be 10% less than that of the untreated AD effluent.
663The effect of increasing the sonication time and frequency of sonication during pretreatment
664caused the VS reduction in an anaerobic digester to increase gradually. The specific methane
665yield declined with an increase in the HRT, for instance, at 15 days HRT, the methane yield
666was 61% more than the untreated sample, whereas the methane yield was only 41% higher
667compared to the control at 25 days HRT. It is postulated that the increase in the specific
668methane yield occurs due to the increase in the net surface area of the particles and the
669disintegration of complex organic compounds [74]. Ultrasonic pretreatment has been
670implemented on a full scale in the water treatment industries [22]. Ultrasonic pretreatment
671may be an effective method of POME pretreatment since the amount of biogas produced may
672cover the usage of the energy of the ultrasonicator. The ultrasonic pretreatment technique is
673technical, environmentally, and economically feasible.

674Some pretreatment techniques tested on POME include all the techniques mentioned above,
675as well as photocatalytic treatment, pyrolysis, and steam fractioning. Photocatalytic treatment
676is a potential POME treatment since POME is composed mostly of water (more than 90%)
677and organic contaminants, which act as electron donors. It is a potential feedstock for
678photocatalytic water splitting process to generate H₂ [78]. A study on the pyrolysis of banana
679and orange peels was performed at different temperatures to produce biochar. The latter was
680studied as an adsorbent in the treatment of POME. The use of the produced biochar to treat
681the POME showed a removal efficiency of up to 57% in reducing the concentrations of
682BOD, COD, TSS, and O&G of POME to a satisfactory level below the environmental

683standards stipulated by the Malaysian Government [79]. While in a steam reforming process
684that falls under gasification pathway, biomass (a carbonaceous compound) can be thermally
685converted into combustible gaseous products, such as syngas, via interaction with steam at an
686elevated temperature, Chen et al. (2019) found that for the non-catalytic run, POME steam
687reforming replaced thermal decomposition and cracking, starting at a temperature of 873 K.
688When incorporating LaNiO₃ into the POME steam reforming, an enhanced syngas production
689was observed, while the reaction occurred at a lower temperature of 773 K [80].

690Nevertheless, further research has to be conducted on the techniques mentioned above to be
691able to assess their viabilities on the implementation of full-scale POME treatment. This
692paper focuses on biological, chemical, thermal, and Ultrasonic pretreatment. From the
693literature obtained, the most common pretreatment techniques conducted on POME were
694chemical pretreatments in terms of acid and alkali pretreatments by using different acids and
695bases along with varying the concentrations of the latter [14,81].

696 5. Biological Pretreatment of POME

697

698 Biological pretreatment provides distinctive advantages in contrast to chemical or
699physical methods, as it is environmentally friendly; therefore, it neither causes pollution nor
700requires distinct equipment. Biological pretreatment can be categorized into two classes as
701follows: first, by adding industrial or endogenous enzymes prior to the AD processes and,
702second, by incorporating particular bacteria that can secrete specific enzymes. This method
703demonstrated satisfactory results in enhanced biodegradation, dewatering characteristics of
704the sludge, improved methane production, and is widely researched in lab- and full-scale
705plants [45,82–84]. Most biological pretreatment studies undertaken on POME used
706microalgae and enzymes. The results of these studies are described below.

707 Rasit and Ooi (2018) conducted a study to develop and characterize a
708biocatalytic garbage enzyme as well as to investigate its influence on
709POME as a pretreatment process before AD. They produced the garbage
710enzyme by fermenting a blend of molasses, pre-consumer supermarket
711residues, and water at a concentration ratio of 1:3:10 for three months.
712They evaluated the influence of the produced garbage enzyme on the oil
713& grease (O&G), TSS, and COD of POME. Different dilutions of 5%, 10%,
714and 15% of garbage enzyme to POME samples were investigated over six
715days. The results showed that the garbage enzyme consisted of a
716biocatalytic enzyme, for instance, amylase, protease, and lipase were
717detected. The pretreatment showed a 90% removal of O&G in the 15%
718dilution of the garbage enzyme. While the TSS and COD reduction in a
719dilution of 10% of the garbage enzyme were recorded to be 50% and 25%
720respectively, the 5% dilution of the garbage enzyme achieved a
721percentage reduction of TSS and COD of only 32% and 13%, respectively
722[85].

723 Studies concerning POME treatment using photosynthetic microalgae remain new and
724few. Many challenges and limitations have been encountered with such a system, particularly
725the technical practicability of upscaling, dewatering, and harvesting of microalgal biomass.
726Attributable to these drawbacks, only a few ventures in microalgae industries have been made
727on POME treatment, and research concerning the practicality of microalgae for POME
728treatment is only limited to the laboratory scale [85]. Previous studies concerning biological
729pretreatment have reported that POME can be treated using *Spirulina plantesis* and other
730commercial strains, exhibiting significant reductions in the nitrogen, phosphorus, and COD
731concentrations [86].

732 Nur et al. (2017) showed the effect of 3 different strains of microalgae on POME
733wastewater at a low concentration. They observed a decreasing growth rate with an
734increasing POME concentration, except when treated with *C. vulgaris*, which exhibited an
735increasing growth rate with an increasing POME concentration. The COD content was
736decreased along with organic carbon, which was consumed as the source of energy. The lipid
737and carbohydrate contents were also affected by the POME concentration, except when
738treated with the *D. salina* strain, which showed no significant improvement. The best option
739for POME treatment was noted to be *C. vulgaris* due to the carbohydrate and lipid
740productivity and high COD removal ability [87].

741

742

743 6. Chemical Pretreatment of POME

744

745 Chemical pretreatment consists of using different types of chemicals to aid the hydrolysis
746step of AD. Chemical pretreatments are known to increase the surface area of the complex
747organic matter by causing it to swell, thus making the treated waste more vulnerable to
748enzymatic attack by microorganisms [50]. In the present study, chemical pretreatment is
749divided into the following two categories: first, acid and alkali pretreatment (Section 5.1-5.2)
750and, second, chemical reactions, such as ozonation and peroxidation (Section 5.3-5.4).

751

752 6.1 Acid Pretreatment

753

754 The following studies analyse the effect of different acid treatment conditions on
755POME to enhance the hydrolysis in AD. Safa et al. (2017) carried out a study to ascertain the
756pretreatment parameters, such temperature, hydrolysis extent and acid concentration, that can
757produce the highest amount of monomeric sugars, which will be used to generate

758 biohydrogen from POME through mixed culture dark fermentation. Phosphoric acid and
759 nitric acid were utilized for the pretreatment of POME. The highest production of
760 biohydrogen was attained when using a pre-treated POME in contrast to raw POME by a
761 mixed culture. POME pre-treated with 0.8% (w/v) phosphoric acid was found to yield 97%
762 higher amounts of biohydrogen from dark fermentation than the control. POME pre-treated
763 with 1% (w/v) of nitric acid displayed an enhancement of 65% in biohydrogen production in
764 comparison to the control (untreated POME) [14].

765 Based on Parviz et al.'s (2011) study, the chemical pretreatment was carried out by
766 the addition of 0.1% (v/v) chloroform into the sludge for a duration of 24 h at ambient
767 temperature. The acid pretreatments were undertaken by regulating the pH of the sludge to
768 3.0 by adding HCl (6N), which was maintained for 24 h, and to adjust the pH for AD to 5.5, a
769 NaOH solution (2N) was added. They observed that the acid pretreatment method resulted in
770 a nearly 51% COD removal efficiency, while the biohydrogen produced from this method
771 was 0.32 mmol H₂/g COD compared to the control sample, which achieved a COD removal
772 of 66%, with a minimum biohydrogen yield of 0.12 mmol H₂/g COD [88].

773 In another study, POME enriched with *Thermoanaerobacterium* was pre-
774 treated with a HCl concentration range of 0-2.5% w/v by stirring for 30
775 min, followed by adjusting the pH, which is the investigating parameter,
776 with 1 M NaOH. It was found that the organic matter solubilization by acid
777 pretreatment increased up to 15.7% of the preliminary sCOD content of POME, while a
778 carbohydrate solubilization of 32% of the initial soluble carbohydrate in POME was achieved
779 [89].

780 6.2 Alkali Pretreatment

781

782 Parviz et al. (2011) also investigated the effect of alkali treatment on POME before
783 AD. The alkali pretreatment was carried out by regulating the pH of the sludge to 12.0 by

784adding NaOH (6N), which was subsequently set aside for 24 h, and the pH was adjusted to
7855.5 by adding HCl (1N). For alkali pretreatment methods, a COD removal efficiency of
786nearly 59% was achieved along with a biohydrogen production of 0.37 mmol H₂/g COD [88].
787It was observed that the alkali pretreatment method proved to be more efficient in terms of
788the COD removal as well as the biohydrogen yield compared to acid treated POME.
789Seengemyoung et al. (2013) investigated the effect NaOH on POME at a concentration range
790of 0-2.5% w/v, by stirring for 30 min, followed by adjusting the pH with 1 M
791HCl. Organic matter solubilization for the alkaline pre-treated sample improved up to 28% of
792preliminary sCOD in POME, which corresponded to a carbohydrate solubilization of 41% of
793the initial soluble carbohydrate content. A total biohydrogen production of 4.6 l H₂/L-POME
794was attained from pre-treated POME with 1.5% w/v NaOH, which was 3 times superior to
795untreated POME and 1-fold superior to POME pre-treated with 1.5% w/v HCl. They also
796found that the optimal conditions for improved biohydrogen production from POME pre-
797treated with alkali using *Thermoanaerobacterium*-rich sludge was at an initial pH of 5.5 and
798a temperature of 60°C, which produced the highest biohydrogen yield of 5.2 l H₂/L-POME,
799which resulted in an increase of 51% in comparison to the control untreated raw POME [89].
800In another study where the effects of thermo-alkaline pretreatments were investigated, Chou
801et al. (2010) studied the effects of the NaOH concentration, temperature, and reaction time on
802the solubilization of POME by using the Response Surface Methodology (RSM). The results
803showed that the NaOH concentration, temperature, and reaction time had all distinct
804substantial impacts on the solubilization of POME. A maximum solubilization of COD of
80582.63% was estimated under the optimal conditions of 32.5°C, 8.83 g/L of NaO, and a 41.23
806h reaction time [90].

807

808 6.3 Ozonation Pretreatment

809

810 Ozone is a robust oxidant that can oxidize different types of organic and inorganic
811 compounds. Ozone has sturdy cell lytic activity, which can eradicate the microorganisms
812 present in sludge while further oxidizing the organic substances discharged from the cells.
813 Sludge ozonation is commonly defined as the successive decomposition reactions of the
814 disintegration of flocs, solubilization, and subsequent oxidation of the released organic matter
815 into carbon dioxide, also known as mineralization. Ozone reacts with complex molecules,
816 such as proteins, lipids, and polysaccharide constituents of the cell membrane, and converts
817 the latter into smaller molecules with lower molecular weights. Thus, the cell membrane is
818 ruptured, resulting in the release of cytoplasm in the form of sCOD [22]

819 Sumate et al. (2010) studied an ozonation pretreatment undertaken on POME using
820 the anaerobic sequencing batch reactor (ASBR). The ozone loading rate was 4000 mg/L
821 (generated from Trailgaz, Model 5LO), which accounted for an ozone dosage of 0.4 gO₃/L
822 POME or 9.7 mg O₃/g TS. They observed that ozonation increased the BOD/COD by 37.9%,
823 with a COD removal of 3.3%. The ozonated POME achieved a significantly higher removal
824 of TCOD at loadings of 6.52 and 9.04 kg COD/m³/d, but the experiment was unsuccessful at
825 a loading of 11.67 kg COD/m³/d. The generated RSM results showed that higher load
826 conditions (6.52–11.67 kg COD/m³/d), longer HRT, and shorter cycle times achieved higher
827 methane yields and organic removal efficiency [91].

828 Pisutpaisal et al. (2014) studied the effect of ozonation on different
829 POME concentrations; the ozonated POME was formulated using pre-
830 settled POME ozonated (using Ozonator, model OZ-553, Thailand) with an
831 ozone loading rate of 300 mg/h (mg COD: mg ozone = 102.8). AD was set
832 up at a pH of 4.0-6.0, with a POME content varying from 5,000 to 37,000
833 mg/L undertaken at a mesophilic condition (37°C). They observed that a
834 pH of 6.0 was the optimal pH where the highest hydrogen yield of 28.3

835mL/g COD was attained. The hydrogen production with ozonated POME at
836a concentration of 30,000 mg/L demonstrated a yield of 182.3 mL/g COD,
837achieving 49% higher solubilization than that of raw POME. At a COD
838concentration of 25,000 mg/L ozonated POME, the highest production rate
839of 43.1 mL/h was observed, while a COD removal of 44% was achieved
840with a COD concentration of 15,000 mg/L ozonated POME [92]. Tanikkul and
841Pisutpaisal (2014) investigated the effect of ozone pretreatment of POME under a
842thermophilic AD condition. The batch AD experiment was undertaken at a temperature of
84355°C. Ozonated POME was prepared using pre-settled POME with an ozone loading rate of
844300 mg/h and with a ratio of COD to ozone of 102.78. The concentration of POME was
845varied from 5,000 to 35,000 mg COD/L. They observed that the biohydrogen generation with
846ozonated POME reached the highest yield of 77.1 mL/g COD at 35,000 mg COD/L, which
847was 20% higher than that of the untreated POME. They also noted that a biohydrogen
848production rate of 10.8 mL/h was achieved at the ozonated POME concentration of 25,000
849mg COD/L. The COD removal in the ozonated POME was 25% more significant in
850comparison to that of the control experiment [93].

851 6.4 Peroxidation Pretreatment

852

853 The resources on peroxidation or advanced oxidation processes (AOPs) as pretreatment
854proved to be very limited. Most of the research found was focusing on AOPs undertaken on
855POME as a polishing technology, which is also known as the tertiary treatment, which takes
856place after AD or the coagulation-flocculation treatment, which will be discussed later in this
857paper. Lim et al.'s (2017) study was the only peroxidation pretreatment study undertaken on
858POME as a pretreatment method. They used the electro-Fenton technique for the treatment of
859POME by enhancing OH• radical production from hydrogen peroxide (H₂O₂) under a low
860voltage input (1.5–6.0 V). An electro-Fenton system was configured using graphite as the

861cathode and stainless steel as the anode. They observed that the results were presented in
862terms of COD removal efficiency. They found that the optimal conditions to breakdown
863organic matter in POME were a 4 h retention time with a concentration of 0.05 M of H₂O₂
864along with 0.10 M of a FeSO₄ catalyst, and a power input of 1.5 V. A high COD removal
865efficiency of 94% was obtained. The electro-Fenton treatment was observed to have a higher
866efficiency than the conventional Fenton treatment, which achieved a COD removal efficiency
867of only 48% [94].

868 7. Thermal Pretreatment of POME 869

870 7.1 Conventional Heating 871

872 Thermal pretreatment is a process where heat is introduced in a system to raise the
873temperature of the sludge to ameliorate disintegration and solubilization of solid sludge
874significantly. Thermal pretreatment is a reliable and commercially implemented technique
875[16,64]. The thermal energy necessary is usually achieved through direct steam injection or
876passing steam through the heat exchangers. Most studies were undertaken between the
877temperatures of 60°C and 270°C. Thermal pretreatment is classified into the following 2
878categories: first, temperatures higher than 100°C and, second, temperatures lower than
879100°C. However, studies on low-temperature thermal pretreatments are scarce. The primary
880purpose of the sludge thermal pretreatment technique is to improve the sludge
881biodegradability and methane production at the lowest possible retention time in an anaerobic
882digester.

883 7.1.1 Optimum Temperature

884 Most of the research undertaken [20,21,54,55,64] reported an optimum temperature of
885120-180°C, treatment times from 30- 60 min, and the pressure associated with temperature
886might vary from 600-2500 kPa [54]. It was also observed that the treatment time has little

887 impact on this temperature range. However, treatments at an excessively high temperature (>
888 190°C) lead to a decrease in the sludge biodegradability, even with high solubilization
889 efficiencies. This occurrence can be explained by the Maillard reactions, during which the
890 carbohydrates and amino acids form melanoidins, which are nearly impossible to degrade
891 [64]. It is, therefore, essential to find the appropriate temperature whereby the amount of
892 biogas being produced can easily compensate for the amount of energy input in the system to
893 raise it to the latter.

894 7.1.2 Conventional heating thermal pretreatment of POME

895 The thermal pretreatment study was carried out by heating the sludge to 100°C for 1 h
896 in a water bath [95]. In Parviz et al.'s (2011) study, the thermal pretreatment referred to as
897 heat-shock pretreatment was noted to be the most efficient in enhancing the biological H₂
898 production when cultivated for up to 48 h, which appeared to concur with the levelling out of
899 the COD removal efficiency. The H₂ yield from the pre-treated experiment was 0.41 mmol
900 H₂/g COD, while the H₂ yield from the control experiment was the lowest, i.e., 0.12 mmol H₂/
901 g COD. The H₂ yield following the heat-shock treatment was 3.4 times higher than that of the
902 control. The heat-shock pretreatment showed a maximum COD removal efficiency of 86%
903 in concurrence with an H₂ yield of 0.41 mmol H₂/g COD [95]. In the study conducted by
904 Zinatizadeh et al. (2017), where the thermal pretreatment was undertaken by heating the
905 sludge to 90°C for 1 h in a water bath, they observed that the thermally pre-treated granular
906 sludge exhibited the highest biohydrogen production (127 mL) at 72 h incubation. They
907 established that the cumulative biohydrogen yield of the thermal pretreatment was 40% and
908 76% higher than the chemical pretreatment method and untreated POME, respectively [81].

909 As per the study mentioned above, the treatment temperatures investigated were 90-
910 100°C, which is relatively close to the discharge temperature of POME; this, in turn, may be
911 insufficient to breakdown the organic matter, which reduces the efficiency of AD. Moreover,

912it was observed that the thermal pretreatment produces better results at the optimum
913treatment temperature of 120-180°C. It can be concluded that there is still much room for
914improvement in regard to the thermal pretreatment of POME.

915 7.2 Microwave Irradiation

916

917 The process of microwave irradiation (MI) consists of 2 properties, i.e., a thermal, and
918an athermal effect. The term athermal describes an influence that is not associated with an
919increase in temperature, whereas the thermal effect is governed by temperature. It was
920established that the athermal effect occurs due to polarized macromolecules aligning with the
921poles of the electromagnetic field, which results in the disruption of the existing Hydrogen
922bonds [71]. The thermal effect generates heat due to the absorption of MI by water, causing
923the organic matter to exhibit an induced or permanent polarization. This phenomenon results
924in the alteration of the secondary or tertiary structure of proteins.

925 The only study found was that of Saifuddin et al. (2009), which summarized the effect of
926MI on sludge disintegration. They observed that the ratio of sCOD to tCOD increased as the
927MI time increased. The latter showed that many cytoplasmic constituents of the sludge were
928released into the medium owing to cell wall disintegration. The samples started to over boil
929after 7 min of MI. The highest solubilization in terms of sCOD/tCOD ratios were 0.45, 0.65,
930and 0.77, which occurred after treatment times of 7, 9, and 11 min, respectively. For methane
931production analysis, a 58 mL/L-sludge of methane was produced after 3 min of MI, which
932accounted for an energy consumption of 252 kJ/L-Sludge. The latter resulted in a methane
933production 56.7% higher than the control.

934 8. Ultrasonic Pre-treatment of POME

935

936 Ultrasound propagates in a medium to generate rarefactions and compressions in cycles.
937Compressions exert positive pressure on the liquid, which pushes the molecules together. On

938the other hand, rarefactions use negative pressure by pulling the molecules apart.
939Microbubbles are formed owing to disproportionately large rarefactions. These microbubbles
940grow progressively through each cycle until they reach an unstable diameter and collapse
941violently, generating shock waves of a temperature of approximately 5000°C and a pressure
942of 500 atm [73]. This collapse is known as cavitation. Some characteristics that impact
943cavitation are the gas and particulate matter present in the medium, externally applied
944pressure, solvent viscosity, temperature, and surface tension. The cavitation process directs
945the disintegration of sludge.

946 8.1 Factors influencing Ultrasonication of Sludge

947

948 Factors, such as the ultrasound frequency, the specific energy input, the power input,
949and the sonication time, must be taken into consideration to achieve the most effective
950outcome from the ultrasonic pretreatment.

951 The ultrasonic wave frequency is a factor that plays a crucial role in the sludge
952pretreatment since the operating frequency directs the critical size of the cavitation bubbles.
953Low frequencies (<100 kHz) are known to improve the mechanical and physical degradation,
954while higher frequencies (>100 kHz) enhance the sonochemical properties. In sludge
955treatment, low frequencies (20-40 kHz) were observed to perform better than the higher ones.
956[77]

957 It was noted that pretreatment at a higher ultrasonic energy input was more effective
958than that at lower ones; it is often reported that the optimal specific energy input ranges from
9591000 to 1600 kJ/kg total solid (TS) for significant sludge solubilization and enhanced
960biological digesters performance. The specific energy input is a parameter that integrates the
961transferred power, sonication time, and sample mass [54].

962 The ultrasonic power input has a significant effect on the formation and behaviour of
963 the cavitation bubbles and sludge disintegration. In many studies [22,62,74,77], it was found
964 that the sonication process can be improved by enhancing the ultrasonic density while
965 keeping the sonication time short; this, in turn, saves energy.

966 8.2 Ultrasonic Pretreatment of POME 967

968 Budiman et al.'s (2016) study attempted to enhance the biohydrogen production by
969 implementing ultrasonic pretreatment onto a blend of effluents, consisting of 25% and 75%
970 (v/v) of POME and paper pulp mill effluent (PPME), respectively. The ultrasonication
971 treatment was operated at a frequency of 20 kHz. Ultrasonic irradiation was dispersed into
972 the effluents at the different amplitudes of 30%, 50%, 70% and 90% for durations of 5 min,
973 15 min, 30 min, 45 min, and 60 min preceding the photofermentation [96]. Ultrasound
974 irradiation was observed to efficiently solubilize organic matter from the solid to the liquid
975 phase with enhanced sCOD/tCOD in contrast to the control. The implementation of
976 ultrasonication augmented the solubilization of organic matter by increasing the sCOD/tCOD
977 ratio in comparison to that of the control in the range of 16 to 252% with the application of
978 different ultrasonication energy inputs ranging from 48 to 1245 J/mL. With an ultrasonication
979 energy input higher than 775 J/mL, relatively low solubilization and biohydrogen production
980 were observed; however, when the energy input was increased further (from 775 to 1031-
981 1245 J/mL), there was no notable alteration in biohydrogen production compared to the
982 control. For higher amplitude values when the ultrasonication duration was increased from 45
983 to 60 min, there was no significant change in the solubilization of organic matter. The highest
984 solubilization of substrate occurred at an amplitude of 70% with a duration of 45 min, an
985 amplitude of 70% with a duration of 60 min, an amplitude of 90% and duration of 60 min and
986 an amplitude of 90% and duration of 45 min with sCOD/tCOD ratios of 0.85 ± 0.05 ,
987 0.87 ± 0.01 , 0.87 ± 0.02 and 0.88 ± 0.01 , respectively. After 72 h of photofermentation, the

988 biohydrogen productions using an amplitude of 70% with a duration of 45 min and an
989 amplitude of 90% with a duration of 45 min as treatment conditions achieved improved H₂
990 yields of 86.8% and 88.4%, respectively, compared to the control [96].

991 In another study undertaken by Leñaño et al. (2012), they observed that the highest
992 COD removal of 58.07 and 62.24% occurred at ultrasound doses of 143 and 195 J/mL,
993 respectively, compared to the control, which had a COD removal of 52.08% after 10 days of
994 fermentation. The biohydrogen production increased from 0.64 to 1.12 mmol/gCOD when
995 treated with an energy input of 143 J/mL, while when treated with an energy input of 195
996 J/mL, the latter increased from 0.68 to 1.19 mmol/gCOD [97]. Saifuddin et al. (2009) stated
997 that the most critical parameter in ultrasonic pretreatment is the sonication time. The
998 ultrasonic intensity slightly affects the sCOD/tCOD ratio. The highest ratio of sCOD/tCOD
999 achieved was approximately 0.32 after a 30 min treatment using an ultrasonic intensity of 0.5
1000 W/mL, whereas the sCOD/tCOD ratio was approximately 0.29 after a 30 min treatment using
1001 an ultrasonic intensity of 0.20 W/mL compared to the control experiment, which had a
1002 sCOD/tCOD ratio of 0.11 [98]. The solubilization of POME in terms of the sCOD/tCOD
1003 ratio was recorded to be 0.14 and 0.29 when subjected to ultrasonic treatment durations of 10
1004 and 30 min, respectively. In the ultrasonic pretreatment, approximately 44 mL of methane
1005 was generated after 10 min of treatment, which corresponded to an energy consumption of
1006 120 kJ/L-Sludge. These conditions for ultrasonic pretreatment showed an increase of 19% in
1007 methane production over the control.

1008 The studies above show that ultrasonic pretreatment has the potential to increase
1009 solubilization as well as biogas or biohydrogen production depending on the required
1010 applications. However, the latter must be more thoroughly studied to reduce the consumption
1011 of energy.

1012 **8.3 Combined Microwave and Ultrasonic pretreatment of**
1013 **POME**
1014

1015 An alternative pretreatment technique investigated was combining the microwave and
1016ultrasonic pretreatment techniques. Saifuddin et al. (2009) observed that the maximum ratio
1017of sCOD/tCOD achieved was 29% after 30 min of ultrasonic treatment, whereas the latter
1018reached 45% solubilization after 7 min of MI. The BOD/sCOD ratio improved after the
1019pretreatments, implying that the disintegration of the soluble organic material improved
1020during the treatment; they observed that the total volatile fatty acid (TVFA) released was
1021increased after both pretreatments were conducted, with the MI treatment displaying a higher
1022yield of TVFA. In the combined pretreatment, with treatment conditions of 3 min of MI and
102310 min of ultrasonic, approximately 98 mL/L-Sludge of methane was produced, which
1024accounted for nearly 164% more methane in contrast to the control. The maximum
1025improvement in methane production was observed at 3 min MI, followed by a 10 min
1026ultrasonic treatment. This study shows that the use of MI pretreatment combined with
1027ultrasonic pretreatment not only produces better results; the amount of energy required for the
1028combined treatment is less than using the ultrasonic pretreatment individually. In turn, the
1029amount of energy consumption can be compensated since the amount of biogas produced is
1030higher than the conventional method. The microwave, in combination with the ultrasonic
1031method, would be a rapid and economical method for sludge pretreatment for the
1032enhancement of biogas production[98].

1033 **9. Coagulation treatment of POME: Coagulation and**
1034 **Flocculation, Electrocoagulation, Chemical**
1035 **Coagulation**
1036

1037 This section reviews the main coagulation pretreatment methods that have been
1038studied on a laboratory scale for the POME treatment. The coagulation method consists of a

1039 few different categories of coagulation, for example, coagulation and flocculation,
1040 electrocoagulation and chemical coagulation. Several studies have been undertaken to
1041 optimize the AD of POME [99–102]. However, none have been implemented on a full scale,
1042 since many factors need to be taken into consideration.

1043 Table 3 below shows the results obtained for coagulation treatment. Bhatia et al.
1044 (2007) investigated the effect of *Moringa oleifera* seeds (MOAE), which are described as an
1045 environmentally friendly and natural coagulant for the pretreatment of POME. They observed
1046 that adding NALCO 7751 as the flocculent yields better results compared to using MOAE as
1047 a coagulant individually. It was also noted that the coagulation-flocculation process resulted
1048 in better SS removal and COD reduction when undertaken at a lower temperature of 30°C
1049 compared to 70°C. The dewaterability was improved as the sludge volume index was
1050 observed to have decreased at the end of the experiment. The turbidity of POME was
1051 enhanced [99].

1052 The effect of electrocoagulation on POME was investigated by [101]; using a current
1053 density of 20 A/m² for 5 min at 313 K and pH 5, they obtained an optimal percentage of oil,
1054 COD, SS, and TS removal by electrocoagulation. They noted that the electrical consumption
1055 under the optimum condition was 0.10 kWh/m³ and that by adding NaNO₃, the
1056 electrocoagulation was improved. At high current densities and a long electrocoagulation
1057 time, the POME treatment produced electrolytic bubbles, which hindered the oil removal,
1058 reducing the efficiency of the process. However, chemical coagulation limits the oil removal
1059 from POME due to the higher pH condition. Agustin et al. (2008) observed that the decrease
1060 in a 5 day BOD test (BOD₅) and COD confirmed partial removal of the dissolved organic
1061 substances from POME after electrocoagulation. The mean values for COD and BOD₅
1062 prior to and after electrocoagulation were significantly dissimilar at the 95% confidence
1063 level. The mean COD level in POME before electrocoagulation was 36,800 mg/L, which

1064 decreased to 25,600 mg/L. The BOD₅ reduced from 23,400 mg/L to 14,400 mg/L. From
 1065 the studies mentioned above, it is important to note that electrocoagulation is less
 1066 environmentally damaging compared to conventional chemical coagulation. However, the
 1067 downside of the process is the formation of scum or electrolytic bubbles that affect oil
 1068 removal; this phenomenon was not observed in the conventional coagulation process.

1069 The following four different conditions for coagulation for POME pretreatment were
 1070 investigated by [102]: coagulation by chitosan, the addition of ferrous sulfate (FeSO₄),
 1071 chitosan with hydrogen peroxide (H₂O₂) and chitosan with Fenton oxidation. They concluded
 1072 that the process that yielded better results was the treatment by chitosan with H₂O₂.

1073 Table 3: Coagulation pretreatment of POME

Conditions	Results	References
Coagulation and Flocculation		
1000-5000 mg/L MOEA, pH 3-7, settling time (30-50 min)	95.0% removal of Suspended Solid (SS), 52.2% COD removal	[100]
1000-5000 mg/L MOEA, 5000-9000 mg/L NALCO 7751, pH 3-7, settling time (30-50 min)	99.3% removal of SS, 52.5% removal of COD, 50.3% of water removal	[100]
coagulant dosage 15000 mg/L, flocculent dosage 300 mg/L, pH 6	Improved turbidity from 118 NTU to 19 NTU. Water removal of 78%	[99]
Electrocoagulation		
Current density 20A/m ² , 40°C, pH 5	72.0% oil removal, 64.0% COD removal, 53.0% removal of SS, 43.0% TSS removal.	[101]
Current 1.4-2.0 A, voltage 3.5-12.0 V, 6 hr, pH 4.3	30% COD removal, 38% BOD removal and removal of some heavy metals	[104]
Chemical Coagulation		

500-12,500 mg/L FeSO ₄ , 15-150 min, pH 2-7	62.6% COD removal, 98.7% removal of TSS	[102]
500-7500 mg/L H ₂ O ₂ , 15-150 min, pH 2-7	82.8% COD removal, 89.9% removal of TSS	[102]
500-12,500 mg/L Chitosan, 15-150 min, pH 2-7	70.2% COD removal, 85.6% removal of TSS	[102]
Fenton Oxidation, 2500 mg/L of Chitosan and FeSO ₄ , 500 mg/L H ₂ O ₂ , 15-150 min, pH 2-7	73.1% COD removal, 100% removal of TSS	[102]

1074

1075 10. Applicability of different pretreatment techniques 1076 on POME

1077

1078 Biological pretreatment consists of treating POME with biological agents, for
1079 example, enzymes, bacteria, or biological agents, such as mature compost. The amount of
1080 methane gas production and biodegradability using biological methods of treatment were
1081 observed to be relatively high, showing that the biological pretreatment is an effective
1082 technique. As aforementioned in section 4.0, most of the studies conducted with biological
1083 pretreatment were undertaken at a temperature of 70°C. The temperature in this technique is a
1084 fundamental factor since enzymes are sensitive to it. The latter can be problematic since the
1085 discharge temperature of POME is 80-90°C. In the application of biological pretreatment to
1086 the treatment of POME, it is crucial to identify whether the enzymes can perform at such a
1087 temperature, thus preventing the denaturation of the latter. If the enzymes denature, the
1088 structure of the enzymes loses its shape. Since the enzymes and substrates work like a lock
1089 and key arrangement once the enzymes are denatured, the substrates cannot fit into the
1090 enzymes' structure; therefore, the substrates cannot be digested [105]. Consequently, the
1091 whole process is rendered unproductive. Moreover, if biological pretreatment is implemented

1092 on a full scale, commercial enzymes must be continually prepared, which is a costly process;
1093 further economic analysis must be undertaken to know if this technique is feasible on a plant
1094 scale.

1095 As mentioned in section 4.2, acid pretreatment does not significantly enhance AD
1096 compared to the alkali pretreatment. Moreover, the fact that the effluent may require re-
1097 neutralization after acid pretreatment is detrimental in regard to the treatment of POME. It
1098 was found that the most affordable alkali pretreatment reagent was quicklime, Ca(OH)_2 .
1099 Using Ca(OH)_2 is an attractive option since it is inexpensive enough to be applied on a full
1100 scale. It was perceived that the methane yield using Ca(OH)_2 was considerably high.

1101 Nevertheless, since POME consists of more water, i.e., approximately 90% more than
1102 solid matter, when treated with Ca(OH)_2 , it may form hard water due to the presence of the
1103 Ca^{2+} ions [106]. This phenomenon will affect the anaerobic digesters' performance and may
1104 cause deposits in the digesters. The effluent quality after digestion may be of poor quality and
1105 may require softening.

1106 Ozonation pretreatment is an effective treatment that has advantages, such as it
1107 improves settleability, dewaterability, and biogas production. Ozonation is a full scale
1108 implemented technology that performs well and does not alter the pH or drastically impact
1109 the effluent quality. Ozonation is a reliable option for the treatment of POME since the latter
1110 is known to prevent the formation of scum and bulking. The latter will help to enhance the
1111 performance of the anaerobic digesters. Ozonation is a costly treatment that requires high
1112 energy during the process. For such a treatment to be economically feasible, it is crucial to
1113 know whether the amount of biogas produced can compensate for the high energy usage [22].

1114 Fenton peroxidation is a method tested only at laboratory scale. Peroxidation is
1115 beneficial in terms of disintegration and biogas production. In terms of POME treatment, this

1116pretreatment method does improve the process, but the low pH of the system may inhibit the
1117performance of the anaerobic digester, and the effluent may require re-neutralization to
1118conform to effluent quality standards. Furthermore, since it is an extremely corrosive process
1119due to the low pH condition, it may result in high operation and maintenance costs [22]. For
1120the pretreatment of POME, the addition of chemicals may not be an adequate technique since
1121the effluent quality is compromised and may necessitate re-neutralization or softening for the
1122effluent to conform to the environmental standards.

1123 Thermal pretreatment has many advantageous aspects. The thermal pretreatment
1124technique was implemented on a full scale a long time ago. The performance of the thermal
1125method is not only fool proof, it guarantees the destruction of high volatile solids (60-70%), a
1126lower digester volume, high biogas yield (~ 150%), well-dewatered cake (~ 35%) and
1127pasteurized products (i.e., A class biosolids). Since the optimum treatment temperature is
1128120-180°C and the discharged temperature of POME is 80-90°C, this implies that the amount
1129of energy required to raise the temperature of the process is less compared to an ambient
1130temperature system [64]. AD of POME is feasible for both mesophilic and thermophilic
1131conditions. However, thermophilic AD has not been performed on a full scale. The
1132thermophilic temperature of the digester can be maintained using a heat exchange from the
1133gas engine, where the waste heat is generally more than enough to cater for the operation
1134[4]. Another advantage of the conventional heating pretreatment technique for the POME
1135treatment is that the latter does not have a negative impact on the effluent quality.

1136 Furthermore, the biosolid produced after pretreatment is of better quality – so much so
1137that the biosolid can be used as fertilizer, primarily organic fertilizers, which may, in turn, be
1138a source of revenue. The treated effluent does not have to undergo additional treatment to
1139conform to environmental standards. Some advantages and disadvantages of thermal
1140pretreatment are listed below.

1141 Advantages of thermal pre-treatment:

- 1142 • As observed from many literature sources, e.g., [54,55], thermal pretreatment
1143 enhances the biodegradability and dewaterability of sludge;
- 1144 • Thermal pretreatment can increase methane production (by 60% and in some cases
1145 100%) and up to 36% decrease of VSS is noted;
- 1146 • Thermal pretreatment also enables the reduction of the hydraulic retention time
1147 considerably;
- 1148 • WAS biodegradability increases with temperature to an optimum near 175°C, after
1149 which a decrease in gas production and formation of inhibitory compounds is
1150 observed;
- 1151 • Thermal pretreatment enhances the sludge sanitation, reduces the sludge viscosity,
1152 and improves sludge handling;
- 1153 • No extra energy is needed due to excess biogas production and a positive energy
1154 balance.

1155 Disadvantages of thermal pretreatment:

- 1156 • May cause fouling of heat exchangers;
- 1157 • Formation of odourous compounds associated with thermal treatment;
- 1158 • High operation and maintenance costs are a significant drawback of the pretreatment
1159 method [64].

1160 The MI pretreatment technique, though not implemented on the full scale, can be a
1161 viable option for POME treatment since it has a positive impact on dewaterability and
1162 settleability [71]; both of these are desirable effects for a better performance of the anaerobic
1163 digester. MI has been proven to be more effective than ultrasonic pretreatment in terms of
1164 improved solubilization and less energy is required to raise the temperature of the system
1165 [98]. Further studies must be conducted to observe how the biogas production and energy

1166input respond to the pretreatment process. Correspondingly, the maintenance of the MI
1167device is relatively easy, along with the low-cost of the maintenance and operational
1168expenses [71].

1169 Ultrasonic pretreatment is a full scale implemented technique. This technique can be a
1170possible option for the treatment of POME since it enhances dewaterability, settleability,
1171solubilization, and biogas production [96]. The effluent quality after pretreatment is not
1172compromised, and no additional treatment is required. There are many parameters in the
1173ultrasonic pretreatment that impact on the technique's performance [77]. Therefore, the
1174fundamental parameters, such as the specific energy, ultrasonic frequency, power input, and
1175the sonication time, must be appropriately established to obtain the optimum condition for the
1176treatment. For POME treatment, additional research must be undertaken to comprehend how
1177the different parameters of the ultrasonic pretreatment affect the treatment process and if the
1178biogas production can compensate for the ultrasonicator's energy requirement. Though the
1179energy requirement of the ultrasonicator is relatively elevated, the operational and
1180maintenance costs are reasonable; furthermore, the installation and operation procedures of
1181the ultrasonicator are straightforward.

118211. Conclusion

1183

1184 Based on the information disseminated in this paper, it is possible to clearly
1185understand the impacts of different pretreatment techniques and their efficiency in enhancing
1186AD and biogas production. It can be established that biological pretreatment can be too costly
1187to commercialize and may therefore be economically unfeasible at the full scale for POME
1188treatment. Chemical pretreatment techniques compromise the treated effluent quality and
1189require the extra step of softening and re-neutralizing to conform to the standards. It should
1190be noted that compared to ultrasonic pretreatment, MI pretreatment is more efficient for the

1191solubilization of organic matter and requires much less energy to operate. Finally, it can be
1192concluded that the most promising pretreatment method is thermal pretreatment, whether MI
1193or conventional heating. Since the discharge temperature of POME is already high, less
1194energy is required to raise it to the optimal temperature for thermal pretreatment. Thermal
1195pretreatment has proved to enhanced solubilization, biogas production as well as treated
1196effluent quality. The increase in biogas production can easily compensate for the energy
1197added to the system. Moreover, thermal pretreatment in terms of conventional heating has
1198already been implemented on a full scale.

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1204Nomenclature

1205AD	Anaerobic Digestion
1206ACF	Alkaline Carbon Fibre
1207BOD	Biological Oxygen Demand
1208COD	Chemical Oxygen Demand
1209CPO	Crude Palm Oil
1210CST	Capillary Suction Time
1211DMDO	Dimethyldioxirane
1212DS	Dissolved Solid
1213FOG	Fat Oil and Grease
1214EPS	Extracellular Polymeric Substances
1215HRT	Hydraulic Retention Time
1216MI	Microwave Irradiation
1217OFMSW	Organic Fraction of Municipal Solid Waste
1218PAA	Peracetic Acid
1219POMS	Peroxymonosulphate
1220PPME	Paper Pulp Mill Effluent
1221sCOD	Soluble Chemical Oxygen Demand
1222SRT	Sludge Retention Time
1223SS	Suspended Solid
1224SVI	Sludge Volume Index
1225tCOD	Total Chemical Oxygen Demand
1226TS	Total Solid

1227TSS	Total Suspended Solid
1228VFA	Volatile Fatty Acid
1229VS	Volatile Solid
1230VSS	Volatile Suspended Solid
1231WAS	Waste Activated Sludge
1232WWTP	Waste Water Treatment Plant

1233References

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