

Phosphorous recovery in a pretreated effluent by wet oxidation of sewage sludge.

J. Canas-Jimenez^(1,2), S. Alvarez-Torrellas⁽¹⁾, B. Hermana⁽²⁾, J. García⁽¹⁾

¹Complutense University of Madrid. Dept. of Chemical and Materials Engineering. Research group: Catalysis and separation processes, Avda. Complutense s/n, 28040 Madrid, Spain

²Ecolotum, Energía Recuperable, S.L., Calle del Cidro, 3, 28044 Madrid, Spain.

Javier Canas-Jimenez, +34 661373553, jcanas@ucm.es / javier.canas@ecolotum.es.

Abstract

OSCAR® is a technological process of wet oxidation (WO) of sewage sludge developed by ECOLOTUM and validated in a pilot plant operated in a Spanish wastewater treatment plant (WWTP). At elevated temperature (30-300°C) and pressure (20-200 bar) conditions were achieved high organic matter (up to 85% in COD) and total solids (up to 75%) reductions. Both the liquid and solid effluent after OSCAR® process contain amounts of phosphorus that can be recovered. Considering the circular economy around the phosphorus, the objective of this research is to maximize its valorization in both effluents.

In the liquid effluent, phosphorus present (up to 90 mgP/L, mostly as orthophosphates) is recovered by chemical precipitation as struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6 \text{H}_2\text{O}$), a slow-release fertilizer. P recoveries greater than 95% were achieved, and struvite purity reached 90%. The solid effluent analyzed after filtration and drying (68 mgP/g solid) was treated by acid leaching to achieve up to 60% of phosphorus recovery. All phosphorus extracted was in orthophosphate form.

Keywords

Wet oxidation, phosphorus, struvite, valorization, leaching, circular economy.

Introduction

Wet oxidation (WO) is a well-known process for the treatment of liquid wastes with high organic matter content. Its efficient and cost-effective compared to those technologies applied to wastes too diluted to be incinerated but too concentrated or poorly biodegradable to be treated by usual biological or chemical treatments [1]. This includes sewage sludge from municipal or industrial wastewater, without the need for dewatering and/or drying, with a solids content of up to 5% and mainly organic in nature. WO process has been studied in a wide range of operating conditions, relative to temperature (100-330°C) and pressure (40-230 bar). Using pure oxygen maintaining the liquid phase is the way to break C-C bonds to the large amount of toxic and hazardous organic compounds [2].

The end products of WO are CO_2 and H_2O , but the presence of short-chain organic compounds as reaction intermediates is common, such as volatile fatty acids (VFA). All are easily biodegradable, but refractory to oxidation, being acetic acid the most important. Also, liquid effluent is free of pathogens, while in the case of nutrients, nitrogen in the form of ammonium and phosphorus in soluble form, as orthophosphates, were detected. The solid effluent is stabilized and mostly inorganic, with the presence of heavy metals and insoluble phosphorus, while the gaseous effluent is free of the toxic compounds usually produced in incineration. For all these reasons, wet oxidation is currently installed in about 200 industrial-scale plants around the world for the treatment of municipal and industrial sewage sludge [3].

OSCAR®, the technology property to ECOLOTUM, Energía Recuperable S.L., is the implementation of this process operating in continuous flow and under subcritical water point, to maintain liquid phase. It is key to ensure a high oxygen content dissolved and to promote the oxidation kinetics. Supercritical conditions should also be avoided because of the precipitation and corrosion problems. For this, stability in the reaction must be ensured, maintaining a stable temperature to avoid the formation of hot spots that could lead to a supercritical phase, and also favor the extraction of the energy produced by the oxidation. Due to its exothermicity, self-thermal from a certain organic matter content, energy recovery is one of the keys to the technology.

Nutrients play an important role in WWTPs from both a sustainability and legislative point of view. In order to avoid eutrophication, a pollution caused by excessive nutrient content in the water, the discharge legislation in WWTPs regarding nutrients is becoming more and more restrictive, especially with respect to phosphorus.

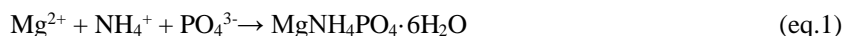
P is an indispensable component for life, necessary for the formation of genetic chains (DNA and RNA), constitutes the cell membrane of organisms and is key to their energy metabolism (ATP). Widely used as an agricultural fertilizer, phosphorus is a non-renewable resource obtained from phosphate rock mines, of which multiple authors and agencies confirm its depletion in short-medium term (30-200 years) [4]. Furthermore, the high geographic concentration (up to 72%) of phosphate rock reserves in Morocco and Western Sahara, is a challenge about environment, but also of political nature [5].

All this has driven the need for its recovery, around the concept of circular economy, being sewage sludge a very interesting source for this purpose. After the discovery of struvite deposits formed by spontaneous precipitation in the pipes of several WWTPs, this compound began to receive great interest. It is a white mineral with a crystalline structure and a pyramidal orthorhombic structure. Also, its easy to recovery and valuable as a slow-release fertilizer [6]. Current ways of recovering phosphorus in the form of struvite are mainly carried out on the liquid fraction resulting from anaerobic sludge digestion, since it concentrates large amounts of $P-PO_4^{3-}$ as well as $N-NH_4^+$ generated by the microorganisms.

The usual treatments in WWTPs are designed to remove phosphorus from the water line in order to ensure discharges below the legislation. The addition of metallic coagulants such as $FeCl_3$, $Ca(OH)_2$ or $Al_2(SO_4)_3$ in primary treatment and settling after secondary treatment means that more than 90% of the phosphorus at the inlet of WWTP ends up in the sludge line [7], [8]. Moreover, according to own characterizations, it has been found that the solid present in the sludge concentrates almost 20 times more phosphorus than the accompanying liquid fraction, being analyzed a mixed sludge coming from an urban WWTP. This is the reason why it is very interesting to recover phosphorus from the whole sludge and not only from the liquid fraction, as is currently mostly done.

Several industrial scale applications have been developed for the recovery of phosphorus in the form of struvite using sewage sludge as raw material. The AirPrex® process, currently operated at several WWTP in Germany and the Netherlands, uses the effluent after anaerobic digestion of the sludge without being dewatered, while the PHOSHPAQ process, implemented in a WWTP in the Netherlands, treats the liquid fraction of the dewatered sludge. Both require the addition of Mg and through aeration achieve CO_2 stripping to reach a slightly basic pH (8-8.5) which causes spontaneous precipitation of struvite in presence of ammonium [9]. At laboratory scale, phosphorus recovery as struvite was studied in a liquid effluent after WO of municipal sewage sludge [10] similar to that applied in this work.

Struvite is obtained following the equation 1, using equimolar relations between the three main components, in an aqueous media. The presence of other compounds, mainly Ca, can compete in the phosphate precipitation process at basic pH values, forming different compounds such as hydroxyapatite or monocalcium phosphate [11], [12].



Struvite precipitation occurs spontaneously at basic pH, being the main operative parameter that governs it together with the excess of Mg, which implies the supersaturation of the medium. The agitation speed and time or temperature also affect the process, but in this work, they are kept constant. Furthermore, in order to consider obtaining struvite on an industrial scale, other parameters specific to the formation and growth of crystalline structures must be taken into account, with the aim of obtaining certain particle sizes [6]. However, these factors are not taken into account in this work, since it is simply intended to demonstrate the technical feasibility for the recovery of phosphorus in the form of struvite.

The stabilized and mostly inorganic solid effluent after WO contains amounts of inorganic phosphorus that can be valorized. Direct agricultural application is not possible due to the toxicity generated by the high presence of heavy metals. Therefore, studies are focused on extracting the phosphorus from the solid matrix into an aqueous one, as soluble orthophosphates, similar to the recovery of incinerated sewage sludge ash (ISSA) which is comparable in composition to the solid after WO [13].

Several studies about chemical extraction of phosphorus reported good results both using ISSA to solid after WO [14], [15]. Phosphorus leaching was studied in a preliminary approximation to determine the P-extractable in solid, aiming to maximize its recovery.

The objective of this work is to evaluate an integral valorization of phosphorus from both liquid and solid effluents generated after the wet oxidation of sludge. Liquid effluent, which contains dissolved orthophosphates, was used to obtain optimal conditions for struvite precipitation. About solid effluent, was

treated by acid solutions to maximize phosphorus leaching. A simulation of integrating phosphorus recovery in both effluents is carried out based on the mass balance.

Material and methods

OSCAR® wet oxidation process

WO process was carried out in a pilot plant of the OSCAR ®technology, owned by ECOLOTUM; Energía Recuperable S.L., and situated in a Spanish wastewater treatment plant (WWTP). The sewage sludge used was mixed, obtained by collecting the sludge obtained from the primary and secondary treatment of the WWTP.

The reactor operates at high pressure and temperature, in subcritical water conditions, but close to it, in a continuous flow of 50 L/h and using oxygen pure in a poorly excess respect to stoichiometric. Specific operating conditions and other technical aspects are omitted in order to preserve business knowledge. The effluent obtained after WO, a mixture of liquid and solid, was collected and conveniently transported and stored in a refrigerator at 5°C to carry out the phosphorus valorization experiments. The mixture was separated by vacuum filtration on filter paper. Solid collected was dried at 105°C during 12h and after that, was ground and sieved to 355µm.

Both the inlet sewage sludge and the outlet stream from the WO process were analyzed to evaluate the organic matter and solid content using standard methods [16]. Parameters measured were chemical oxygen demand (COD), by spectrophotometry following colorimetric method using acid-dichromate commercial vials. Total solids (TS), total volatile solids (TVS) and total fixed solids (TFS) was analyzed by gravimetric method.

The solid after WO was analyzed by X-ray fluorescence (XRF) and microelemental analysis to determine both heavy metals and other inorganic compounds to organic C and N content.

Struvite precipitation experiments

Struvite experiments were carried out using a Jar Test installation to carry out several experiments at the same time. In 600 mL borosilicate beakers, 500 mL of liquid effluent are placed and the precise amount of the NaOH, 1M solution and the solid $MgCl_2 \cdot 6H_2O$ is dosed. The struvite obtained was separated from the liquid by vacuum filtration on filter paper (0.45µm) and, after washing with ultrapure water, it was dried at room temperature for 24 h before characterization.

An experimental design was carried out to optimize the operating conditions evaluated, pH and molar ratio Mg/P used, evaluating the maximization of phosphorus recovery in the liquid after precipitation.

In the liquid, both before and after precipitation, the orthophosphate content is analyzed. Total phosphorus was analyzed to verify that the presence of orthophosphates is the major form of soluble phosphorus. Both measurements were performed by spectrophotometry UV-Vis in a pF11 Macherey-Nagel photometer, using commercial vials according to the molybdate/ascorbic acid blue method. pH was monitored using a micropH meter 2002 by Crison Instruments.

The chemicals used were: NaOH (quality analysis, provided by Merck), $MgCl_2 \cdot 6H_2O$ (99% purity, provided by Panreac).

The solid obtained was characterized both by microelemental analysis (MA), by combustion, and inductively coupled plasma optical emission spectrometry (ICP-OES), after acid digestion in 1% of nitric acid and heating up to 100°C. In addition, some XRF analysis was performed to discard the co-precipitation of other ions, and by X-ray diffraction (XRD), the crystalline structure of the precipitated solid was analyzed and compared to pure struvite.

Solid leaching experiments

For each experiment, miliQ water and solid effluent dried, ground and sieved was used. It was placed in a 250mL Erlenmeyer flask with constant magnetic stirring at 450rpm, at ambient conditions and measuring pH in continuous mode.

An initial sequential extraction was carried out using 1g of solid after WO was performed to determine the fractions of phosphorus bound to other compounds in solid. Between each extraction, solid was washed

with 20mL KCl, 1M and filtered again. The procedure was reached following the next steps according to EN-12457:2002 [15]:

- i) *NaHCO₃ fraction*. 20mL of NaHCO₃, 0,5M to determinate P fraction weakly bound.
- ii) *NaOH fraction*. 20mL of NaOH, 0.1M to determinate Al and Fe bound P.
- iii) *HCl diluted fraction*. 20mL of HCl, 1M to define Ca bound P.
- iv) *HCl concentrated and hot fraction*. 12mL of HCl, 10M in a water bath at 80°C to extract stable P. This fraction needs a large amount of energy to be recovered.

After that, preliminary tests were carried out using HCl to evaluate P leaching by varying the contact time, acid concentration and liquid/solid contact ratio, using 1g of solid in each.

The chemicals used were: KCl (chemically pure crystal provided by Probys), NaHCO₃ (chemically pure provideb by Solvay), HCl (fuming $\geq 37\%$ quality analysis provided by Honeywell Fluka).

The solid used as raw material was characterized by X-ray fluorescence (XRF) and microelemental analysis (MA), while total phosphorus content was measured after digestion in aqua regia following the UNE-EN 13657:2003, using commercial vials. The liquid before and after leaching experiments was characterized for phosphorus content as orthophosphates also using commercial vials, as in the struvite experiments. Total phosphorus after leaching was analyzed in some cases, to compare how much was extracted in orthophosphate form.

Results and discussion

OSCAR® wet oxidation process

Several WO sewage sludge experiments were accomplished for two weeks maintaining the operational conditions of temperature, pressure and oxygen dosage as constant. The initial sewage sludge used in each experiment was mixed to obtain an average value to evaluate the constant operation of the process on different days. The final OSCAR® effluent was also mixed in order to compare the efficiency of the process, which is shown in Table 1. After that, mixed effluent was separated obtaining liquid and solid effluents, which were used as raw material for both valorization processes. The characterization of the separate effluents is shown in Table 2.

Table 1. Characterization inlet and outlet of OSCAR® process.

Parameters	Sewage sludge inlet	Effluent mixture outlet	Conversion (%)
COD (gO ₂ /L)	17,9	3,61	79,94
TS (g/kg)	27,80	7,05	74,64
TVS (g/kg)	12,1	0,60	95,04
TFS (g/kg)	9,2	6,70	27,17

WO sewage sludge treatment achieved conversions of up to 80% in chemical oxygen demand (COD) and up to 75% in total solids (TS), of which 95% were total volatile solids (TVS), associated with organic matter. The fixed total solids (TFS) are related to inorganic compounds, phosphorus being the most valuable. OSCAR® is confirmed as a viable technology to destroy toxic and hazardous organic compounds present in the initial sewage sludge.

Table 2. Characterization of liquid and solid effluents after OSCAR® process.

Parameters	Liquid effluent	Parameters	Solid WO effluent
COD (g O ₂ /L)	3,11	Total, P (mg P/g solid)	68,2
Total, P (mg P/L)	93,1	FRX analysis	Oxides (%)
P-PO ₄ ³⁻ (mg P/L)	86,11	Si	14,01
Total, N (mg N/L)	975	Fe	12,79
N-NH ₄ ⁺ (mg N/L)	848	Al	7,39
Mg ⁺ (mg Mg/L)	2,5	Ca	5,84
Ca ²⁺ (mg Ca/L)	7,1	Mg	2,4
		K	1,8
		∑ (Ti, Na, Zn, Cu, Mn, Ga, Pb, Ba, Sr, Co, Cr, Zr, Ni, Rb, Sn)	1,39

The phosphorus content in the liquid and solid effluent motivates valorization studies as an integral mechanism to improve the OSCAR® process from a sustainable point of view. WO can be understood as a process of sludge pretreatment for subsequent valorization or recovery in an easier way, in this case, by transforming phosphorus into soluble species that can be easily recovered.

Struvite precipitation experiments

The main objective is to maximize phosphorus recovery, measured in the liquid before and after precipitation. For this purpose, several experiments were performed by varying the two main operating parameters; pH and Mg/P molar ratio. The results are shown in Figure 1.

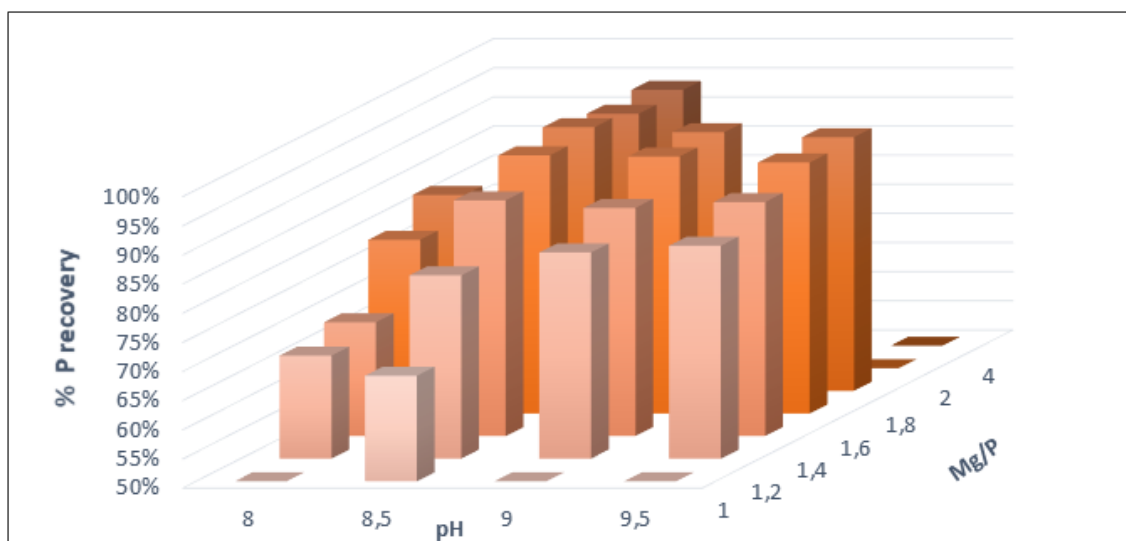


Figure 1. Phosphorus recovery experiments varying pH and Mg/P molar ratio.

It can be observed that pH is the limiting parameter of the process, considering that at pH 7.5 no precipitate was obtained. In addition, it is observed that phosphorus recovery increases up to the pH value of 8.5, while when it is exceeded, P recovery does not increase, and even decreases slightly. Therefore, pH 8.5 is taken as the optimum value for P recovery. Figure 2 (left) shows this comparison.

On the other hand, the Mg/P molar ratio seems to behave in a more linear way, always favoring P recovery when the excess of Mg²⁺ increases. Therefore, the recovery is compared as a function of the molar ratio at pH 8.5, as shown in Figure 2 (right). Thus, the recovery reaches a maximum when the Mg/P molar ratio is 1.8, increasing clearly from 1, and then decreasing slightly at higher values, corresponding to 2 and 4.

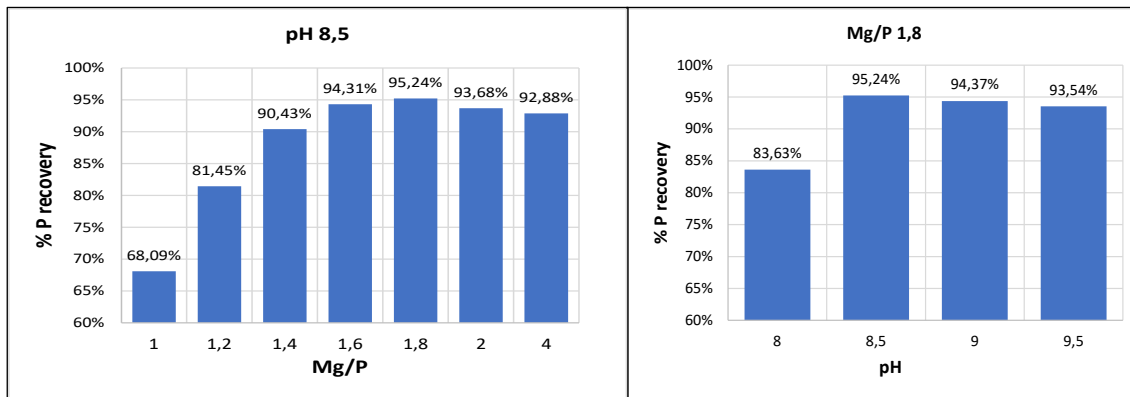


Figure 2. Phosphorus recovery at pH 8,5 (left) and at Mg/P (right).

The optimum struvite (obtained in the operating conditions of pH 8.5 and molar ratio Mg/P 1.8), will be called this way from now on, but it is emphasized that it has been defined only in the range studied and taking into account only phosphorus recovery. An economic study will be necessary to define the parameters that, in addition to maximizing recovery, minimize cost.

After precipitation and filtration, the solid is characterized. The three main components (P, N and Mg) are determined by ICP-OES and compared with the literature reference values to define struvite purity precipitated while organic matter content is analyzed by microelemental analysis (MA). In addition, ICP-OES analysis of the optimum struvite is carried out and includes the presence of other co-precipitated compounds. Results are shown in Table 3.

Table 3. Struvite purity and organic matter content in experiments of struvite (left) and analysis of optimal struvite (right).

pH	Molar ratio Mg/P	%P purity	%N purity	% Mg purity	% C	Compound	Concentration (µg/g struvite)
8	1,2	92,7%	91,1%	65,7%	0,12%	Si	19255
8	1,4	92,7%	91,1%	72,7%	0,17%	Al	1687
8	1,6	91,9%	91,2%	73,7%	0,14%	Fe	563,5
8	1,8	92,7%	91,2%	73,7%	0,1%	As	123,5
8,5	1,2	91,9%	90,0%	73,7%	0,17%	S	72
8,5	1,4	91,9%	89,8%	73,7%	0,18%	Mn	46,5
8,5	1,6	91,9%	89,7%	72,7%	0,17%	Zn	29
8,5	1,8	92,3%	90,0%	72,7%	0,22%	Cu	13,6
8,5	2	91,1%	88,4%	72,7%	0,15%	Cr	12,2
8,5	4	79,2%	76,7%	70,5%	-	Cd	<10* Below the limit of detection (LOD).
9	1,2	90,3%	89,7%	72,7%	0,2%	Mo	<10* Below the limit of detection (LOD).
9	1,4	89,5%	89,1%	72,7%	0,2%	Ni	<10* Below the limit of detection (LOD).
9	1,6	88,0%	87,6%	71,7%	0,26%	Pb	<10* Below the limit of detection (LOD).
9	1,8	87,2%	87,0%	69,7%	0,28%	Hg	<10* Below the limit of detection (LOD).
9,5	1,2	88,0%	89,3%	68,7%	0,33%		
9,5	1,4	87,2%	87,4%	68,7%	0,33%		
9,5	1,6	88,0%	86,9%	70,7%	0,35%		
9,5	1,8	88,0%	87,6%	66,7%	0,35%		

As the pH increases, the struvite becomes less rich in both phosphorus and nitrogen, slightly reducing the purity values. The reduction is very small, just 5%, in both compounds, being the experiments at pH 8 the ones that provide the precipitate richer in P and N. The purity values of both compounds are very high, greater than 90% under optimum conditions. The magnesium content follows a similar trend, but with a much lower richness compared to pure struvite, reaching up to 74%.

In the range of molar ratio Mg/P between 1.2-1.8 it is not seen that the composition of any of the three compounds in the precipitate follows any trend. Similar contents are obtained in all the experiments carried out. In the experiments at pH 8.5, where a greater excess of magnesium is used, it is observed that the content of the three compounds that form part of the precipitate is reduced as the excess increases, being more pronounced at a molar ratio of 4.

On the other hand, the organic matter content behaves in the opposite way. It is minimal at pH 8 and increases as the pH rises. However, the percentage of carbon in struvite does not exceed 0,35% in any case. This is important for the possible application of struvite as a fertilizer.

Regarding the compounds found in the optimum struvite, which can be considered impurities, the sum of all of them amounts to 2,2%, with Si being the majority reaching 1,93%. Micronutrients such as Fe, Cu, Zn or Co are detected, however small amounts of toxic or dangerous compounds to be used in agriculture, such as Cr, As or Hg, are also present.

According to recent European legislation [17] struvite is no longer considered waste but a potential fertilizer product, offering a new alternative towards the future of the circular economy in relation to the valorization of phosphorus in this way.

Solid leaching experiments

Solid characterization is shown in Table 2. Preliminary solid leaching tests were performed following the sequential extraction described above. The average results of the duplicate extraction tests are presented in Figure 3.

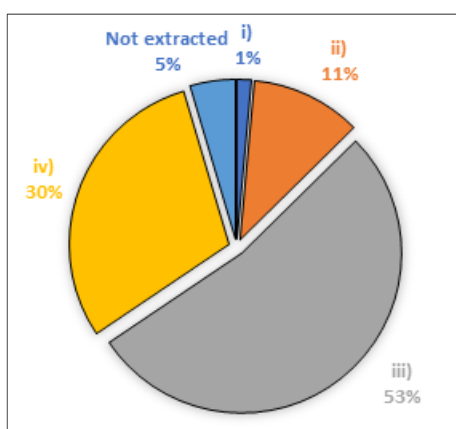


Figure 3. Sequential extraction result of chemical phosphorus extraction.

The majority fraction of phosphorus present in the solid corresponds to that extractable with dilute acid HCl (53%), related to Ca-bound phosphorus. Then, 30% is stable phosphorus, which is extracted with concentrated, hot acid. Thus, more than 80% of the phosphorus present is extracted with acids, while bases account for only 12%, of which 11% corresponds to the strong base NaOH and 1% to the weak base NaHCO₃. A small fraction (5%) was not extracted.

Considering this, leaching tests will be carried out using dilute HCl acid. Some operating parameters were modified to determine the effect on phosphorus leaching, such as the contact time between phases, the concentration of acid used and the liquid/solid ratio employed. In addition, both total phosphorus and phosphorus in the form of orthophosphates will be analyzed in the liquid to define how much is extracted in this form.

Leaching test results varying contact time and comparing phosphorus total and as orthophosphate (P-PO₄³⁻) are shown in Figure 4 while Figure 5 shows the influence of acid concentration and liquid/solid ratio on phosphorus recovery by leaching. The main factor defining the efficiency of each experiment is the amount of phosphorus leached from the solid to the liquid.

Increasing the contact time between the two phases from 3h to 24h does not significantly increase phosphorus leaching, although in one of the tests it reaches 10%. Despite of this, it is considered that the trend of increasing time does not imply a considerable increase in leached phosphorus. It is assumed that this increase is not such as to justify such a large difference.

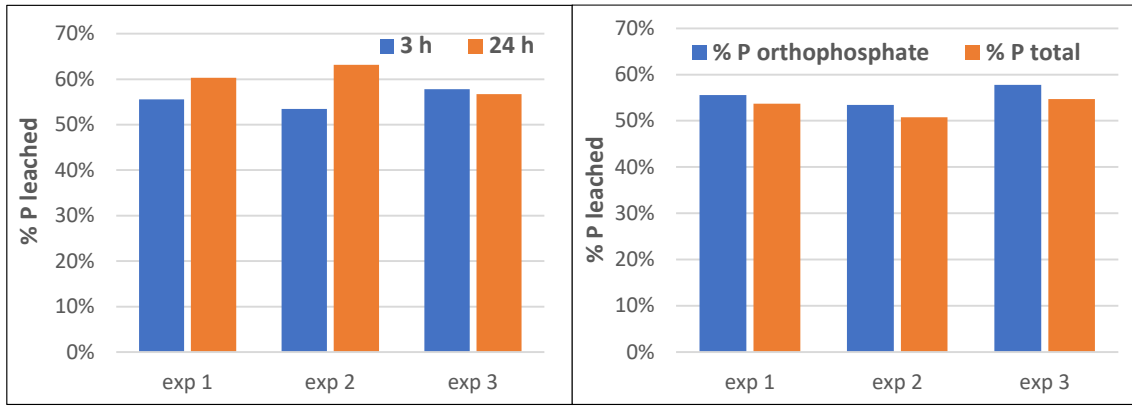


Figure 4. Phosphorus leached varying contact time (left) and comparing phosphorus measurement (right).

Orthophosphates is the form in which phosphorus is obtained after the leaching process of the OSCAR® solid. This is important since this leached phosphorus will be recovered in the form of struvite. Measurements even indicate a higher presence than for total phosphorus, which is assumed as a standard deviation of measure.

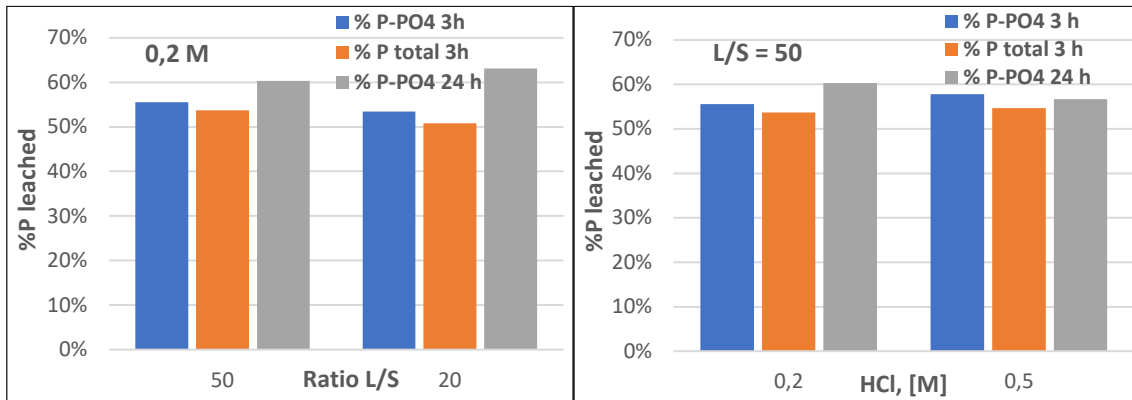


Figure 5. Phosphorus leached varying acid concentration (left) and liquid/solid ratio (right).

Reducing the liquid-to-solid ratio (L/S) from 50 to 20 only reduces phosphorus leaching over 2% by average, so it is considered that the amount of liquid is, in both cases, in sufficient excess not to be a limiting factor in the process.

About varying HCl concentration, the objective is to reduce the amount of acid, evaluating whether phosphorus recovery is reduced. Phosphorus leaching tests using 0.5M and 0.2M HCl reported similar values, with deviations of 2%, which is considered negligible. This indicates that the minimum acid dose to maximize phosphorus leaching recovery may be even lower than 0.2M, HCl. Future experimentation will be oriented towards this optimization, in addition to evaluating different acids and applying economic criteria.

Conclusions

Several WO experiments of sewage sludge using OSCAR® technology were carried out in a pilot plant property of ECOLOTUM. The sewage sludge used was the mixture after the primary and secondary decantation of a Spanish WWTP.

The process achieved high organic matter reduction up to 80% in COD and up to 75% in TS. Of these, a reduction of up to 95% was reached in TSV, associated with organic matter. In addition, the liquid and solid effluents generated contain quantities of phosphorus that can be recovered. In liquid effluent (86,11 mgP/L), it is found as orthophosphates, easily recoverable by struvite precipitation while in solid (68,2 mgP/g solid), it is extracted also as orthophosphates by leaching using acid.

Phosphorus recovery from the liquid effluent by struvite ($\text{MgNH}_4\text{PO}_4 \cdot 6\text{H}_2\text{O}$) precipitation reaches 95.24% at pH 8.5 and Mg/P molar ratio of 1.8, which are the optimum values in range studied. Increasing the excess of Mg above this value does not increase phosphorus recovery, as is the case with pH. An economic study will be necessary to define optimal parameters, also taking into account the economic cost.

The purity of the struvite related its nutrient content (P, N) is greater than 90% in the experiments at pH 8 and 8.5, decreasing slightly as the operating pH increases. Magnesium presence in precipitated also follows the same trend, but its content with respect to the reference mineral is lower, up to 74%. As the Mg/P molar ratio increases above 2, the purity of struvite decreases, while below 2, no significant differences are appreciated.

The organic matter content in struvite is minimal at lower pH to the range, always below 0,35%, useful to be used in agriculture. The presence of co-precipitated impurities in optimal struvite is mainly of harmless inorganic compounds such as Si, which represents 89% of them. There are also micronutrients such as Fe, Cu and small amounts of toxic heavy metals such as Cr, As or Hg.

Initial characterization of the solid effluent was performed in a sequential extraction in which 53% of the phosphate present was leached using a dilute acid solution (HCl, 1M), related to Ca-bound phosphorus. Preliminary experiments were conducted by varying the contact time, acid concentration and liquid/solid (L/S) volumetric ratio.

Increasing the leaching contact time from 3h to 24h has not significantly raised the phosphorus recovery. As for the liquid/solid (L/S) ratio, experiments using 50 and 20 reported similar results on leached phosphorus, so it is considered to have sufficient excess of liquid. Respect to acid concentration, experiments using 0,5M and 0,2M reported similar values, so it is considered that its dosage can still be further reduced. Spot measurements of both total phosphorus and orthophosphates were carried out, confirming that it is found in this form in its totality.

Future experimentation will be aimed at optimizing these operating parameters and applying economic criteria.

Acknowledgments

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