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Phosphorus recovery from the sludge generated from a continuous bipolar mode electrocoagulation (CBME) system

M. Damaraju, H. Yoshihara, D. Bhattacharyya, T. K. Panda and K. K. Kurilla

ABSTRACT

Phosphorus is known to be a limited non-renewable resource. Phosphorus is obtained from phosphate rock, which is likely to be depleted in the next few decades. Therefore, it is very important to find alternate sources of phosphorus from which phosphorus can be recycled and recovered. This study focuses on the recovery of phosphorus from the sludge generated from a Continuous Bipolar Mode Electrocoagulation (CBME) system, used for treating a Palm Oil Mill Effluent (POME). The sludge generated from the CBME system is leached with oxalic acid and sulphuric acid for phosphorus recovery with and without thermal treatment. Acid leaching was carried out at various time intervals using various liquid/solid (L/S) ratios of acids and sludge. CBME system caused a 73% removal of phosphorus from POME, where phosphorus is precipitated in sludge as iron phosphates or adsorbed as phosphates depending on the pH in the system. Acid leaching resulted in nearly 85% recovery of phosphorus with both sulphuric acid and oxalic acid for sludge combusted at 900 °C. Statistical analysis was carried out to find out the significance of the operational conditions on the phosphorous yield. Acid leaching results in the formation of orthophosphates, which can be used as a raw material for synthesis of chemical fertilizers.

Key words | bipolar mode electrocoagulation, phosphorus recovery, phosphorus removal

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INTRODUCTION

Phosphorus is an essential element for the growth of organisms and also has wide applications as fertilizer. Phosphorus is known to be a limited non-renewable resource. Phosphorus is obtained from phosphate rock, which is likely to be depleted in the next few decades. Therefore, it is very important to find alternate sources of phosphorus from which phosphorus can be recycled and recovered (Biswas *et al.* 2009). Phosphorus can be recovered from phosphorus-rich sources like manure, agricultural wastes, sewage sludge, etc. The disposal of domestic and industrial wastewaters containing phosphorus without proper treatment leads to eutrophication (Li *et al.* 2019).

Industrial wastewaters, like those generated in agro-based industries, contain a significant amount of phosphorus. Palm oil mill effluent (POME)is one such source rich in phosphorus. POME is generated in high volumes in palm oil processing industry. Treatment of POME can lead to the generation of phosphorus-rich sludge (Madaki & Lau 2013). The biological treatment is usually employed for the removal of pollutants from POME. However, there is a need for a technology that can polish the biologically-treated effluent and recover phosphorus from wastewater. Electrochemical treatments have been studied for the removal of phosphorus from various wastewaters (Daghrir *et al.* 2016). Electrochemical treatment leads to precipitation of phosphorus in sludge, making the phosphorus recovery easier.

The recovery of phosphorus from sewage sludge, which contains 1–5% of phosphorus, has been extensively studied (Cieślik & Konieczka 2017). Crystallization and leaching are commonly used techniques for phosphorus recovery (Desmidt *et al.* 2015; Reuna & Väisänen 2018; Li *et al.* 2019). The fluidized homogenous crystallization of phosphorus resulted in 90% phosphorus granulation (Caddarao *et al.* 2018). Biological leaching methods were also applied for the recovery of phosphorus from sewage sludge (Mehta *et al.* 2015). Precipitation through hydroxyapatite was studied by some of the authors (Song *et al.* 2002; Lee *et al.* 2018). Acid leaching and base leaching techniques were used for

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the recovery of phosphorus from sludge generated from biological treatment methods (Biswas *et al.* 2009; Lee *et al.* 2018). Leaching methods are found to be an inexpensive technique among other recovery methods. The sludge combustion to ash leads to more availability of phosphorus for recovery. Recovery of phosphates from ash has been tried by various authors (Schaum *et al.* 2007; Adam *et al.* 2009; Atienza-Martínez *et al.* 2014). Phosphorus recovery was found to be 85% using sulphuric acid for the sewage sludge ash containing 6–10% of phosphorus (Cohen 2009). Phosphorus recovery from dried sludge without incineration using sulphuric acid was found to be 55% (Lee *et al.* 2018). Phosphorus recovery studies using organic acids were found to be successful (Sagoe *et al.* 1998; Kpomblekou-A & Tabatabai 2003). The sewage sludge incineration is commonly found a technique for recovery of phosphorus, nowadays. Fast pyrolysis methods have been developed to address the drawbacks of incineration (Atienza-Martínez *et al.* 2014). However, combustion (heating in the presence of oxygen) of sludge, which results in gaseous and solid compounds, is a comparatively inexpensive technique for phosphorus recovery (Atienza-Martínez *et al.* 2014).

The novel continuous flow bipolar mode electrocoagulation (CBME) reactor has been developed in our laboratory. The reactor was used for the removal of phosphorus from a Palm Oil Mill Effluent. The new system facilitates a higher removal of pollutants (phosphorous) with operational ease and lower costs compared to the conventional monopolar mode electrochemical reactors. Phosphorus was observed to be precipitated out from the wastewater as iron phosphate. This study describes the recovery of phosphorus from the POME sludge that was generated in the system. The study focused on evaluating the efficacy of conventional acid leaching methods in phosphorus recovery from combusted and un-combusted POME sludge. Sulphuric acid and oxalic acids were used at different liquid-to-solid ratios for extraction of phosphorus. Effect of contact time on leaching was also studied. Statistical significance test was performed to evaluate the significance of each parameter on phosphorus recovery.

METHODS

Sampling of wastewater

A biologically treated POME was used in this study. The wastewater was sampled at the outlet of an Effluent treatment plant (ETP) of a palm oil mill industry located in Andhra Pradesh, India. The ETP has adopted an anaerobic process for treating the POME. The sample collected was stored at 4 °C until use.

Experimental set-up and procedure

A continuous bipolar mode electrocoagulation (CBME) reactor was used for treating the POME. The CBME set-up is shown in Figure 1. The CBME was fabricated using a poly-acrylic sheet. The reactor had an effective volume of 765ml. The electrode dimensions were 15cm × 3.4cm. A total of 15 electrodes were used. The two end electrodes were connected to a power supply. The remaining electrodes were not connected to a power supply and used as sacrificial electrodes. Iron electrodes were used for the

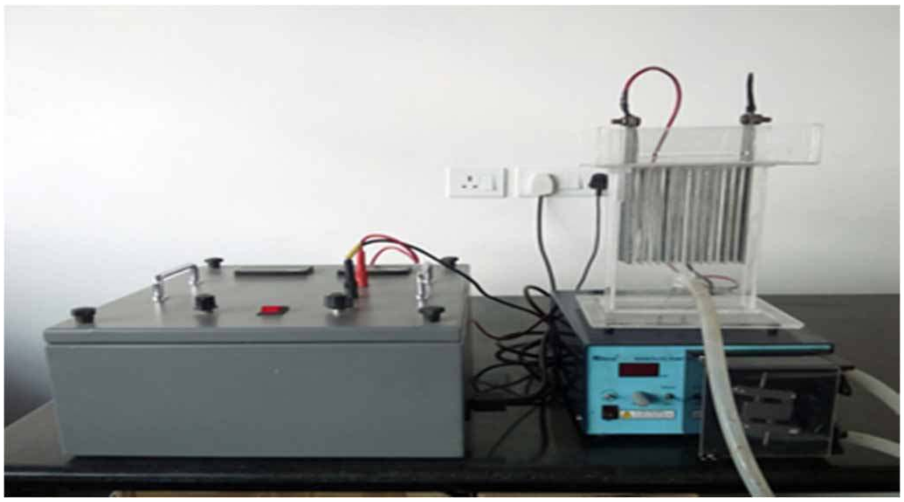

Figure 1 | Electrocoagulation set-up (1. DC power supply 2. CBME chamber 3. Peristaltic pump).

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study. The power supply pack had an input voltage of 250 V and a variable output of 0–220 V. The POME enters the reactor from the bottom and moves to the top. Central Composite Design (CCD) was used to design the experimental runs and Response Surface Methodology was used to optimize the operating parameter (pH, current density, reaction area and reaction time) for maximizing the removal of phosphorus from wastewater. The details of the process optimization have been explained elsewhere (manuscript under review). This paper describes the phosphorus recovery study (from sludge) that was conducted under an optimized condition of the CBME system.

The sludge generated from the CBME system under optimum condition was used as raw material for the recovery of phosphorus. The sludge was collected and dried in an oven at 105 °C for 24 hours. The sludge was then combusted at 600 °C, 750 °C and 900 °C in a muffle furnace. These three different combustion temperatures were tested to study the effect of temperature on phosphorus recovery from ash generated from POME. The lower and upper limits of combustion were selected based on the minimum combustion temperature and melting points of ash, respectively (Atienza-Martínez *et al.* 2014). Acid leaching tests were carried out with sulphuric acid (0.5 M) and oxalic acid (0.5 M) solutions at room temperature under continuous stirring. The liquid to solid ratios used were 50ml/g, 150ml/g and 200ml/g. The samples were collected at fixed time intervals of 2h, 4h, 8h, and 24h. Experiments were carried out in duplicates. Phosphorus yield in the leachate was calculated using Equation (1) (Atienza-Martínez *et al.* 2014). Figure 2 represents the flow chart of the methodology.

$$\text{P yield } (\%) = \frac{mass\ of\ P\ in\ leachte}{mass\ of\ P\ in\ ash} * 100 \quad (1)$$

Analysis methods

The phosphorus in the liquid was analyzed using *Standard Methods for the Examination of Water and Wastewater* (*Standard Methods*, APHA/AWWA/WEF 2012). Proximate analysis was carried out using ASTM standards (Vamvuka *et al.* 2009). The elemental analysis was carried out in a Euro Victor EA3000 model CHNS-O instrument to estimate the percentages of C, H, N, S, and O in the sludge. The samples were analyzed for mineral composition with an energy dispersive X-ray system (EDS) (Model: Zeiss FESEM Supra 40). The sludge was characterized using X-ray diffraction (XRD) (Make: PAN Analytical). XRD was performed with copper anticathode. The diffractograms were interpreted using Xpert data collector software for phase identification. The parameters were measured with 4 s counting time per 0.0167°2θ step.

Statistical analysis

The data obtained were statistically analyzed using analysis of variance (One-way ANOVA) with a confidence interval of 95%. The effect of contact time, combustion temperature, L/S ratio for each acid type on the phosphorus yield was evaluated based on the statistics. The significance F-test with ANOVA revealed the significance of each operating condition on the P yield (p value < 0.05).

RESULTS AND DISCUSSIONS

Wastewater treatment

The wastewater was treated in a CBME system which has multiple electrodes, with a surface area to volume ratio of

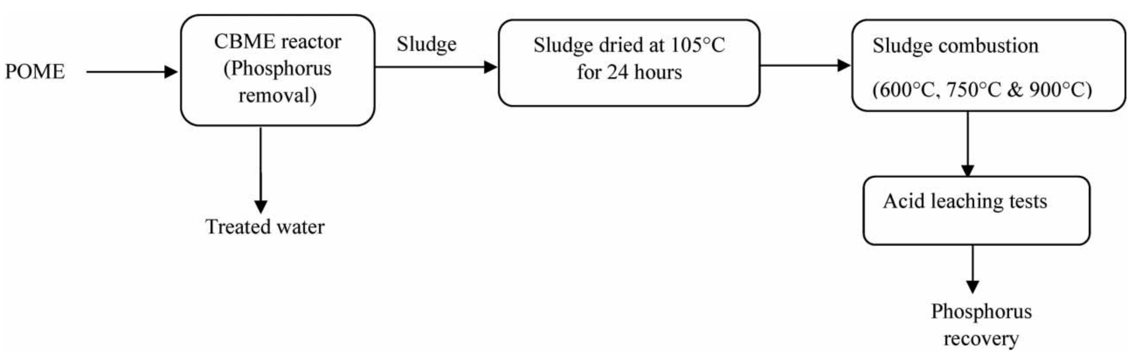

Figure 2 | Flow sheet of phosphorus recovery.

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76.5 m^{-1}. This reactor provides more surface area for the reaction, thereby, increasing the removal efficiency. The process optimization of the CBME system was carried out to maximize the removal efficiency of phosphorus from the POME. The CBME system resulted in optimized operating conditions at pH-6.4, reaction area –765 sq cm, reaction time-7.69 min and current density-77.5 A/sqm. The initial and final phosphorus concentrations were evaluated. The removal efficiency of phosphorus was found to be 73%. The phosphorus removal mechanism from the CBME reactor includes adsorption and chemical precipitation. Iron electrodes were used for the study, which leads to the formation of insoluble iron phosphate ($FePO_4$) or free phosphates in the sludge (Sincero & Sincero 2003).

Sludge characterization

The sludge, obtained from the CBME system, was oven dried and sieved. The sludge characterization was carried out. The properties of dried sludge are shown in Table 1.The mineral composition revealed that the phosphorus in the sludge was around 11.8%, unlike the sewage sludge which has 2–4% by weight depending on the source of wastewater (Günther 1997).The higher percentage of phosphorus in the sludge will facilitate more phosphorus recovery. The sludge generated from the CBME reactor was found to be rich in iron (43.07%). The phosphorus was found to be bound with iron as iron phosphates. Iron phosphates are highly insoluble with a K_{sp}, value of 10^{-36}. The phosphorus has to be released from the iron complex to be available for effective leaching. Oxidation reaction enhances the phosphorus release from iron. Combustion of sludge was performed to produce more soluble phosphorus in the sludge. Combustion at higher temperatures leads to the formation of acid insoluble iron compounds like oxides (FeO) and acid-soluble phosphorus from iron phosphates (Beck *et al.* 2004).Temperatures beyond 800 °C convert phosphorus to volatile oxides, which on condensation (between 400 °C and 600 °C) form phosphorus pentoxides (Cieślik & Konieczka 2017). The X-ray diffractograms of the sludge combusted at 600 °C, 750 °C,and 900 °C are shown in Figure 3. The sludge characterization study revealed the formation of haematite (Fe_2O_3) when the combustion temperature increased. The sludge contains phosphoric acid (A-JCPDS-030201), iron oxides or haematite (B-JCPDS-330664), iron phosphates(C-JCPDS-180649) and phosphorus pentoxide (D-JCPDS-050318). Figure 3(a) represents oven dried sludge, which contains all the four compounds, however, dominated by the presence of iron phosphates. Figure 3(b) represents the sludge combusted at 600 °C. The iron oxides formation was slightly increased, which is represented by B. An additional peak for phosphorus pentoxide (D) was observed and iron phosphate (C) peak intensity was found to decrease. Figure 3(c) represents the sludge combusted at 750 °C. The iron oxide formation was found to be more when compared to sludge combusted at 600 °C. Iron phosphate peak intensity was found to be further decreased. Figure 3(d) represents the sludge combusted at 900 °C. A large number of peaks were observed for iron oxide, which indicates the increase in the formation of insoluble iron when compared to the sludge combusted at 600 °C & 750 °C. The formation of phosphorus pentoxides is also evident at this temperature. Therefore, it can be concluded that as the combustion temperature increases the formation of acid-soluble phosphorus also increases in the sludge ash. The insoluble iron present in the sludge prevents its leaching into the solution.

Table 1 | Properties of the CBME sludge

Property	Units	Analytical standard	Dried sludge
Proximate analysis			
Ash	%	ASTM D3172-89	36.2
Volatile matter	%	ASTM D3172-89	50.1
Total solids	gm/l	*Standard Methods* (APHA/AWWA/WEF 2012)	22.5
Elemental Analysis			
Carbon	%	CHNS analyzer	15.44
Hydrogen	%	CHNS analyzer	2.448
Nitrogen	%	CHNS analyzer	–
Sulphur	%	CHNS analyzer	0.957
Oxygen	%	EDS analysis	13.8
Mineral composition			
Phosphorus	%	EDS analysis	**11.8**
Silicon	%	EDS analysis	2.12
Chlorides	%	EDS analysis	35.6
Calcium	%	EDS analysis	8.39
Iron	%	EDS analysis	43.07

Acid leaching tests

The acid leaching was carried out using an organic acid (oxalic acid) and inorganic acid (sulphuric acid). The sulphuric acid is a strong acid, which has high proton

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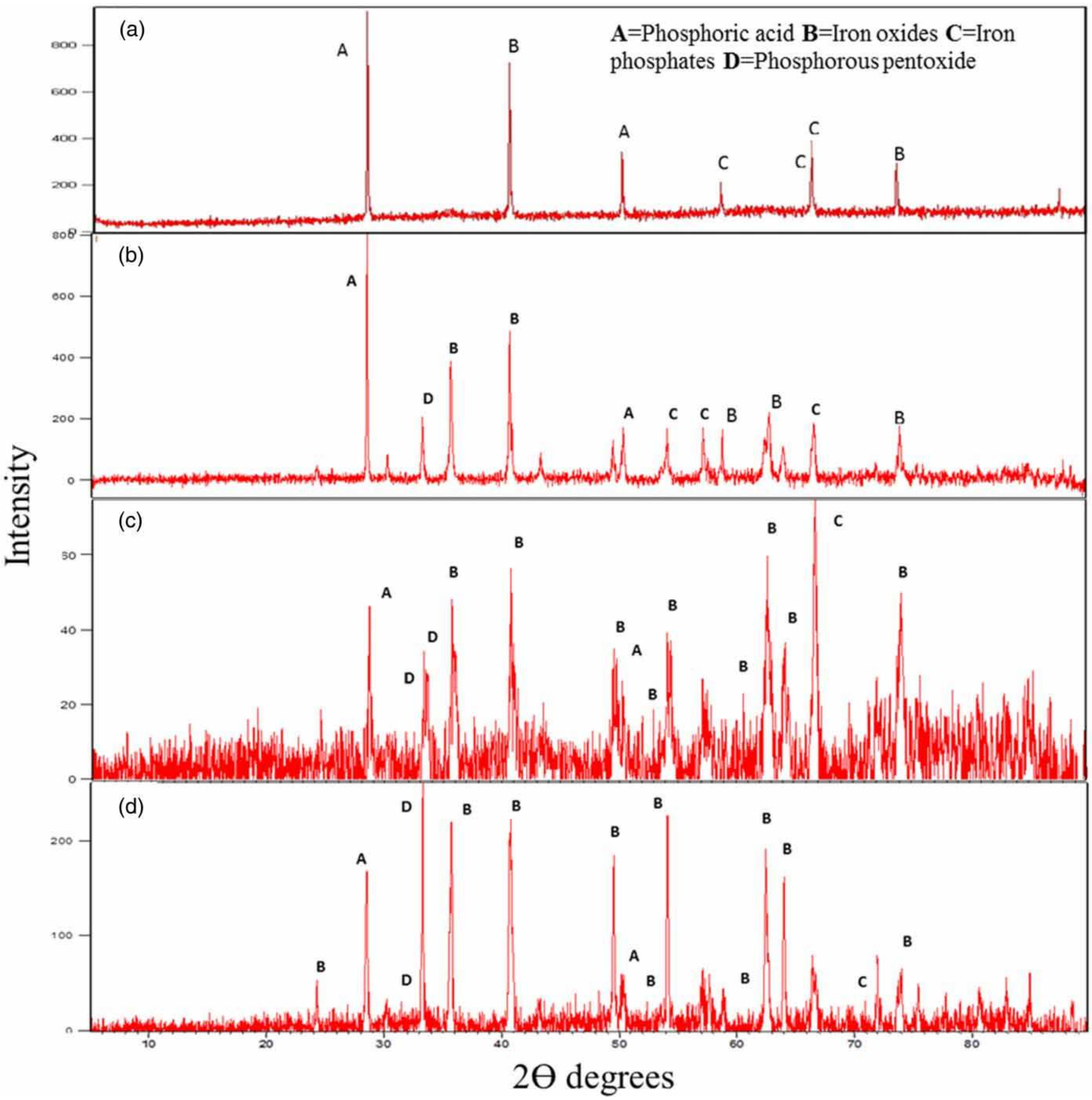

Figure 3 | Sludge (a) Oven dried (b) Combusted at 600 °C (c) Combusted at 750 °C and (d) Combusted at 900 °C.

donating ability and oxalic acid can form complexes with metal ions like iron which can prevent their leaching into acid (Atienza-Martínez *et al.* 2014). Liquid extraction may allow other inorganic compounds like potassium, calcium, magnesium, and sulphur also to be extracted along with phosphorus. The obtained liquid can be directly applied as fertilizers (Cieślik & Konieczka 2017). The phosphorus yield from oxalic acid leaching ranged from 25% to 83%. Phosphorus yield from sulphuric acid leaching ranged from 40% to 85%.

Effect of liquid-to-solid (L/S) ratio of acids

Leaching of phosphorus at various L/S ratios of oxalic acid is shown in Figure 4. Figure 4(a) and 4(b) represent phosphorus yield for oven dried sludge and sludge combusted at 600 °C, respectively. The highest phosphorus yield increased from 50% to 53% in oven-dried sludge and 55% to 65% in the sludge combusted at 600 °C. Figure 4(c) and 4(d) represent phosphorus yield for the sludge combusted at 750 °C & 900 °C, respectively. The highest phosphorus

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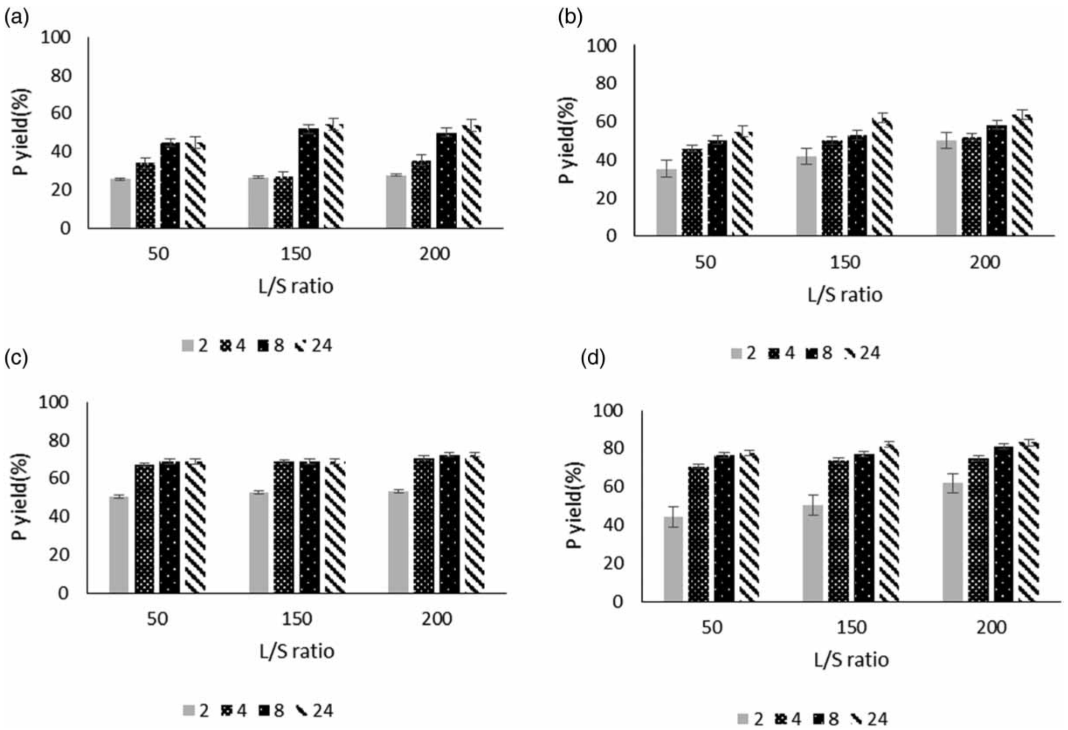

Figure 4 | Effect of liquid to solid ratio with oxalic acid on sludge (a) Oven dried (b) Combustion at 600 °C (c) Combustion at 750 °C and (d) Combustion at 900 °C.

yield was increased from 68% to 72% and 77% to 83%, for L/S ratio of 50–200 ml/g, in the sludge combusted at 750 °C & 900 °C, respectively. Leaching of phosphorus with various L/S ratios of sulphuric acid is shown in Figure 5. Figure 5(a) and 5(b) represent phosphorus yield for oven dried sludge and sludge combusted at 600 °C

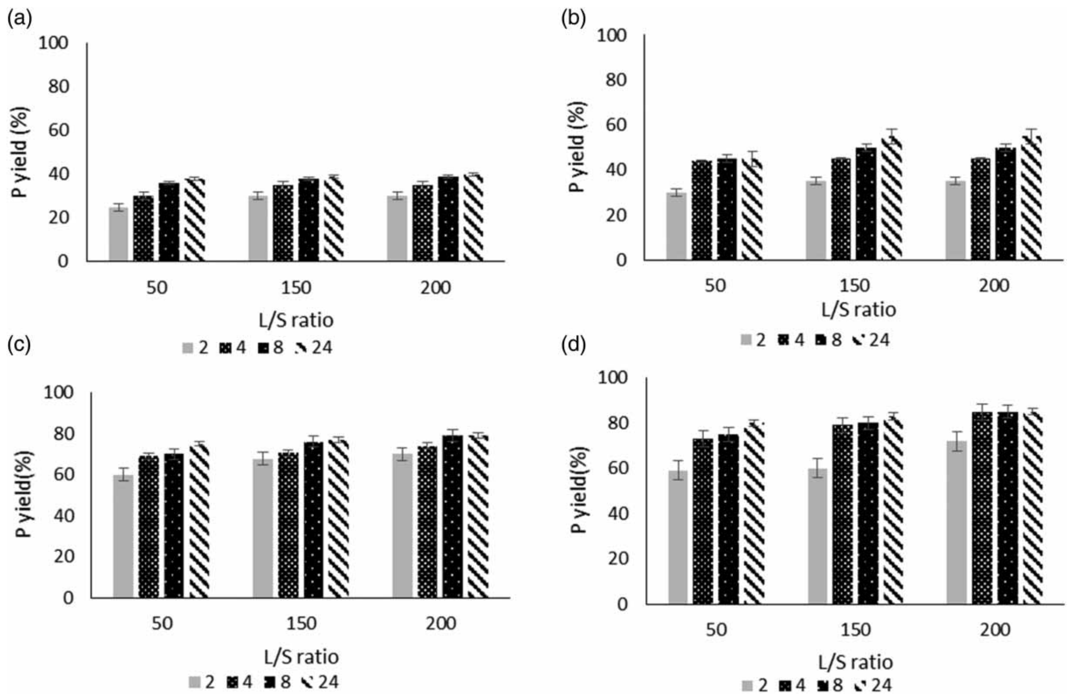

Figure 5 | Effect of liquid to solid ratio with sulphuric acid on sludge (a) Oven dried (b) Combustion at 600 °C (c) Combustion at 750 °C and (d) Combustion at 900 °C.

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respectively. The highest phosphorus yield increased from 38 to 40% in oven-dried sludge and 49% to 55% in the sludge combusted at 600 °C, with an increase in the L/S ratio. Figure 5(c) and 5(d) represent phosphorus yield for the sludge combusted at 750 °C & 900 °C, respectively. The highest phosphorus yield was increased from 75% to 79% and 80% to 85%, for L/S ratio of 50 to 200 ml/g, in the sludge combusted at 750 °C &900 °C respectively. The phosphorus yield was found to be increasing with an increase in the L/S ratio from 50 ml/g to 200 ml/g for oven dried sludge and sludge combusted at 600 °C. For the sludge combusted at 750 °C and 900 °C, the highest phosphorus yield was found to be increased to a lesser degree in both the cases. This phenomenon was due to more availability of soluble phosphorus at combusted sludge at high temperatures. L/S ratio influenced the phosphorus yield for the sludge combusted at lower temperatures than the sludge combusted at higher temperatures. Increase in L/S ratio increased the phosphorus yield in oven dried and sludge combusted at 600 °C. The optimum L/S ratio with sulphuric acid was observed to be 20 ml/g, where 90% of phosphorus yield was present for the sludge incinerated with a fluidized bed when phosphorus percentage was 5–7% in the sludge ash(Donatello *et al.* 2010). The optimal L/S ratio of sulphuric acid 150 ml/g, where phosphorus yield of 90% was achieved for incinerated sewage sludge (Biswas *et al.* 2009). The optimal L/S ratio of sulphuric acid and oxalic acid was found to be 150 ml/g, where phosphorus yield of 90% was achieved for char combustion ash of sewage sludge for a *P* value of 5–7% in the sludge (Atienza-Martínez *et al.* 2014). At combustion temperatures of 750 °C and 900 °C, the optimal L/S was found to be at 150 ml/g. The optimal L/S ratio for oven dried sludge and the sludge combusted at 600 °C temperatures were found to be 200 ml/g for both the acids. This study represents the phosphorus yield with oven dried sludge, and the sludge combusted using muffle furnace. The F-statistic test revealed the significance ($p < 0.05$) of L/S ratio on P yield at a contact time of 24 hours and combustion temperature of 900 °C for both sulphuric acid and oxalic acid. The phosphorus yield values obtained in this study were comparable with the literature.

Effect of time

Leaching phosphorus with acids was carried out at various time intervals for 2 hrs, 4 hrs, 8 hrs,and 24 hrs. Figure 4 representing the oxalic acid leaching, depict that the highest phosphorus yield was found to be 53% and 63% at 200 ml/g L/S ratio after 24 hours of leaching for oven dried and sludge combusted at 600 °C, respectively. However, for the sludge combusted at 750 °C & 900 °C highest phosphorus yield of 72% and 83%, respectively, was observed to be within 8 hours. Figure 5 representing the sulphuric acid leaching, depict that the highest phosphorus yield was found to be 40% and 55% at 24hours of leaching for oven dried and sludge combusted at 600 °C. However, for the sludge combusted at 750 °C & 900 °C highest phosphorus yield of 79% and 85%, respectively, was observed to be within 8 hrs time. Acid leaching of phosphorus was quicker for the sludge combusted at a higher temperature. However, for 2 hours of retention time minimum of 62% and 72% yield was found for both oxalic acid and sulphuric respectively, for the sludge combusted at 900 °C, at L/S ratio of 200 ml/g. The optimal time for maximum phosphorus yield was found to be 2 hours for the sludge incinerated with the fluidized bed when the phosphorus percentage was 5–7% in the sludge ash (Donatello *et al.* 2010). The optimal time of 4 hours was achieved for incinerated sewage sludge (Biswas *et al.* 2009). The optimal time for phosphorus yield of 90%, was found to be 8hours at all the combustion temperatures char combustion ash of sewage sludge for a *P* value of 5–7% in the sludge (Atienza-Martínez *et al.* 2014). The optimal time found in this study was 24 hours for the sludge combusted at 600 °C and oven dried sludge, 8 hours for sludge combusted at higher temperatures. However, sulphuric acid was more effective for a minimal contact time of 2 hours. The contact time was found to be a statistically significant ($p < 0.05$) parameter for P yield at 200 ml/g L/S ratio and at a combustion temperature of 900 °C.

Effect of combustion temperature

The effect of combustion temperature was studied for recovery of phosphorus. Figure 6(a) represents the highest phosphorus yields at 200 ml/g, L/S ratio for various combustion temperatures for oxalic acid. The phosphorus yield was found to increase with increasing combustion temperature. The minimal phosphorus yield of 55%, was found to be for oven dried sludge and the highest yield was found to be 83% at a combustion temperature of 900 °C. Figure 6(b) represents the highest phosphorus yields at 200 ml/g L/S ratio for various combustion temperatures for sulphuric acid. The phosphorus yield was found to be increased with an increase in combustion temperature. The minimal phosphorus yield of 40%, was found to be for oven dried sludge and the highest yield was found to be 85% at a

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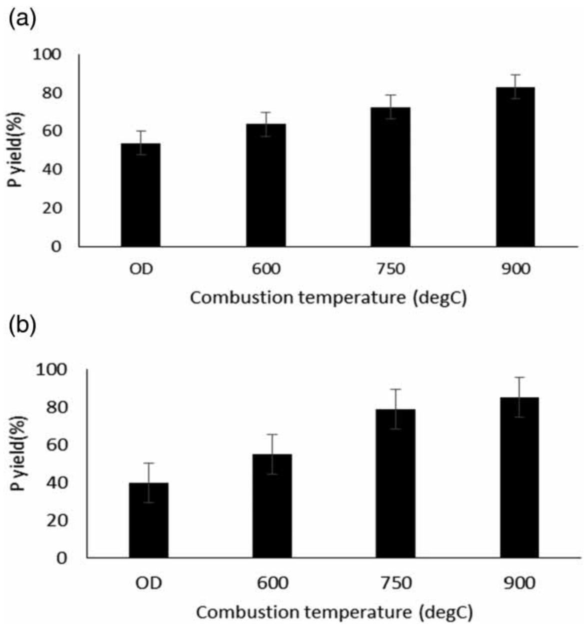

Figure 6 | Effect of combustion temperature (a) Oxalic acid (b) Sulphuric acid.

combustion temperature of 900 °C. However, the increase in temperature from 750 °C and 900 °C, could not show a significant effect with sulphuric acid. For oxalic acid, the temperature effect was prominent. Increase in combustion temperature resulted in high acid-soluble phosphorus. Phosphorus yield in sulphuric acid was found to be better at a combustion temperature of 750 °C and 900 °C. The combustion temperature has shown a significant effect ($p < 0.05$) on P yield at L/S ratio of 200 ml/g and at a contact time of 24 hours for oxalic acid and sulphuric acid.

This study shows that phosphorus can be recovered effectively from POME sludge using conventional acid leaching process. In this study 235 ml of sulphuric acid and 241 ml of oxalic acid were needed for recovery of 1 gm of phosphorus. Sulphuric acid is used as the most common leaching acid because of its commercial availability in the market and its relatively lesser cost (Cieślik & Konieczka 2017). However, further study is required on the recovery and recycle of acid in the phosphorus recovery process which will have a significant effect on the economics.

CONCLUSIONS

The present study focussed on the leaching and extraction of phosphorus from POME sludge, with and without thermal treatment, using sulphuric and oxalic acids.

About 73% of phosphorus was removed from the wastewater stream in the form of sludge. The mineral composition revealed that the phosphorus in the sludge was around 11.8% and was bound with iron as iron phosphates. The phosphorus was released from iron complexes by combusting the sludge at higher temperatures which lead to the formation of acid insoluble iron compounds like oxides (FeO) and acid-soluble phosphorus from iron phosphates. The sludge recovery study indicated the highest yield of phosphorus was 85% and 83% for the sludge combusted at 900 °C, at L/S ratio of 200 ml/g, using sulphuric acid and oxalic acid, respectively. The sulphuric acid performance was found to be superior to oxalic acid when the combustion temperatures were750 °C and 900 °C. However, the performance of oxalic acid was found to be better than sulphuric acid for oven dried sludge and the sludge combusted at 600 °C. The oxalic acid forms complexes with iron and also release protons. This dual phenomenon with oxalic acid leads to higher recovery of phosphorus for the sludge combusted at 600 °C and for oven dried sludge. The phosphorus recovery mechanism was found to be dependent on combustion temperatures, as the sludge contains 43% of iron by weight, which has to release phosphorus. Effective removal of phosphorus from POME in a bipolar mode electrocoagulation (CBME) system has been demonstrated. This study showed that the phosphorus could be recovered effectively, through conventional acid treatment, from the sludge that is generated in the process. The recovered phosphorus may be used for manufacturing fertilizers and other useful chemicals. CBME system can provide a zero-waste treatment solution to palm oil mill effluent. However, further study is required on acid recycle in the phosphorus recovery process which will have a significant effect on the economics of the overall treatment process.

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