PHORWater

Integral Management Model
for Phosphorus recovery
and reuse from Urban Wastewater

LIFE12 ENV/ES/000441

Good Practice Manual to maximise phosphorus recovery.

Project Deliverable D.1.9

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1. BACKGROUND

The main objective of PHORWater project is to demonstrate the viability and sustainability of the correct global management of phosphorus recovery in Wastewater Treatment Plants (WWTP) in order to maximise struvite ($MgNH_4PO_4 \cdot 6H_2O$) recovery by crystallization.

Phosphorus recovery by struvite crystallization is one of the most widely recommended technologies for treating sludge digester liquors especially in WWTP with enhanced biological phosphorus removal (EBPR). In the EBPR process, phosphates and other ions (i.e., Mg^{2+}, K^{2+}) are taken up and stored as polyphosphates (Poly-P) inside the Phosphorous Accumulating Organisms (PAO). During the anaerobic digestion process, Poly-P are released to the liquid phase increasing notably the phosphorus (P), magnesium (Mg) and potassium (K) concentration. Moreover, ammonium (NH_4) concentration increases significantly as proteins are degraded, and dissolved Mg, P, calcium (Ca) and K concentrations increase due to the cell lysis.

The increase in the concentrations of dissolved components and the high pH achieved during anaerobic digestion increase the P precipitation potential in this stage of the treatment system. This uncontrolled precipitation causes significant operational problems which increases maintenance costs and reduces P concentration in the sludge liquors that could be recovered.

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Therefore, in order to guarantee P-rich streams and thereby obtain high struvite production in the crystallization process, the uncontrolled P precipitation in the digester should be reduced.

As a first step, intensive analytical campaigns must be carried out under normal operating conditions at different points of the water and sludge lines of the WWTP. The aim of this characterisation is to optimise the EBPR process in the activated sludge system and to assess P precipitation in the plant from routine experimental analysis and simple mass balances. From the results obtained, it will be possible to decide the best strategies that reduce P precipitation in the sludge line and maximise P recovery as struvite in WWTP.

2. OBJECTIVE

The purpose of this deliverable (D1.9) is to develop a Good Practice Manual to maximise phosphorus recovery in WWTP.

At first, a general overview of the WWTP performance has to be carried out in order to assess the fate of P in each stage of the water and sludge treatment lines. From the data obtained in the sample campaigns P removal efficiency in the water line as well as precipitation processes in the sludge line can be evaluated.

Increasing the efficiency of the EBPR process and reducing the uncontrolled P precipitation in the sludge line is crucial to maximise P recovery in the WWTP. In order to achieve this objective, Good Practices in the water and sludge line operation are proposed.

3. ASSESSMENT OF PHOSPHORUS FATE IN WWTP

Figure 1 shows a typical biological nutrient removal WWTP flow diagram, where the main unit operations and sample points of the water and sludge lines are identified. Table 1 and Table 2 summarise the sampling points in the water and sludge line, respectively.
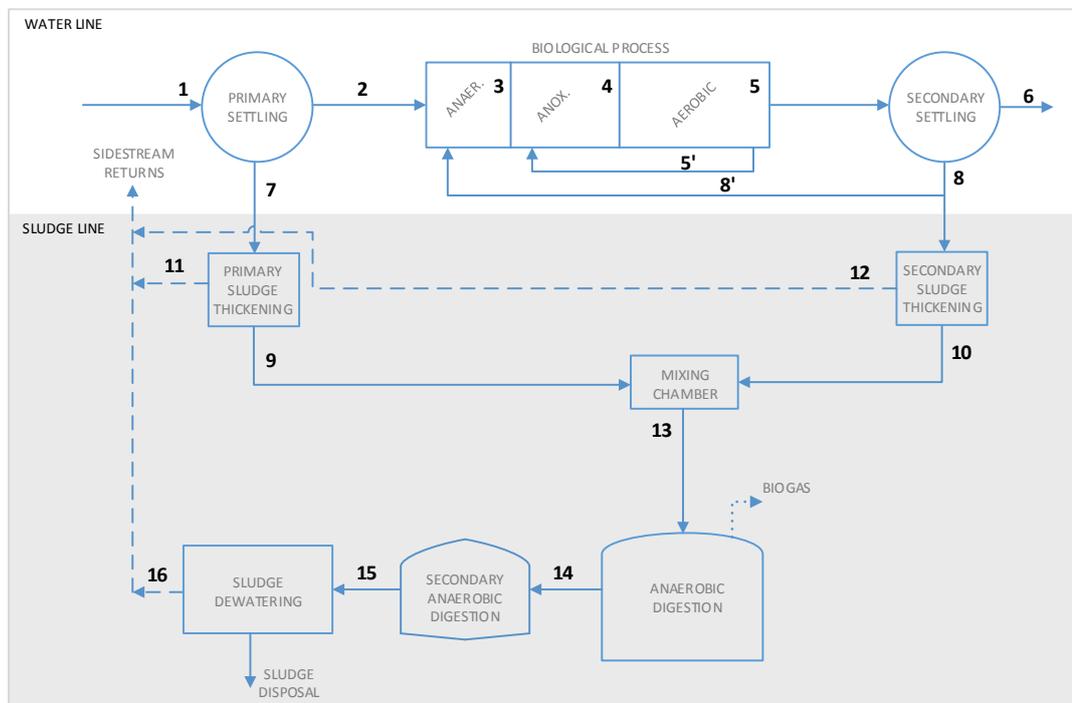

Figure 1. Typical flow diagram in WWTP with biological nutrient removal.

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Table 1. Sampling points in WWTP water line in accordance with Figure 1.

Sample number	Sample description
1	Inlet to primary settler (downstream of sidestream returns 11, 12 and 16)
2	Influent to biological process
3	End of anaerobic zone
4	End of anoxic zone
5	End of aerobic zone/effluent of the biological process
5'	Internal recycle
6	Secondary effluent

Table 2. Sampling points in WWTP sludge line in accordance with Figure 1.

Sample number	Sample description
7	Primary sludge
8	Secondary sludge (Waste activated sludge, WAS)
8'	Return Activated Sludge (RAS)
9	Thickened primary sludge
10	Thickened WAS
11	Primary sludge overflow
12	WAS liquor
13	Mixed sludge
14	Digested sludge
15	Thickened digested sludge
16	Filtrate/Centrate

3.1. Water treatment line

In order to study the fate of P in the wastewater line, sampling campaigns must be carried out under normal operating conditions. A general overview of the P fate in the wastewater treatment line is described below.

The efficiency of the EBPR process can be evaluated from the data obtained in the sampling campaigns.

Primary settling

The first stage where a fraction of phosphorous is removed is in the primary settler. Here, a fraction of the suspended matter that arrives to the WWTP is removed (sent to the sludge line) and thus the suspended phosphorous related with this settleable matter will be also eliminated.

In case of fermentation taking place in the primary settler, volatile fatty acids (VFA) will be produced. As this is the substrate used by PAO it is important to know whether VFAs are present or not in this stage.

Biological process

A typical biological process for nutrient (P and N) removal is composed of three contiguous zones. The first one is the anaerobic zone (Figure 1, sampling point 3). Here, PAO take up the VFAs and release P (in form of phosphates) to the bulk. Consequently, in the anaerobic zone it is expected to observe an increase of phosphates and a decrease of the VFA.

The next chamber is the anoxic zone (Figure 1, sampling point 4), where the denitrification process takes place (nitrate is reduced to nitrogen gas). In this zone, PAO are able to use nitrate as an electron acceptor and take up the phosphates from the bulk to store them as Poly-P. Therefore, a reduction of the phosphates content is expected in this chamber.

In the last zone, the aerobic zone (Figure 1, sampling point 5), NH_4 is oxidized to nitrite which is then oxidized into nitrate. In this aerobic zone PAO behave as in the anoxic

zone but more efficiently using oxygen as electron acceptor. Thus, an important reduction of the phosphates content is expected.

If the EBPR process is working correctly, it is expected to observe a reduction of the phosphates content at the end of the aerobic zone (Figure 1, sampling point 5) compared with the phosphates content in the influent of the biological process (Figure 1, sampling point 2).

Secondary settling
Despite a physical process like the secondary settling is supposed not to affect the concentration of phosphates, it has been observed in some cases a small increase of them. This is caused by the anaerobic conditions that could occur at the bottom of the clarifier and the decay processes.

3.2. Sludge treatment line

In order to study the fate of P in the sludge line, sampling campaigns must be carried out under normal operating conditions. Using the data obtained and performing a methodology previously developed from routine experimental analysis and mass balances in a digestion pilot plant (Martí et al., 2008a), P precipitation can be assessed.

This methodology has been used to assess P precipitation in two Spanish WWTP, located in Murcia (Barat et al., 2009a) and Tarragona (Barat el al., 2009b), respectively.

Predicting P precipitation is the starting point to design sludge management alternatives that minimise uncontrolled phosphorous precipitation and maximise P recovery by

crystallization in the WWTP.

Primary sludge thickening

With regard to the P dynamics in the primary sludge thickener, it is expected that phosphate release takes place due to the hydrolysis and subsequently fermentation of the organic matter. Moreover, as a consequence of this organic matter degradation, NH_4, K, Ca and Mg soluble concentrations also increase.

Despite the increase in ion concentrations, no precipitation processes are expected in the primary thickener due to the low pH values achieved as a consequence of the fermentation process.

Secondary sludge thickening

Secondary sludge (WAS sludge) can be thickened by gravity or flotation, or can be mechanically thickened.

Both gravity and flotation thickening can lead to a significant P release if anaerobic conditions are achieved inside the thickener. Under anaerobic conditions, fermentation of organic matter takes places increasing ion concentrations, as explained before. Also, as a consequence of the fermentation process, VFA are formed and PAO release the internal Poly-P to the bulk solution, increasing P concentrations. Although ion concentrations increase, precipitation processes are not expected due to the low pH values normally obtained.

Unlike gravity or flotation thickening, mechanical thickening avoids anaerobic zones formation, because the sludge residence time in this operation units is very

low.

Mixing chamber

Thickened primary sludge and thickened WAS are mixed before being stabilised in the anaerobic digester.

Under the conditions achieved in the mixing chamber (anaerobic conditions, WAS with PAO bacteria replenished with Poly-P, and presence of VFA from the thickened primary sludge) significant P can be released from Poly-P stored by PAO. As a consequence, K and Mg concentrations also increase. Moreover, if long retention times are maintained in the mixing chamber, hydrolysis and fermentation of particulate organic matter occur increasing P (and also NH_4, K, Ca and Mg) soluble concentrations in the system.

Since pH is normally low, P precipitation in the mixing chamber is not expected.

Anaerobic digestion

During the anaerobic digestion process, the stored Poly-P is released to the liquid phase increasing notably the soluble P, Mg and K concentration. Moreover due to the organic matter degradation, concentrations of NH_4, Mg, P, Ca and K also increase. However, in most anaerobic digesters a decrease of P, Mg and Ca soluble concentrations is observed which can be explained by the formation of precipitates. Both the increase in the concentrations of dissolved components and the high pH achieved during anaerobic digestion increase the P precipitation potential in this stage of the sludge treatment line.

Different phosphate compounds are likely to precipitate in anaerobic digesters causing significant operational problems that lead to an increase in maintenance costs (Wild et al., 1997). Moreover, this uncontrolled precipitation reduces P concentration in the sludge liquors decreasing the potential for P recovery by struvite crystallization.

Therefore, in order to guarantee P-rich streams and thereby obtain high struvite production in the crystallization process, the uncontrolled P precipitation in the digester should be reduced.

Post-digestion processes

Secondary anaerobic digestion and sludge dewatering are also critical operation units for P precipitation since high ion concentrations are present and pH values are high. Therefore, the uncontrolled P precipitation in the post-digestion processes should be controlled.

4. METHODOLOGY: MASS BALANCES TO ASSESS PHOSPHORUS RELEASE AND PRECIPITATION

In order to study the fate of P in the mixing chamber and in the anaerobic digester, a simple method based on PO_4-P, Ca^{2+}, Mg^{2+}, and K^+ mass balances was developed (Martí et al., 2008).

To perform these balances, several assumptions must be taken into account:

- Phosphorus stored as Poly-P is released during anaerobic digestion. In the mixing chamber Poly-P will be released if proper conditions are maintained (anaerobic conditions and presence of VFA).

- During Poly-P hydrolysis, the release of P is followed by a release of K and Mg at the same ratio as observed during the P uptake. Literature show values of 0.25-0.28 g Mg/g P (η_{Mg-P}) and 0.29-0.42 g K/g P (η_{K-P}) (Arvin and Kristensen, 1985; Brdjanovic et al., 1996; Jardin and Pöpel, 1996; Barat et al., 2005).

- Organic matter degradation produces a release of P, K and Ca associated with the content of each element in the organic matter. This release must be quantified when the mass balances are performed considering that thickened primary sludge and thickened WAS are similarly degraded in the organic matter during the hydrolysis process.

- As explained before P precipitation in the mixing chamber is not expected since pH is normally low. In the digester, several Ca and Mg phosphate compounds are likely to precipitate. It can be assumed that there is no chemical fixation of the K released in the digester. Only in the case of low NH_4 concentrations, different authors (Schuiling and Andrade, 1999; Wilsenach et al., 2006) have pointed out that potassium struvite (K-MAP, $KMgPO_4 \cdot 6H_2O$) could precipitate instead of ammonium struvite (MAP, $MgNH_4PO_4 \cdot 6H_2O$). However, in anaerobic digesters high concentration of NH_4 are achieved so no K-MAP precipitation is expected.

4.1. P, K, Mg and Ca content in the organic matter

As previously mentioned, the organic content of P, K, Mg, and Ca in the sludge must be determined. Since both thickened primary sludge and WAS sludge are treated in the sludge line, it is necessary to distinguish between the organic P, K, Mg, and Ca content in the two types of substrate:

- Thickened primary sludge:
From primary sludge characterisation, calculations could be made taking into account that the suspended P, K, and Mg were associated to organic matter. This assumption can be made if no precipitation of P, K, and Mg is detected in the primary sludge. However, it is impossible to accurately determine the Ca content in the organic matter of primary sludge due to its typically high inorganic content

mainly as calcium carbonate. Therefore organic Ca has been neglected. Experimental data obtained in different WWTP (Martí et al., 2008a; Barat et al., 2009a) are shown in Table 3.

- Thickened WAS:

In the thickened WAS, it is not possible to distinguish between the organic P, Mg, and K content and the content associated with Poly-P structure. Therefore, reported values for biomass composition must be assumed (Metcalf and Eddy, 2003) (See Table 3).

Once the organic P, K, Mg, and Ca content in the primary and WAS thickened sludge is determined, the organic content of these elements in the mixed sludge can be calculated taking into account the proportion of each sludge.

Table 3. Organic P, K, Mg and Ca in primary and secondary sludge.

	Primary sludge	WAS
P_{org} (mg/mgSV)	0.004-0.013	0.02
Mg_{org} (mg/mgSV)	0.001-0.005	0.004
K_{org} (mg/mgSV)	0.001-0.006	0.008
Ca_{org} (mg/mgSV)		0.004

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4.2. Mass balances

In accordance with the assumptions stated above and using the results obtained in the sampling campaigns, mass balances under steady state conditions can be carried out using the following equations:

- From the K mass balance (Eq. 1), the potassium released by PAO (K_{PAOrel}, M/T) can be calculated, considering that K_{ORGrel} (M/T) represents the potassium released from organic matter degradation, Q_{in} and Q_{out} (L^3/T) the influent and effluent flow rates and K_{in} and K_{out} (L^3/T) the influent and effluent soluble potassium concentrations.

$$K_{PAO_{rel}} = K_{TOTrel} - K_{ORGrel} = \left(\sum Q_{out} \cdot K_{out} - \sum Q_{in} \cdot K_{in}\right) - K_{ORGrel} \qquad (1)$$

- The phosphate released by PAO (P_{PAOrel}, M/T) can be calculated (Eq. 2) bearing in mind the mass of potassium released per mass of phosphate released (η_{K-P}). Then, from the phosphate mass balance, the phosphate fixed (P_{fix} (M/T) and %P_{fix}) by precipitation or adsorption can be obtained (Eq. 3 and Eq. 4): being P_{ORGrel} (M/T) the amount of organic phosphate hydrolysed and P_{in} and P_{out} (M/L^3) the influent and effluent phosphate concentrations.

$$P_{PAO\,rel} = K_{PAOrel} / \eta_{K-P} \qquad (2)$$

$$P_{fix} = \sum Q_{in} \cdot P_{in} + P_{ORGrel} + P_{PAOrel} - \sum Q_{out} \cdot P_{out} \qquad (3)$$

$$\%P_{fix} = \frac{P_{fix}}{\sum Q_{in} \cdot P_{in} + P_{ORGrel} + P_{PAOrel}} \cdot 100 \qquad (4)$$

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- The Mg mass balance can be applied in order to determine the magnesium precipitated (Mg_{prec}, M/T and $\%Mg_{prec}$) (Eq. 5 and Eq. 6) considering that Mg_{PAOrel} (M/T) represents the magnesium released by PAO, Mg_{ORGrel} (M/T) the magnesium released by organic matter degradation, Q_{in} and Q_{out} (L^3/T) the influent and effluent flow rates and Mg_{in} and Mg_{out} (M/L^3) the influent and effluent soluble magnesium concentrations.

$$Mg_{prec} = \sum Q_{in} \cdot Mg_{in} + Mg_{ORGrel} + Mg_{PAOrel} - \sum Q_{out} \cdot Mg_{out} \qquad (5)$$

$$\%Mg_{prec} = \frac{Mg_{prec}}{\sum Q_{in} \cdot Mg_{in} + Mg_{ORGrel} + Mg_{PAOrel}} \cdot 100 \qquad (6)$$

- Finally, the calcium precipitated (Ca_{prec}, M/T and $\%Ca_{prec}$) can be obtained from the calcium mass balance (Eq. 7 and Eq. 8), being Ca_{ORGrel} (M/T) the calcium release due to organic matter degradation, Q_{in} and Q_{out} (L^3/T) the influent and effluent flow rates and Ca_{in} and Ca_{out} (M/L^3) the influent and effluent soluble calcium concentrations.

$$Ca_{prec} = \sum Q_{in} \cdot Ca_{in} + Ca_{ORGrel} - \sum Q_{out} \cdot Ca_{out} \qquad (7)$$

$$\%Ca_{prec} = \frac{Ca_{prec}}{\sum Q_{in} \cdot Ca_{in} + Ca_{ORGrel}} \cdot 100 \qquad (8)$$

5. GOOD PRACTICES TO MAXIMISE PHOSPHORUS RECOVERY BY CRYSTALLIZATION IN WWTP

It is strongly recommended the use of a software tool for simulating and optimising the WWTP performance in order to maximise P recovery. This software tool allows simulating different scenarios before the implementation in the WWTP.

It must be highlighted that the necessary analytical characterization of the water and sludge lines (see Deliverable D1.3) could change depending on the model implemented in the software tool employed.

The model suggested as appropriate for this purpose should be a general model able to simulate every operation unit in a WWTP considering the most important physical, chemical and biological processes. One of the models that consider these requirements is the BNRM2 (Barat et al., 2013).

BioWin®, GPS-X®, SIMBA®, WEST® and DESASS® are some of the most used simulation platforms for WWTP's modelisation and simulation (Ferrer et al., 2005).

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5.1 Water treatment line

The aim of the EBPR is to eliminate the phosphates from the wastewater transferring them into Poly-P which will be forwarded to the sludge line. In order to maximize this EBPR, the next good practices can be applied to the water line.

Primary settling

As previously mentioned, VFA are the main substrate for PAO. It has been reported that PAO are promoted when using VFA/P ratios in the influent wastewater (Figure 1, point 2) between 10 and 20 mg COD/mg $P-PO_4$. In WWTP where this ratio is not achieved, it is possible to elutriate the primary sludge in order to enhance the sludge fermentation and thus the production of VFA.

However, it is important to keep the VFA/P ratios in the influent wastewater below 50 mg COD/mg $P-PO_4$ to avoid the growth of Glycogen Accumulating Organisms (GAO) especially if the influent pH is below 7. GAO outcompete PAO resulting in a poor biological phosphorus removal efficiency.

Biological process

There are several factors that can promote the development of PAO. One of the most important is to use low sludge retention times (around 8-10 days). However, special attention must be paid to the nitrification process, since low sludge retention times could negatively affect to this process.

Another important factor is to keep the anaerobic zone free of nitrates. This would reduce the phosphate release and thus, the overall process efficiency. Therefore, the RAS flow rate (Figure 1, point 8') must be controlled in order to avoid the inlet of

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nitrates to the anaerobic chamber. Increasing the internal recirculation flow rate (Figure 1, point 5') could prevent the arrival of an excess of nitrates to the secondary settler and thus to the anaerobic zone.

Secondary settling

If an increase of the phosphates is observed between the inflow and the effluent of the secondary settler (Figure 1, points 5 and 6), it is recommended to increase the RAS flow rate in order to avoid anaerobic zones at the bottom of the secondary settler.

5.2 Sludge treatment line

Using the results from the mass balances in the sludge line it will be possible to describe good operational practices to minimise the P precipitation and to maximise the potential P available for the crystallization step.

In the anaerobic digester, the P precipitation potential increases due to both the increase in the concentrations of dissolved components and the increase in pH. The uncontrolled P precipitation avoids its subsequent recovery as struvite in the crystallizer. Since the pH in the digester is not an easy-control parameter, decreasing the available P (i.e. the soluble P concentration) in the digester will lead to a decrease on its precipitation.

The release of P in the digester results from the organic matter degradation and from Poly-P hydrolysis. The organic matter degradation process that takes place in the anaerobic digester is a consequence of the sludge stabilisation and cannot be reduced. However, the release of P from Poly-P hydrolysis in the digester can be reduced by enhancing its release in a previous stage before digestion.

Therefore, the sludge line operation must be designed taking into account this objective. Previous studies (Martí et al., 2008b, Barat et al., 2009c) have shown that the mixing chamber combined by elutriation of mixed sludge in the primary thickening is an effective management of the sludge line to extract a high P concentration before digestion (See Figure 2). The primary thickener supernatant stream will result in a P-rich stream that can be fed to the crystallizer. With this aim, good operational practices in each stage of the sludge line are described next.
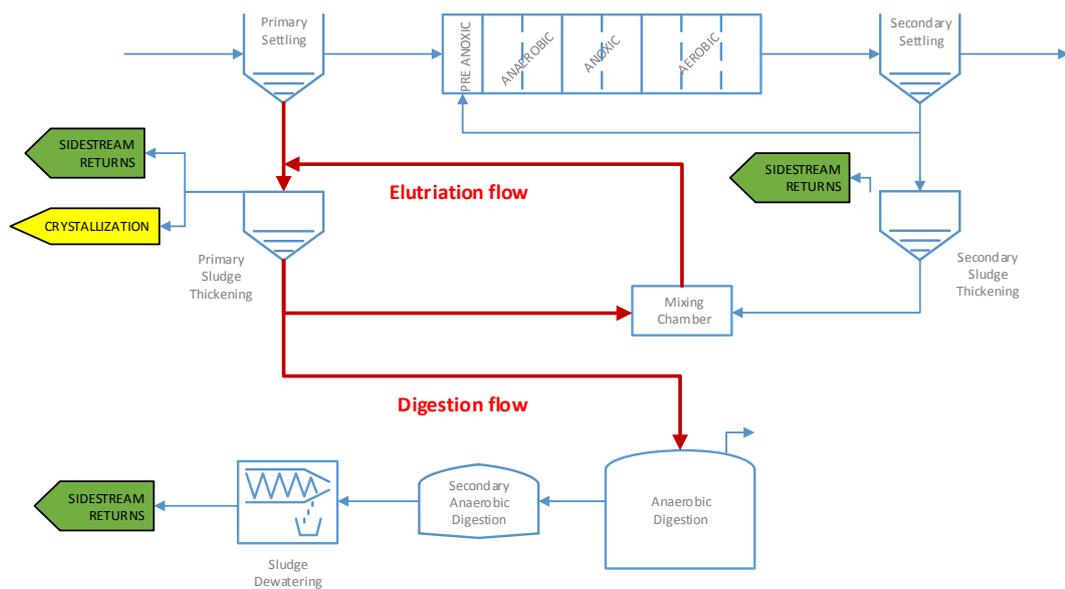

Figure 2. Sludge line configuration to maximise P recovery in WWTP

Mixed sludge thickening in the primary thickener

The thickening and elutriation of the mixed sludge only will be possible if the primary thickener is oversized.

In the case of mechanically mixed anaerobic digesters, special attention must be paid to the total solids concentration in the thickened sludge in order to avoid an anomalous performance of the agitator.

The control of the sludge blanket in the primary thickener is a key operation parameter to avoid a high solid loss in the supernatant stream.

Secondary sludge thickening

As explained before, both gravity and flotation thickening can lead to a significant P release if anaerobic conditions are achieved inside the secondary thickener. The release of P in this operation unit is only interesting if the supernatant obtained is able to be used in the crystallizer. Otherwise, the secondary sludge thickening must be operated at low residence times in order to avoid the release of P from Poly-P hydrolysis at this stage. If mechanical thickening is performed, no P release is expected since the sludge residence time is usually low.

Mixing chamber

In this operation unit, the presence of significant VFA concentrations from primary sludge and the anaerobic conditions achieved enhance Poly-P hydrolysis stored by PAO. In order to favour the contact between primary and secondary sludge enough residence time in the mixing chamber must be assured.

The elutriation of the mixed sludge (See Figure 2) in the primary thickener makes it possible to extract in the supernatant stream the soluble P that can be recovered as struvite by crystallization.

Anaerobic digester and post-digestion processes

Despite having extracted a significant amount of P before digestion, precipitation of several phosphate salts can take place in the anaerobic digester due to the P release from organic matter degradation and due to the pH increase.

Also, during post-digestion processes pH can still increase favouring the precipitation of inorganic compounds. In order to control this precipitation in the digestion and post-digestion systems the use of some smooth materials such as PVC instead of steel is strongly recommended (Doyle et al., 2002). Finally, sinuous pipe scheme must be avoided since can favour further precipitation.

It must be highlighted that the supernatant obtained in the digested sludge dewatering could be used in the crystallization process.

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