



Research article

Investigation of struvite crystals formed in palm oil mill effluent anaerobic digester

Muzzammil Ngatiman^a, Mohammed Saedi Jami^{b,*}, Mohd Rushdi Abu Bakar^c, Vijaya Subramaniam^a, Soh Kheang Loh^a^a Engineering & Processing Division, Malaysian Palm Oil Board, Kajang, Selangor, Malaysia^b Faculty of Engineering, International Islamic University of Malaysia, Kuala Lumpur, Malaysia^c Faculty of Pharmacy, International Islamic University Malaysia, Kuantan, Pahang Darul Makmur, Malaysia

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ABSTRACT

The formation of struvite crystals or magnesium ammonium phosphate ($MgNH_4PO_4$) in palm oil mill effluent (POME) occurs as early as in the secondary stage of POME treatment system. Its growth continues in the subsequent tertiary treatment which reduces piping diameter, thus affecting POME treatment efficiency. Hypothesis. The beneficial use of the crystal is the motivation. This occurrence is rarely reported in scientific articles despite being a common problem faced by palm oil millers. The aim of this study is to characterize struvite crystals found in an anaerobic digester of a POME treatment facility in terms of their physical and chemical aspects. The compositions, morphology and properties of these crystals were determined via energy dispersive spectroscopy (EDS), elemental analysis, scanning electron microscopy (SEM) and x-ray diffraction (XRD). Solubility tests were carried out to establish solubility curve for struvite from POME. Finally, crystal growth experiment was done applying reaction crystallization method to demonstrate struvite precipitation from POME. Results showed that high phosphorous (P) (24.85 wt%) and magnesium (Mg) (21.33 wt%) content was found in the struvite sample. Elemental analysis detected carbon (C), hydrogen (H), nitrogen (N) and sulfur (S) below 4 wt%. The crystals analysed by XRD in this study were confirmed as struvite with 94.8% struvite mineral detected from its total volume. Having an orthorhombic crystal system, struvite crystals from POME recorded an average density of 1.701 g cm^{-3} . Solubility curve of struvite from POME was established with maximum solubility of 275.6 mg L^{-1} at pH 3 and temperature $40 \text{ }^\circ\text{C}$. Minimum solubility of 123.6 mg L^{-1} was recorded at pH 7 and temperature $25 \text{ }^\circ\text{C}$. Crystal growth experiment utilizing POME as the source medium managed to achieve 67% reduction in phosphorous content. This study concluded that there is a potential of harnessing valuable nutrients from POME in the form of struvite. Struvite precipitation technology can be adapted in the management of POME in order to achieve maximum utilization of the nutrients that are still abundant in POME. At the same time maximization of nutrient extractions from POME will also reduce pollutants loading in the final discharge.

1. Introduction

Struvite or magnesium ammonium phosphate ($MgNH_4PO_4$) has recently become the subject of interest in palm oil mill effluent (POME) treatment. This is due to the nuisance of uncontrolled crystal formation that is believed to be a struvite in POME treatment system jeopardizes the efficiency of the system. There have also been discussions on the potential of harnessing valuable elements from POME utilizing struvite precipitation technology. Precipitation of struvite from POME was considered as a rare subject in palm oil milling industry with only few

fundamental research studies explored on struvite formation from POME (Tadza et al., 2015; Salsabili et al., 2016). Other wastewater treatment industries such as swine, sewerage as well as cattle manure (Jordaan et al., 2010; Zeng et al., 2018; Rico 2011) have gone far with struvite precipitation as one of the methods to recover nutrients from waste stream. For example, in swine farming industry, struvite precipitation of its wastewater was explored with the purpose to remove ammonia nitrogen and simultaneously recover phosphate from its wastewater stream (Huang et al., 2016) (Taddeo et al., 2016). In dairy and municipal wastewater also, several works were done with the aim to recover

* Corresponding author.

E-mail address: saedi@iium.edu.my (M.S. Jami).<https://doi.org/10.1016/j.heliyon.2021.e05931>

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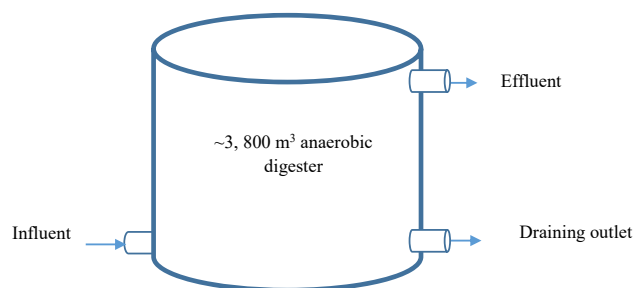


Figure 1. Schematic diagram of the anaerobic digester where sample was obtained.



(a)



(b)

Figure 2. Crystal sample (a) from anaerobic digester tanks, (b) after washed and dried.

phosphate mineral repurposing it to be alternative mineral fertilizer (Aleta et al., 2018; Hallas et al., 2019).

Phosphate recovery is measured by its removal percentage against its initial concentration in the wastewater. In struvite precipitation process, high yield of struvite means high percentage of phosphate removal which also means high removal of ammonia nitrogen as it also constitutes struvite. Removal of these elements from wastewater, on the other hand, produces a treated wastewater with less concentrations of PO_4^{3-} , Mg^{2+} and NH_4^+ which also mean less pollutants in its final discharge. Studies has also been carried out to experiment various methods in controlling crystal size, purity and yield of the struvite (Mersmann, 1995). It was also found that by optimization of several operation parameters (pH, temperature, reaction time and molar ratio), uncontrolled formation of struvite crystals as well as releasing of harmful minerals to the environment could be avoided (Rahman et al., 2014).

In palm oil mills, uncontrolled crystal formation happens primarily in the POME treatment system either in the piping setup or in the tanks that contain the POME for treatment process. This includes anaerobic digester tanks that have become increasingly popular. It can be in the form of bulk crystal or scale crystal that attaches itself to the inner wall of the tank. Crystal deposit formed in POME anaerobic digester tank is also believed

to be a form of struvite. Its formation is empirically influenced by the characteristics of the POME itself during anaerobic treatment stage. During treatment, POME values of pH (5–7), temperature ($\sim 36^\circ\text{C}$), concentration of Mg^{2+} ($\sim 380\text{ mg L}^{-1}$), NH_4^+ ($\sim 220\text{ mg L}^{-1}$) and PO_4^{3-} ($\sim 120\text{ mg L}^{-1}$) (Loh et al., 2013) are the main factors favorable for these crystal formations. Clogged discharge pipes, tank overflow, reduced flow rate and reduction in effective capacity of the digester tank are among the problems associated with these crystal formation in an anaerobic digester tank Muzzammil et al. (2018) In this study, crystal deposit samples were collected from a POME biogas anaerobic tank and the physical and chemical properties of the samples were determined in order to characterize the crystal deposit found in POME treatment system specifically in anaerobic digestion stage. This will contribute in helping the oil palm industry to identify measures and methods to effectively address the issue of crystal deposition in a POME treatment system.

2. Materials and methods

2.1. Sampling of POME crystal

POME crystal samples were collected from a $3,800\text{ m}^3$ biogas digester tank (Figure 1) in a 50 tonnes hr^{-1} palm oil mill located in west of Selangor, Malaysia. These samples were collected during a scheduled cleaning of the biogas digester. The crystals collected were in the form of non-uniform blocks with average maximum diameter of 26 cm. It was later crushed into pieces (Figure 2a), washed to get rid of POME sludge attached to them and dried in an oven at 105°C (Figure 2b).

2.2. Crystal morphology and elemental content

A scanning electron microscope (SEM), Hitachi S3400N, coupled with Bruker Xflash energy dispersive spectroscopy (EDS) were used to characterize the morphology of the POME crystal samples as well as determining the elements on the crystal's surface. The samples were ground for SEM/EDS analysis. An accelerated voltage of 10 kV was used and the probe current was set at $30\ \mu\text{A}$ with working distance of 10.4 mm. A CHNS analyser, LECO 628 Series from Leco Instruments, USA was used to determine the C, H, N and S contents. Prior to CHNS analysis, the crystals were pulverized and 0.15 g of the pulverized crystal was used for the analysis.

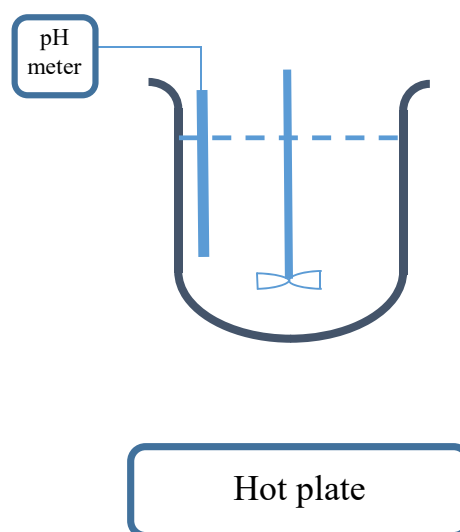


Figure 3. A solubility unit used in the test.

Table 1. Chemicals used in performing experiments and analysis in this study.

No	Chemical/Reagents	Model/Company	State
1	Sulfuric Acid, H ₂ SO ₄	R&M Chemicals, UK	Liquid
2	Sodium Hydroxide, NaOH	R&M Chemicals, UK	Solid

2.3. Crystal properties

The structure and crystal properties were determined via X-ray diffraction (XRD) using Advance D8 by Bruker AXS, Germany. Crystal density was determined using a density meter, ML204/02 from Mettler Toledo, Switzerland according to EN 61326-1. The samples were ground and sieved to meet the requirement of particle size of 50 μg or less for XRD analysis. For bulk density measurement, 10 pieces of the struvite were selected and the density of each was measured using standard balance with density meter kit XPR-S XSR-S from Mettler Toledo. The average value of density measured for each piece of the struvite was recorded.

2.4. Solubility test

Solubility test was conducted with the aim to establish a solubility curve uniquely describing the effect of two main parameters; temperature and pH, on the structural integrity of POME struvite crystal. This study adapted solubility test as was reported in Bhuiyan et al. (2007). The test was carried out using five sets of solubility units (Figure 3) that were subjected to various pH and temperature conditions. pH was regulated by using concentrated sulphuric acid and sodium hydroxide that will make up a 500 mL solvent together with distilled water as base medium (Table 1). Heat was introduced by using a hot plate for each solubility unit and was regulated with a standard mercury thermometer.

The pulverized struvite crystal was first weighed for approximately 200 mg using an analytical balance. 0.1 M of sulfuric acid solution and 0.1 M of sodium hydroxide solution were prepared. Using these two solutions, five solubility units were prepared in which the initial pH value for each of the solubility unit was set at 3, 5, 7, 9 and 11. The 200 mg pulverized struvite was then poured into each of the solubility unit. All the solubility units were then subjected to four sessions of stirring process where in each stirring process the temperature was set at specific value which were 25, 30, 35 and 40 °C respectively. The range of pH and temperature in this experiment were selected with the objective to imitate realistic condition of a conventional POME treatment facility. The stirring process was done for 1 h at a rotational speed of 300 rpm. The residue was then filtered and left to dry overnight. The dried sample was weighed by using analytical balance and the final mass was recorded. The

difference between the initial mass (200 mg) and the final mass gave the solubility in mg L^{-1} (Equation 1).

$$S = \left[\frac{m_f - m_i}{V_s} \right] \times 2$$

S = Solubility (mg L^{-1})

m_f = final mass

m_i = initial mass

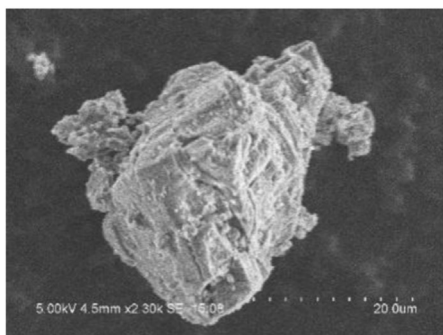
V_s = volume of solvent used (500 ml)

2.5. POME crystal growth

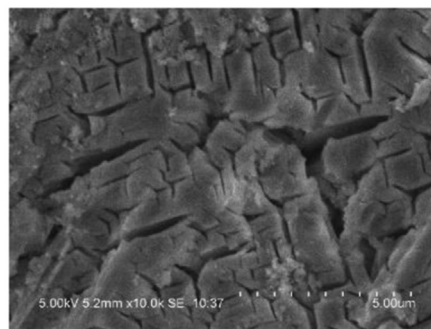
Crystal growth test was done using POME as the base medium to demonstrate the adaptation of reactive crystallization method to produce a controlled struvite precipitation from POME. Controlled struvite precipitation is a potential approach to prevent scattered formation of struvite crystal along the POME treatment process. It will concentrate the formation of struvite at only one point in the treatment system which will then leave the remaining POME with lesser concentration of struvite producing elements; phosphorous and magnesium.

A solution with magnesium chloride hexahydrate ($\text{MgCl}_2 \cdot 6\text{H}_2\text{O}$) and ammonium chloride (NH_4Cl) were prepared with molar concentration ratio of Mg^{2+} to NH_4^+ as 1:1 in a 0.5 L solution. The solution was then added to 200 ml of POME in a 1L beaker. The pH value for this sample was fixed to 10 by the addition of sodium hydroxide solution. The volume of sodium hydroxide solution added was controlled using a pH meter. The solution was mixed at 150 rpm for 30 min at the temperature of 30 °C. The sample was then left for 5 h. Struvite crystal formed in the solution was extracted after each hour interval. The extracted crystal was filtered and then dried in the oven at 103 °C for 24 h to remove any residual moisture. It was then weighed using analytical balance to record the mass of crystal produced by the experiment for each hour interval.

In order to get the percentage of nutrient removal from the solution, the solution was initially tested for its content of total phosphorus and magnesium using Hach DRB 200 and DR 2800 by Hach Company, United States of America. After completion of the experiment, the remaining solution was again tested for the phosphorus and magnesium concentration. The difference between both final and initial concentration of



(a)



(b)

Figure 4. SEM analysis of POME crystal (a) in a non-uniform manner (2,300x magnification), (b) with visible cracks on its surface (10,000x magnification).

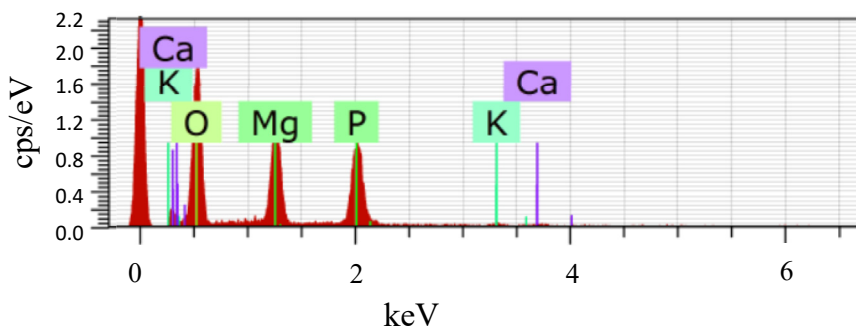


Figure 5. EDS spectrum generated from the analysis of POME crystal sample.

Table 2. Elemental contents of POME crystal.

Elements	Values, wt%
Phosphorous, P	24.85
Magnesium, Mg	21.33
Potassium, K	1.24
Calcium, Ca	1.05
Carbon, C	2.28
Hydrogen, H	3.53
Nitrogen, N	2.00
Sulphur, S	0.07
Oxygen, O	46.20

these two elements was recorded as the removal percentage for the process.

3. Results and discussion

3.1. Crystal morphology and elemental content

Under 2,300 times magnifications in the SEM analysis, a small portion of the sample presented itself in a non-uniform shape with slight glimpse of crystal edges (Figure 4a). Dendritic cracks on the crystal surface were visible with further magnification to 10,000 times (Figure 4b). This type of cracks was also visible in the struvite crystal from swine manure (Wu et al., 2018). Dendritic cracks on a crystal's surface can be stated as cracks with an arranged branching orientation giving indication of its lattice arrangement. Since the cracks covered a significantly wide area on the crystal surface, the crystal might have experienced stress due to heat exposure. This is possible since the crystals were left in the digester for some time (estimated 5 days) after all anaerobic liquor were drained from the digester tank before being removed. Density of the growth medium can also contribute to the cracks formation as higher density would lead to higher number of cracks (Salsabili et al., 2016). These cracks are believed to have lowered the strength of POME crystals to some extent, making mechanical removal of such stubborn crystals from the outlet pipe of anaerobic digester tank relatively easier. This implies that temperature has a significant influence towards the integrity of POME crystal and mechanical removal method of struvite crystal can be done easier after being exposed to a certain level of heat.

Based on EDS and CHNS analyses, the detected elements were phosphorus (P), magnesium (Mg), potassium (K), calcium (Ca), carbon (C), hydrogen (H), nitrogen (N), sulphur (S) and oxygen (O) (Figure 5). The major elements were O (46.20 wt%), P (24.85 wt%) and Mg (21.33 wt%). Both K and Ca were detected to be less than 2.00 wt%. The contents of C, H, N and S were all recorded below 4.00 wt% (Table 2). The presence of significant amount of P, Mg and O in the crystal sample indicates that the phosphate and magnesium ions in POME are consumed in the POME crystal formation process. Additionally, POME is known to

have high concentrations of phosphate and magnesium. Madaki and Seng in 2013 reported that these two elements (P and Mg) have the average concentrations of 180 mg L^{-1} and 650 mg L^{-1} respectively. It is also an early indication of struvite-laden content in the sample (Xie et al., 2014). A commercial struvite is known to have P and Mg composition percentage of up to 32.9% and 22.2% (Wu et al., 2018). Therefore, from the result, the crystal deposit sample can be seen as almost similar to struvite in terms of its composition.

3.2. Crystal properties

The XRD analysis showed the predominance of struvite mineral making up 94.8% of the sample composition (The International Center for Diffraction Data (ICDD) file no.: PDF 01-077-2303 $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$). Another 5.2% of the composition was analyzed as quartz (ICDD file no.: PDF 01-070-2536 SiO_2) (Figure 6). Peaks generated at two-theta angle of approximately 15, 21 and 31° from POME crystal sample have high similarity to the stick pattern of struvite in the XRD database as well as struvite from previous study (swine wastewater) done by Jordaan et al. (2010) (Figure 7). The composition of struvite mineral in the POME crystal sample is considered high for a naturally occurring struvite. In comparison, a controlled and experimentally grown struvite from other wastewater was reported to have struvite composition of up to 100% (Tadza et al., 2015). The struvite mineral found in the POME crystal sample is confirmed to have orthorhombic crystal structure with a space group of Pmn21. The density of the crystal were recorded at average of 1.701 g cm^{-3} .

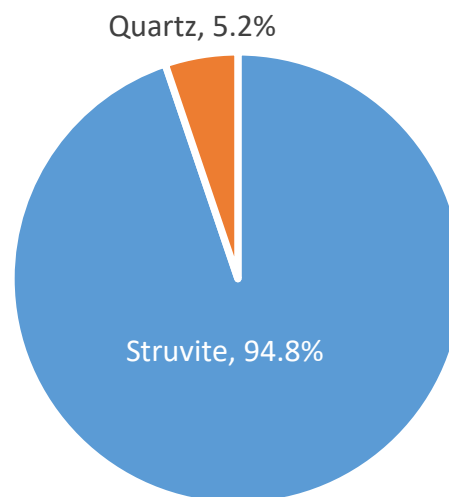


Figure 6. Composition of struvite as analyzed by x-ray diffraction.

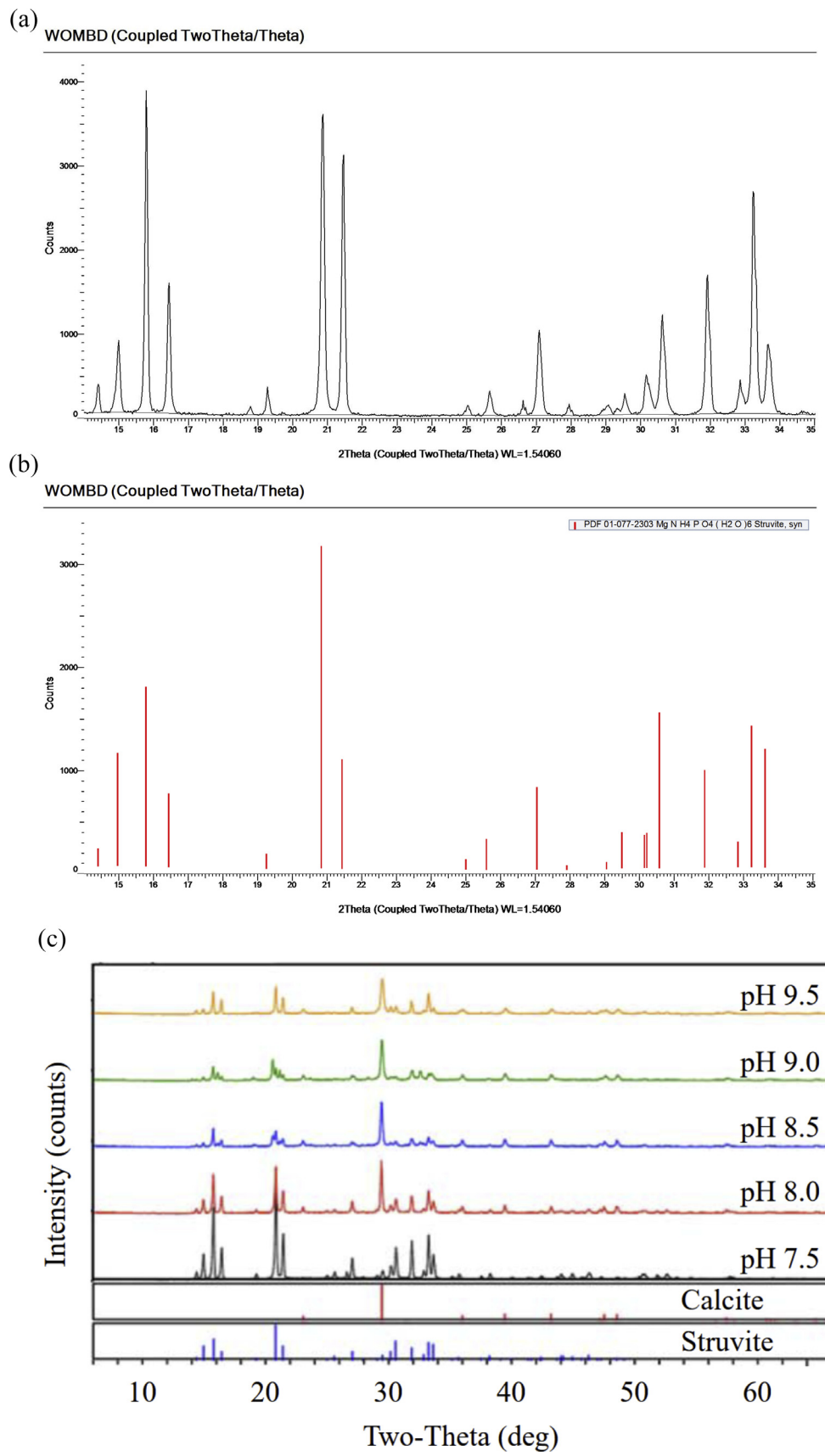


Figure 7. 2-theta XRD graph of (a) POME sample (b) struvite from the ICDD database (PDF 01-077-2303) and (c) struvite from work done by Jordaan et al. (2010).

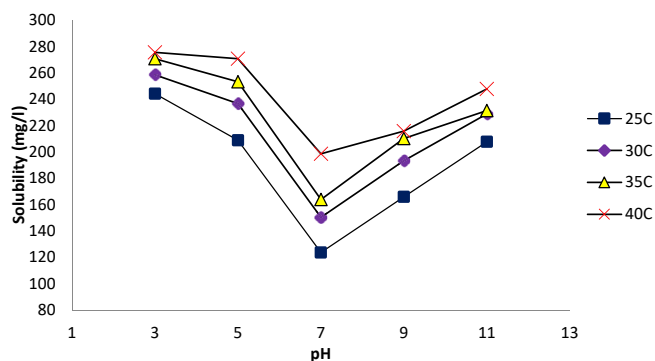


Figure 8. Solubility curve for struvite generated from POME.

Table 3. Solubility result of POME crystal under the influence of temperature and pH.

Temperature (°C)	pH	Solubility (mg/L)
25	3	244.2
25	5	208.8
25	7	123.6
25	9	165.8
25	11	207.6
30	3	258.6
30	5	236.7
30	7	150.4
30	9	193.2
30	11	228.8
35	3	270.8
35	5	253.2
35	7	163.8
35	9	210.2
35	11	231.4
40	3	275.6
40	5	270.8
40	7	198.6
40	9	215.8
40	11	247.8

3.3. Solubility curve

A solubility curve (Figure 8) was established from the solubility test on POME crystal. Solubility results for each combination of pH and temperature exposed to the POME crystal are in Table 3. A combined curve was used in plotting the solubility curve. From the solubility result, it was observed that the sample was more soluble in acidic conditions as well as in strong alkaline solutions. Increase in temperature also affected the solubility proportionately. On the other hand, lowest solubility was

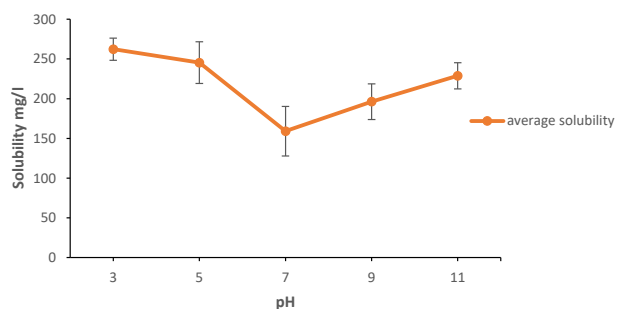


Figure 9. The average solubility curve of struvite against pH value across different temperature.

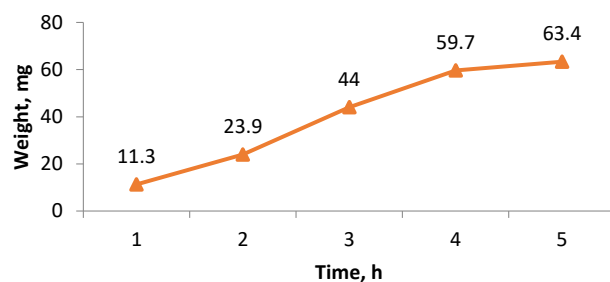


Figure 10. Steady growth of crystal precipitated from POME was recorded.



Figure 11. Filtered crystals precipitated by reactive crystallization.

observed at the point where the pH value was neutral and at lowest temperature.

The average maximum solubility of 275.6 mg L^{-1} was achieved at pH 3 and temperature $40 \text{ }^\circ\text{C}$, a highly acidic condition and higher temperature setting. All the other temperature settings at pH 3 also recorded higher average solubility values than other pH settings. Average minimum solubility of 123.6 mg L^{-1} was recorded at pH 7 and temperature $25 \text{ }^\circ\text{C}$. Strong alkaline solutions had also increased the solubility of POME struvite crystal in which, at pH 11 (highest pH setting), all solubility units for all temperature settings recorded higher solubility. The results implied that structural integrity of struvite formed in POME pipelines can be compromised significantly under strong acidic conditions and by increasing the temperature of the medium.

Based on the definition of solubility by IUPAC (IUPAC, 2008), solubility curve can be described as a graphical representation of the changing in concentration of a given substance in a given solvent. From the experiment, solubility curve generated can be generalized by taking the average solubility across the range of temperature between $25 \text{ }^\circ\text{C}$ to

Table 4. Reactive crystallization done on POME showed significant reduction in concentration of struvite elements.

Parameters	Concentration (mg L ⁻¹)		Removal percentage (%)				
	Before	After	This work	Abel-Denee et al. (2018)	Cao et al. (2019)	Xu et al. (2016)	Wilsenach et al. (2007)
Total phosphorous	380	114	67	87	96.7	83	95
Magnesium	75	37	26	-	-	-	-

40 °C. Therefore, the various results showed in Figure 8 can be represented by a single curve of the POME crystal solubility trend against the increase of pH (Figure 9). This has simplified the visualization of the curve hence making it easier to identify different saturation zones on the graph.

3.4. POME crystal growth

Experiment on POME crystal growth showed that reactive crystallization method adapted was able to produce struvite crystal from POME. This is shown by the increase of precipitated crystal mass recorded during the test (Figure 10). However, the process can be further optimized in order to find maximum yield with minimum amount of reagents to be added. After 1 h of reaction time, 11.3 mg of crystal was precipitated in the solution. The precipitation continues and recorded increment in its wet mass with final reading of 63.4 mg at 5th hour. The growth curve showed that the precipitation slowed down after 5th hour. The crystal precipitated from the process was filtered and sludge attached to it was carefully removed (Figure 11). Elemental analysis was done on the remaining POME to determine the concentration of phosphorous and magnesium.

Concentration of total phosphorous was reduced to 114 mg L⁻¹ from initial value of 380 mg L⁻¹ (67% reduction) while magnesium content was reduced to 37 mg L⁻¹ from initial value of 75 mg L⁻¹ (26% reduction) (Table 4). Although it does not reach the reduction level as reported by previous researchers (Abel-Denee et al., 2018; Cao et al., 2019; Xu et al., 2016; Wilsenach et al., 2007), this result implied that the reactive crystallization works well with POME in reducing the elements that will subsequently form struvite naturally in the wastewater, thus reducing the magnitude of the problem of struvite formation in POME treatment pipelines. The experiment was done assuming the temperature and pH were constant at 30 °C and at pH of 10. Future works will explore different set of temperature and pH combination in order to obtain maximum phosphorous removal from POME using this method.

4. Conclusion

The study has confirmed that the crystal deposit found in anaerobic digester tank of a POME treatment facility in a palm oil mill as struvite mineral. The formation of POME struvite was attributed to the presence of Mg²⁺, NH₄⁺ and PO₄³⁻ in the POME. This is reflected in the elemental results of the sample where all these elements were found in the crystal sample. Solubility curve for POME struvite were established and can be utilized in future works as a reference. Crystal growth test that was done met the objective to demonstrate the adaptation of reactive crystallization method to produce a controlled struvite precipitation from POME. Although preliminary, the finding is substantial in providing data and insights into struvite precipitation from POME for the mitigation of natural occurrence of struvite formation in POME polishing plant pipelines. A typical palm oil mill (60 tonnes hr⁻¹) discharges as high as 800 m³ per day of POME which could possibly generate an excessive amount of POME struvite during treatment. Therefore, with the high percentage of phosphate element in POME struvite it can be seen as a potential alternative source of mineral rock phosphate that is widely applied and is currently depleting in agricultural industry. Further work needs to be carried out to extract this valuable element that is abundant in POME. By

doing so, it could also avoid harmful leaching of phosphorous and nitrogen to a nearby watercourse.

Declarations

Author contribution statement

MUZZAMMIL NGATIMAN: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

MOHAMMED SAEDI JAMI & MOHD RUSHDI ABU BAKAR: Conceived and designed the experiments; Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

VIJAYA SUBRAMANIAM & SOH KHEANG LOH: Performed the experiments; Contributed reagents, materials, analysis tools or data.

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Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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