

## Characterization of crystallized struvite on wastewater treatment equipment: Prospects for crystal fertilizer production

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### ABSTRACT

With over-mining of the natural rock-P, food production will plummet sooner than we envisage since they are essential in agro-industry but are non-renewable. Hence, phosphate fertilizers are going to be limited in future. Struvite is a crystalline mineral substance containing equimolar amount (1:1:1) of magnesium and ammonium phosphate(V) ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ), a good source of phosphorus and a slow-release fertilizer. In this study, a sample of the struvite formed in the wastewater treatment equipment of Kilang Kelapa Sawit, Sri Senggora (Palm Oil Mill at Pahang, Malaysia) was collected and characterized to determine its suitability for use as a slow-release fertilizer for agricultural purposes. The formation of struvite in the sewage pipes of wastewater treatment facility causes bottlenecks in the operation of the plant and results in reduced pumping efficiencies and high cost of overall plant maintenance. Due to the P, N and Mg content in the palm oil wastewater streams, struvite formation is triggered, and the treatment equipment are clogged as the struvite precipitates and builds up. The results of the characterization of the struvite through SEM-EDX, FTIR, XRD and TG-DTA/DSC analyses give the morphology and atomic percentage of the different elemental composition, the absorption pattern of the different functional groups, the orthorhombic crystal structure arrangement and the loss of mass against temperature respectively. The results indicate that high quality and large quantity struvite can be recovered from the palm oil wastewater streams. The recovery of struvite will reduce the BOD and COD of the wastewater stream resulting in plant size reduction, small land space requirements and reduced cost. This phenomenon being eco-recycling of phosphorus will serve as a sustainable approach towards food security and can help mitigate the problem of eutrophication.

*Keywords:* Struvite; Phosphorus; eco-recycling; crystal fertilizer; POME; Renewable

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## 1. Introduction

Struvite is a crystalline mineral substance with equal molar concentrations of magnesium and ammonium phosphate(V) combined with six water molecules ( $\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$ ). It is a good source of phosphorus and a slow-release fertilizer. It is a double salt of different solubilities depending on the process conditions [1]. MAP can be recovered from organic wastewater [2], swine wastewater [3–6], industrial wastewater [7], municipal wastewater [8], municipal landfill leachate [9], leather tanning wastewater [10], dairy wastewater [11], wasted sludge [12], digested supernatant [13], palm oil wastewater [14], anaerobic digester effluents [15] and poultry manure wastewater [16]. Struvite has a commercial value, either as a pure high-phosphorus fertilizer; alternative to rock-P or as a supplement to compost or other fertilizers [17]. MAP has qualities superior to standard fertilizers [18]. Over time, much of the concern with struvite precipitation has been in determining how to avoid struvite scale from forming in the piping and equipment of wastewater treatment plants and agricultural waste systems. This is because it causes bottlenecks in the treatment equipment resulting in high running cost.

As concern grows over the management of nutrients in wastewater and manure especially phosphorus, research on and application of controlled struvite precipitation has increased around the world. The first countries where a significant amount of research on struvite precipitation has been completed are the Netherlands, Australia, and Japan. Proprietary struvite recovery processes for municipal and industrial waste waters have been developed both in the Netherlands and Japan. A non-proprietary struvite recovery process is treating veal manure at a full-scale plant in the Netherlands. Since natural phosphate is limited in supply, recovery of phosphorus from palm oil waste waters as struvite and recycling makes it not only sustainable but has a promising future for the fertilizer manufacturers.

The properties of struvite as a slow release fertilizer has also been demonstrated in the United States using hog manure. Application of MAP in the agricultural sector promises to be a profitable investment. It has been demonstrated that generating 1 kg of MAP per day is enough to fertilize 2.6 ha of arable land by applying phosphorus (as  $\text{P}_2\text{O}_5$ ) at a rate of 40 kg/ha y [19]. This helps to create an eco-friendly environment by reducing the need for the natural phosphorus (rock-P). The present consumption of rock-P is over one million tons per year, and utilization of MAP would be an effective alternative for rock-P in the fertilizer industry. Phosphorus recovery from palm oil wastewater streams through struvite synthesis is an important aspect of managing surface water quality and has a desirable fertilizer potential. This mitigates the problem of eutrophication occasioned by a significant amount of contaminants in the wastewater discharged to water bodies. Eutrophication is undesirable and hazardous to aquatic life and affects the quality of groundwater. Although there are impurities such as minerals and heavy metals in the palm oil mill effluent (POME), the concentrations of heavy metals are usually below the sub-lethal levels and are non-toxic to aquatic organisms and plants [20]. When the best conditions for struvite precipitation are determined, the removal process can be optimized. The prime factor in the struvite formation mechanism is the pH of the wastewater and usually in

the range of 7.0–10.5. For a full-scale design of wastewater treatment plant, this process makes smaller the treatment plant as the COD and BOD of the wastewater are reduced following P, Mg and N removal through MAP chemical precipitation as a pre-treatment technique. Phosphorus is an essential element of fertilizer without it crops wither. Like oil, rock phosphate is running out. Statistically, the United States which is the world's biggest producer of rock-P, is expected to exhaust its reserves in 25 years. This informed China's recent slap on export tariff of about 135 per cent on phosphate, choking off exports. This development leaves Morocco sitting on one-third of the world's remaining supply and their reserves are in fast decline.

The fear of exhausting the natural phosphate without plans of recycling has become a serious concern to fertilizer manufacturers globally. The recovery of struvite from palm oil wastewater to produce crystal fertilizer has a promise of solving the problem of shortage of natural phosphate since it can be recycled a number of times. Therefore, the applicability of struvite synthesis in the contaminants removal from palm oil wastewater and struvite suitability for use as eco-fertilizer have been investigated. Characterisation was done for the struvite precipitated on the palm oil wastewater treatment equipment at Pahang, Malaysia. With known composition of palm oil wastewater and an adequate knowledge of the struvite formation mechanism, a proper design of a reactor for maximum struvite recovery is made possible.

## 2. Materials and method

The sample was collected from the aeration pond of the palm oil wastewater treatment plant at Seri Senggora, Pahang, Malaysia. A section of the treatment equipment (piping system) was cut out to reveal the internal substance. The surface of the internal substance was thoroughly cleaned to ensure sample collection without contamination, and the precipitates formed in the internals of the pipe as clogs were scrapped out and crushed with mortar and pestle (Fig. 1). The powdered form of the sample was sieved to equal particle size for characterization (Fig. 2). The sample was then divided into three and each part was taken for SEM-EDX, TG-DTA, and XRD analyses. Fig. 3 shows the mixing pond, the biogas tanks and the ponding system of the plant while Fig. 4 shows the flowchart of effluent treatment system (200 MT/h). The specific values obtained from the analyses of the raw palm oil wastewater at Pahang, Malaysia for P,  $\text{NH}_3\text{-N}$ , Mg and pH (as a function of temperature) were 128 mg/L, 35.5 mg/L, 272 mg/L and 4.3 (at 40°C) respectively. Table 3 and Table 4 present the general characteristics of POME and the composition of minerals and heavy metals of POME respectively.

## 3. Results and discussion

### 3.1. XRD studies

This is primarily used for phase identification and it provides information on unit cell dimensions of the sample under investigation. The precipitates formed inside the oil palm wastewater treatment pipe was scrapped out and

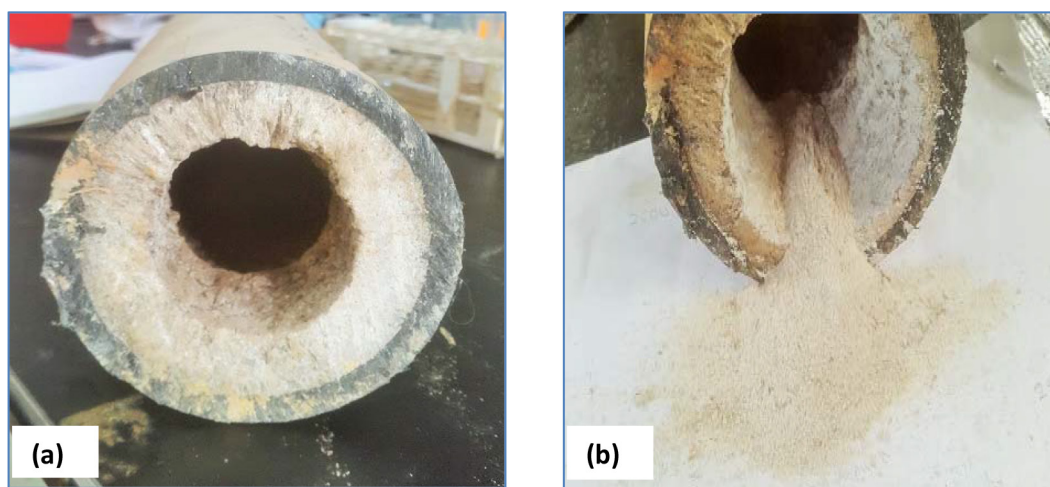


Fig. 1. (a) A section of the piping system (b) Struvite formed as clogs on the pipe being scrapped out.



Fig. 2. Crushed struvite recovered from the pipe.

grinded to powder form using mortar and pestle. The room temperature X-ray diffraction spectra of the powder struvite was recorded with a powder X-ray diffractometer (XRD 6000, Shimadzu, Japan) with Cu-K $\alpha$  radiation ( $\lambda = 0.15405$  nm). The quality of the struvite formed in the pipe is compared using the XRD patterns they produced by matching them with that of the standard struvite of International Centre for Diffraction Data (ICDD). A comparison of the intensities with a standard struvite showed a slight difference in the generated peaks (Fig. 5a and Fig. 5b). The slight difference is due to the trace number of other ions as impurities present in the compound and they interfered with the spectrum. In general, the XRD spectrum of the crystallised struvite matches that of a standard struvite indicative of a crystalline substance and showing the orthorhombic crystal structure arrangement. The spectrum aligns with the standard struvite ICDD Card No. 15-0762 used in the XRD studies of struvite produced from animal wastewater [21,22].

### 3.2. TG–DTA/DSC studies

The thermal decomposition study was carried out on the struvite using a TGA/DSC 1 HT (Mettler Toledo, Switzerland). The experiments were conducted under zero air

flow atmosphere ( $50 \text{ cm}^3/\text{min}$ ). The sample was heated in an open platinum crucible at a rate of  $1.0^\circ\text{C}/\text{min}$  beginning from  $15^\circ\text{C}$  up to  $600^\circ\text{C}$ . The initial weight of the struvite was measured and during thermal decomposition, the change in weight was recorded as a function of temperature and time. The mass loss of the struvite was observed at the temperature interval of  $68.27^\circ\text{C}$  and  $195.95^\circ\text{C}$  resulting in the decomposition of struvite as shown by the TGA curve (Fig. 6a). This is very close to the results gotten from the previous research study where the loss of mass of struvite takes place between  $57.8^\circ\text{C}$  and  $82.6^\circ\text{C}$  [1]. The moisture content at the first peak representing the temperature interval of  $68.27^\circ\text{C}$  and  $195.95^\circ\text{C}$  was 44.21%. Further loss of mass was observed at the temperature interval of  $196.13^\circ\text{C}$  and  $399.32^\circ\text{C}$  representing the second peak. No further significant loss of mass was observed above a temperature of  $399.32^\circ\text{C}$ . The results show that the precipitate formed in the palm oil wastewater treatment equipment is a crystalline substance. The thermograph shows a residue of about 45.82% representing the ash content. The DTA and DSC curves show the effect of the temperature on the physical and chemical properties of the struvite could be seen with a broad melting peak due to the size distribution of the crystallites with  $1,235.05 \text{ J g}^{-1}$  (Figs. 6b and 6c).

### 3.3. SEM-EDX studies

This was performed on the sample struvite crystals to determine the shape, morphology and individual elemental composition of the struvite using SEM (Hitachi S3400 N) that is equipped with EDX (Thermo Scientific Noran System Six) operated at 20 keV. The powdered form of struvite was deposited on a carbon tape and mounted on a sample holder and gold coated platform. The EDX spectra of the struvite from the palm oil wastewater treatment equipment were taken at different wavelength and showed the presence of irregular granular shaped particles (Figs. 8a and Fig. 8b). EDX shows the atomic percentage composition of individual element. The quantitative results are shown in Tables 1 and 2 with P 25.61% and P 32.24% for two different runs on the same sample. The



Fig. 3. (a) Mixing pond (b) Biogas tanks and (c) Ponding system.

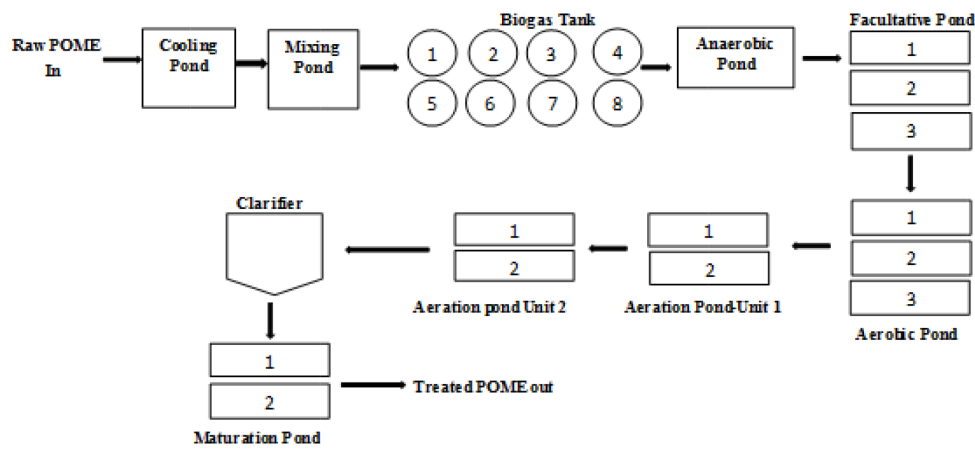


Fig. 4. Flowchart of 200 MT effluent treatment system per hour (Palm Oil Mill at Pahang, Malaysia).

Table 1  
Quantitative results for SEM-EDX analysis of struvite

Element line	Weight %	Atom %	Formula	Compound %
C K	21.47	32.29	C	21.47
Mg K	20.20	15.01	MgO	33.50
P K	25.61	14.93	P	25.61
K K	2.67	1.23	K <sub>2</sub> O	3.21

Table 2  
Quantitative results for SEM-EDX analysis of struvite

Element line	Weight %	Atom %	Formula	Compound %
C K	25.25	48.32	C	25.25
Mg K	23.14	18.38	MgO	38.37
P K	32.24	20.10	p	32.24
K K	3.44	1.70	K <sub>2</sub> O	4.14

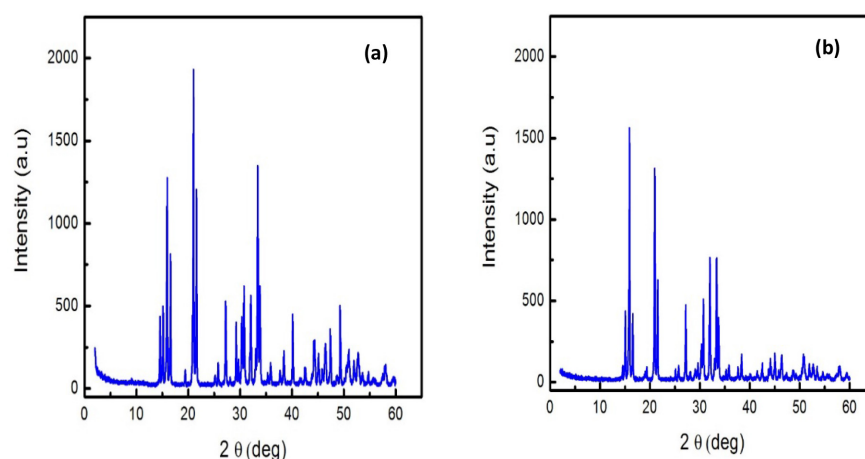


Fig. 5. (a) Powder XRD pattern for the struvite obtained from palm oil wastewater treatment equipment and (b) powder XRD pattern of a standard struvite.

high phosphorus content in the sample struvite is indicated by the highest peak in Figs. 7a and 7b. The struvite also contained potassium ion, magnesium ions, calcium ion, nitrogen and other minerals in trace amount as impurities. From the SEM-EDX results, the struvite formed in the palm oil wastewater treatment equipment could be used as NPK fertilizer considering the elemental composition and their atomic percentages (Table 1 and Table 2). The peaks in the XRD spectra of the crystallised struvite (Fig. 5) and the intensities in the EDX spectra show the elemental composition of the sample struvite with P and Mg having the highest peaks on both sides.

### 3.4. FTIR studies

A spectrophotometer; Perkin Elmer Spectrum 100 was used to determine the infrared spectra. In obtaining the spectra, 1% of the sample struvite is mixed and ground with 99% Potassium Bromide (KBr). The spectrum was taken in the range of 600–4,000  $\text{cm}^{-1}$  at 4  $\text{cm}^{-1}$  resolution. The absorbance spectra produced shows the molecular structure and the unique chemical bonds of the struvite. The absorption spectrum clearly enables for thorough investigation of struvite purity and any organic contaminants as shown in Fig. 9. From the FTIR absorption spectrum for Struvite formed in the palm oil wastewater treatment equipment, there is no clear difference in the absorption pattern as compared to that gotten from animal wastewater [7,22]. The different functional groups were represented indicative of high purity and conformed to the previous research study. The peak intensities are similar on both. An absorption peak seen at 3746.46  $\text{cm}^{-1}$ , 2945.10  $\text{cm}^{-1}$ , 1,738.40  $\text{cm}^{-1}$  and 994.09  $\text{cm}^{-1}$  were the strong and broad peaks due to the O–H stretching vibration motion, showing water of hydration. The weak bands seen between 3,899.18  $\text{cm}^{-1}$ , and 1,841.23  $\text{cm}^{-1}$  are basically due to water phosphate hydrogen bonding. The weak and broad peak at 1,642.02  $\text{cm}^{-1}$  and 1,372.32  $\text{cm}^{-1}$  are due to the H–O–H or H–N–H scissoring effect. The splitting of the peak may be due to the rotation of the ammonium ions. A low intensity but sharp peak is seen in both cases

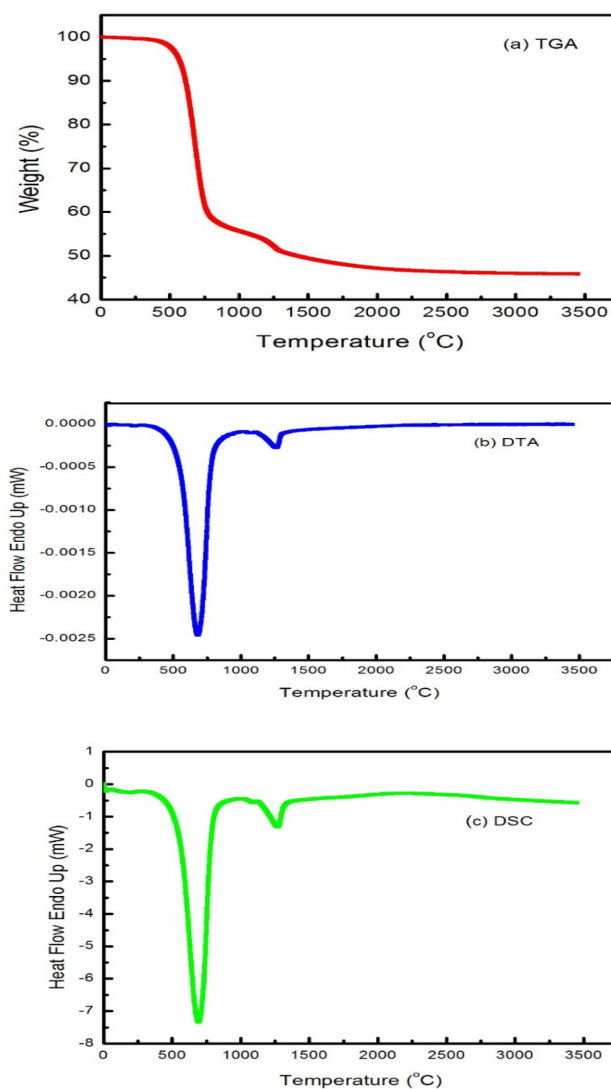


Fig. 6. TGA, DTA and DSC curves of the struvite formed in the pipe.

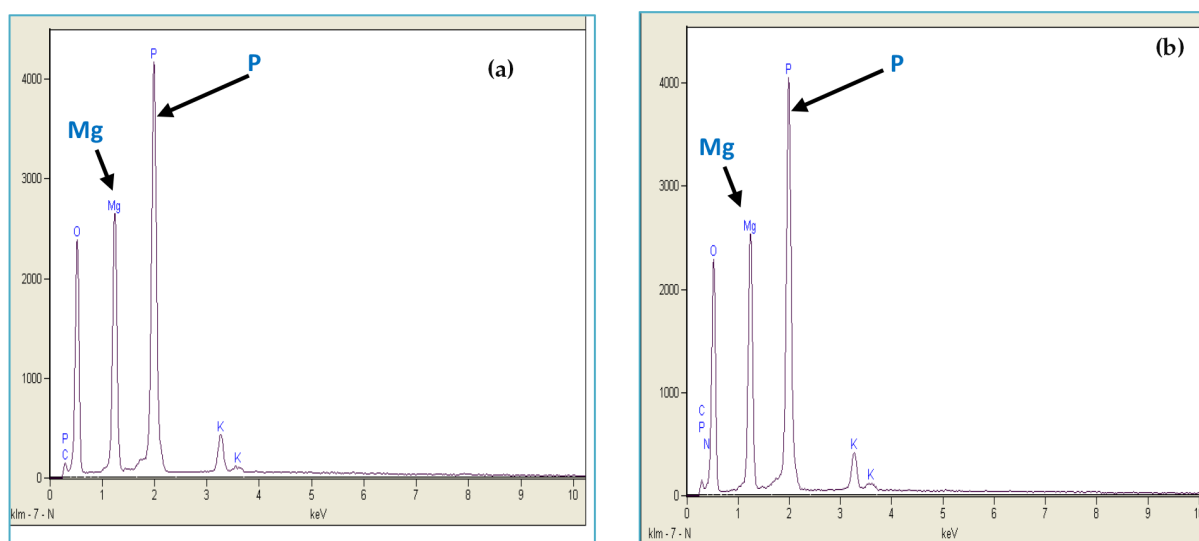


Fig. 7. EDX spectrum of the struvite formed in the palm oil wastewater treatment equipment.

Table 3  
Characteristics of POME  
Source: MPOB [23]

Parameter	Average	Range
pH	4.2	3.4–5.2
Oil and grease	4000	–
Biochemical Oxygen Demand (BOD)	25,000	10,250–43,750
Chemical Oxygen Demand (COD)	51,000	15,000–100,000
Total solid	40,000	11,500–79,000
Suspended solid	18,000	5000–54,000
Total volatile solid	34,000	9000–72,000
Ammoniacal nitrogen (NH <sub>3</sub> -N)	35	4–80
Total nitrogen	750	180–1400

All values are in mg/L except pH

Table 4  
Composition of minerals and heavy metals of POME  
Source:[20]

Minerals	Composition	Heavy metals	Composition
P	143777.38	Fe	11.08
Ca	1650.09	Mn	38.81
Mg	911.95	Zn	17.58
K	8951.55	Cr	5.02
Al	16.60	Cd	0.44
		Pb	5.15
		Cu	10.76

All values are in µg/g dry weight

at 2,945.10 cm<sup>-1</sup> mainly due to the ionic phosphate. Weak peaks at 891.28 cm<sup>-1</sup> and 759.77 cm<sup>-1</sup> are due to the metal-oxygen bond. The FTIR analysis shows presence of water of hydration, N-H bond, P-O bond, metal-oxygen bond, NH<sub>4</sub><sup>+</sup> ion, and PO<sub>4</sub><sup>-</sup> ion.

#### 4. Conclusion

Palm oil wastewater streams contain a good amount of phosphorus and magnesium, reasonable amount of nitrogen and through the struvite synthesis as a pre-treatment to the wastewater these nutrients can be recovered. Struvite as a good source of phosphorus can serve as eco-fertilizer for agricultural purposes. The recovery of struvite from palm oil wastewater stream can reduce the BOD and COD strengths of the effluent which consequently reduces cost due to reduction in the size of the equipment and land space requirements. Also, the pumping efficiency of the plant's pumps would be enhanced since the bottlenecks occasioned by the pipe blockage by the struvite as clogs would have been removed. The concentrations of the impurities such as heavy metals in POME are known to be usually below the sub-lethal levels and thus will have no harmful effects on flora and fauna. Recovering struvite from POME will be a sustainable approach towards nutrients recycling for agro-business and can mitigate eutrophication. It is claimed that the proceeds gotten from the sale of the struvite to fertilizer companies can cater for the overhead and running cost of the treatment plant. The quantitative details of the SEM-EDX in two different runs for the sample struvite formed in the wastewater treatment equipment showed about 25.61% and 32.24% of P respectively. With a controlled system of struvite synthesis in place a high quantity and high quality struvite as well as high recovery efficiency is made possible. The SEM-EDX, FTIR, XRD and TG-DTA/DSC analyses give the morphology and atomic percentage of the different elemental composition, the orthorhombic crystal structure arrangement and the loss of mass against temperature respectively. Even though other sources of phosphate include landfill leachate, industrial wastewater, poultry manure, animal wastewater, waste sludge from domestic and industrial wastewater treatment plant; palm oil wastewater has proved to be a good and potential source of crystal fertilizer which is eco-friendly and renewable. With the high phosphorus, reason-

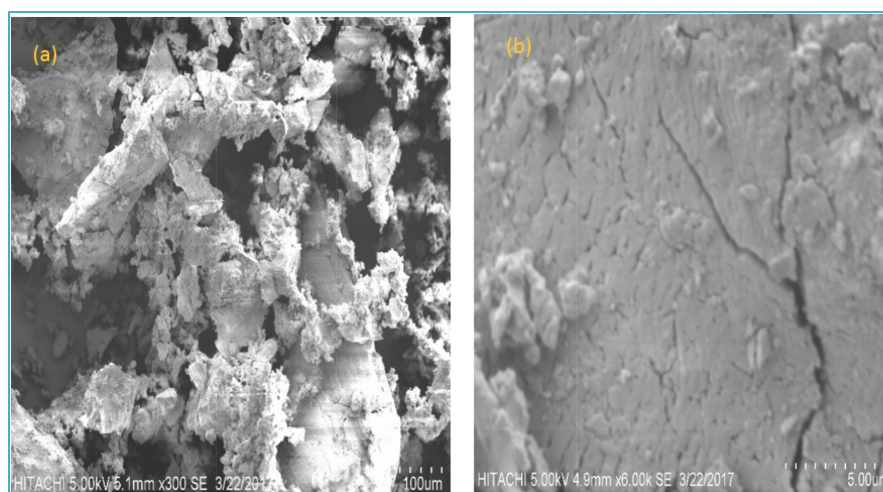


Fig. 8. SEM images of struvite formed on the palm oil wastewater treatment equipment. (a) Irregular crystal structure (b) Smooth structure indicating a homogeneous crystal distribution.

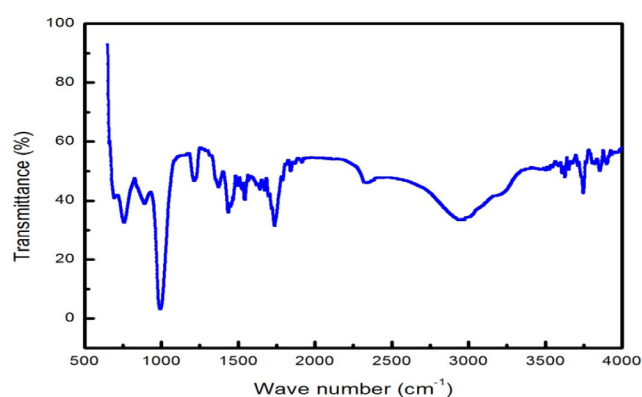


Fig. 9. Infrared Spectrum of struvite formed in the palm oil wastewater treatment plant showing different functional groups of the mineral.

able magnesium, nitrogen and potassium content in the palm oil wastewater streams for the production of struvite, it becomes imperative to encourage more research on struvite production from palm oil wastewater. This is to ensure food security in the future while mitigating water pollution.

#### Abbreviations

BOD	– Biochemical oxygen demand
COD	– Chemical oxygen demand
FTIR	– Fourier transform infrared analysis
MAP	– Magnesium and ammonium phosphate(V)
MPOB	– Malaysian palm oil board
MT	– Metric tons
POME	– Palm oil mill effluent
SEM-EDX	– Scanning electron microscopy and energy dispersive X-ray analysis
TG-DTA	– Thermogravimetric and differential thermal analysis
XRD	– X-ray diffraction

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