Chapter VII

DIGESTATE TREATMENT Phosphorus removal

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Introduction

Phosphorus is one of the main constituents of fertilizers used in agriculture despite the environmental problems arising from its presence at high concentrations. Most of the used phosphorus comes from natural deposits found in phosphate rock mines. However, these deposits are located only in some regions of the planet. The demand for phosphate fertilizers in Brazil has increased significantly and currently, around 60% of the used inorganic P is imported due to the large increase in agricultural areas in recent decades (Associação Nacional para Difusão de Adubos, 2018).

Direct digestate application to the soil can be performed to take advantage of the fertilizer potential of this fraction, which is rich in nitrogen, phosphorus, and potassium. Bachmann et al. (2016) compared the use of digestate with untreated effluent and commercial fertilizer and found a higher P absorption by plants (corn, amaranth, and sorghum) from the digestate than from other fertilizers. However, the cost

involved with transporting the digestate to the farm makes its direct use difficult. Thus, the conversion of this nutrient into a solid form followed by separation from the digestate can be advantageous because it significantly reduces the volume to be transported. In this sense, phosphorus removal from the digestate for later use as a fertilizer, called second-generation fertilizer, is a sustainable solution (Withers et al., 2018).

The phosphorus removal processes can be classified into chemical, physical, and biological. The removed phosphorus is converted into a solid fraction in almost all processes, and the purity in P content depends on the process and the treated effluent composition.

The main processes used to remove phosphorus from digestate are summarized in Table 1, which compares their main characteristics.

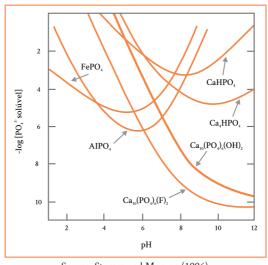
Table 1. Comparison of the main processes used to remove phosphorus from digestate.

Treatment	Optimal pH	Advantages	Disadvantages
Crystallization			
Quicklime	> 9.0	 Relatively cheap Allows P recovery for use as fertilizer or in industry Allows inactivation of pathogens (Viancelli et al., 2015) 	 Requires high pH Additional neutralization step may be required Relatively high generated sludge volume
Struvite	7.5 – 9.0	• Simultaneous removal of N and P	 Mg addition is usually required
Fe	> 7.0	Relatively cheapEffective in P precipitation	Precipitate unsuitable for use as a fertilizer
Al	> 6.3	 Effective in P precipitation Biological process can be associated with Al precipitation at pH 6 	Expensive process Precipitate unsuitable for use as a fertilizer
Physical processes			
		Good P removal efficiency	 Use as little studied fertilizer Expensive process
EBPR			
Enhanced Biological Phosphorus Removal		 Used associated with crystallization processes (struvite and quicklime) Sustainable process Requires little or no addition of chemicals 	 Requires higher control of operating conditions compared to other processes Complexity of facilities Requires more physical space

Chemical processes are most suitable for phosphorus removal and recovery from the digestate due to their low cost, ease of setup and operation, and high efficiency (Peng et al., 2018). The most used consist of precipitation processes in the form of calcium phosphate and struvite. Some precipitation processes use iron and aluminum salts (Raptopoulou et al., 2016). Biological processes, also known as EBPR (Enhanced Biological Phosphorus Removal), are the most suitable for phosphorus recovery from the point of view of process sustainability, although the higher complexity and operational difficulties make their use difficult (Enhanced..., 2005). In general, physical processes present a good phosphorus removal efficiency although process costs are usually high.

Phosphorus removal by chemical processes

Most of the phosphorus present in the digestate is in the chemical form of soluble orthophosphate. Orthophosphates form insoluble compounds with metals such as calcium, magnesium, iron, and aluminum, allowing crystallization reactions to occur in water. These crystallization reactions are pH-dependent, as shown in Figure 1 (Stumm and Morgan, 1996). Thus, the pH adjustment of the process is essential to obtain higher P removal efficiencies.



Source: Stumm and Morgan (1996).

Figura 1. Diagram of the solubility of Fe, Al, and Ca phosphates as a function of pH.

The main crystallization processes used to remove phosphorus from digestate consist of the reaction with calcium at alkaline pH and formation of struvite, according to the following reactions:

$$5Ca^{2+} + 3HPO_{4^{2-}}OH^- \rightarrow Ca_5(PO_4)_3OH$$
 Equation 1
$$Ca^{2+} + HPO_{4^{2-}} \rightarrow CAHPO_4 \qquad \text{Equation 2}$$

$$3Ca^{2+} + 2PO_{4^{3-}} \rightarrow Ca_3(PO_4)_2 \qquad \text{Equation 3}$$

$$Mg^{2+} + H_nPO_{4^{n-3}} + NH_{4^+} + 6H_2O \rightarrow MgNH_4PO_4.6H_2O + nH^+$$
Equation 4

Where:

n = 1, 2, 3, etc. corresponds to the solution pH

Phosphorus removal through precipitation with Ca

Phosphorus removal with calcium compounds is a relatively lowcost process that can be implemented without much difficulty using slaked lime as a calcium supply. In addition, the sludge generated has potential use as a fertilizer (Melia et al., 2017). In addition to removing P, the use of slaked lime also works to clarify the effluent if it has particulate material capable of coagulation/flocculation.

The treatment with slaked lime consists of adding a volume of Ca(OH), solution necessary for P (orthophosphates) precipitation. According to Fernandes et al. (2012), the phosphorus removal efficiency is higher than 90% with the addition of Ca(OH), solution up to pH 8.5 and higher than 96% at pH 9. In this study, the treatment with slaked lime was applied to the effluent that underwent an anaerobic digestion process in a UASB reactor, followed by treatment in a nitrification reactor. The treatment with the addition of slaked lime is advantageous, as it allows satisfactory phosphorus removal, and the pH of the final effluent does not need to be adjusted. This effluent has characteristics that enable its use for cleaning of facilities and irrigation (reuse water), for example.

Another advantage of using precipitation with slaked lime for phosphorus removal is the inactivation of pathogens, which occurs due to an increase in pH. This effect was demonstrated by Viancelli et al (2015), who observed total inactivation of *E. coli*, *Salmonella*, and *P. circo*virus type 2 at pH 10.

Factors that interfere with the phosphorus removal process with slaked lime

Considering that P is in the form of orthophosphate and according to the reaction below:

$$3Ca(OH)_2 + 2PO_{4^{3-}} \rightarrow Ca_3(PO_4)_{2(s)} + 6(OH)^-$$
 Equation 5

Three moles of Ca2+ are required for two moles of PO4-, that is, a Ca/P ratio of 1.5. The reaction of Ca with PO₄ 3- can lead to the formation of several compounds, including hydroxyapatite [Ca10(PO₄)₆(OH)₂], tricalcium phosphate [Ca₃(PO₄)₂], octacalcium phosphate [Ca₈(HPO₄)₂(PO₄)₄.5H₂O], dicalcium phosphate [CaHPO₄], and calcium hydrogen phosphate dihydrate [CaHPO₄.2H₂O], in increasing order of solubility. In addition to the formation of these compounds with a crystalline structure, an amorphous compound known as amorphous calcium phosphate, with an approximate formula of Ca₃(PO₄)₂. xH₂O, similar to tricalcium phosphate, can be formed. The Ca/P molar ratio ranges from 1.3 to 2 for all these compounds. However, the digestate usually has high alkalinity mainly due to carbonate and bicarbonate ions. These ions also react with Ca2+, as shown in the following reactions, causing a higher consumption of slaked lime than calculated by the Ca/P molar ratio:

$$CO_3^{2-} + Ca(OH)_2 \rightarrow CaCO_3 + 2(OH)^-$$
 Equation 6
$$CaHCO_{3-} + Ca(OH)_2 \rightarrow 2CaCO_3 + 2H_2O$$
 Equation 7

The solubility product constant (k_{sp}) of the CaCO₃ precipitation reaction ($k_{sp} = 3.36 \times 10^{-9}$) is higher than the ksp of the $Ca_3(PO_4)_2$ precipitation reaction ($k_{sp} = 2.07 \times 10^{-33}$), that is, under this aspect, it could be assumed that the carbonate ion precipitation would only occur after the total precipitation of PO₄³⁻. However, it is known that the reaction between Ca(OH), and bicarbonate is complete above pH 9.5 while the reaction between $Ca(OH)_2$ and PO_4^{3-} starts above pH 7 and is very slow below pH 9. Therefore, the precipitation of carbonate and bicarbonate ions occurs concomitantly since the addition of quicklime up to a pH higher than 9 is necessary for treatments that use slaked lime to remove P.

The presence of ammonia, which also occurs in the digestate, is another factor that causes an increase in Ca(OH), consumption. NH4+ ions react with Ca(OH), according to the reaction:

$$Ca(OH)_2 + 2NH_4^+ \rightarrow 2NH_3 + Ca^{2+} + 2H_2O$$
 Equation 8

Thus, a larger amount of quicklime is needed to raise the pH until the complete precipitation reaction of orthophosphates (Szogi; Vanotti, 2009).

Furthermore, a high carbon concentration in the digestate causes co-precipitation of part of the organic matter, reducing the purity of sludge and, consequently, its added value for reuse as fertilizer or other products.

Considering the interferences of alkalinity, ammonia, and organic matter, the process of removing phosphorus from the digestate using precipitation with slaked lime must be implemented after an ammoniacal nitrogen removal process, especially if the alkalinity consumption also occurs in this process (Vanotti et al., 2003).

Phosphorus removal through struvite formation

Phosphorus precipitation in the form of struvite occurs when concentrations of the chemical species Mg2+, NH4+, and PO45- exist in a 1:1:1 molar ratio and exceed the solubility product constant, respectively (Peng et al., 2018). Struvite crystallization has a low impurity con-

tent, which is important for phosphorus recovery and reuse (Zhou et al., 2015). This process occurs when the PO₄³⁻ concentration is between 100 mg.L⁻¹ and 200 mg.L⁻¹ and pH above 7.5. As can be observed, simultaneous phosphorus and ammonia removal occur in this case, which may be advantageous for some types of effluents.

$$Mg^{2+} + NH_4^+ + PO_4^{3-} + 6H_2O \rightarrow MgNH_4PO_4.H_2O_{(s)}$$

Equation 9

The Mg²⁺ concentration in most effluents is lower than necessary for struvite crystallization and, therefore, its addition is necessary, which is normally carried out by adding magnesium chloride.

Struvite can also precipitate undesirably in the lines that transport the digestate, which can lead to their obstruction, as shown in Figure 12 of Chapter III. To prevent this from happening, it is not recommended that the effluent remain stationary in the transport line. Washing it in an acidic medium (e.g., muriatic acid) will contribute to its solubilization and subsequent unblocking.

Phosphorus removal through crystallization with Fe and Al ions

 $\mathrm{Fe^{3+}}$ and $\mathrm{Al^{3+}}$ ions react with phosphate to form $\mathrm{FePO_4}$ and $\mathrm{AlPO_4}$, as shown in the following equation:

$$X^{3+} + H_n PO_4^{3-} \to XPO_{4(s)} + nH^+$$
 Equation 10

Where:

 $X = Al^{3+} \text{ or } Fe^{3+}$

Iron and aluminum compounds are widely used in wastewater treatment due to their properties as flocculants for particulate material removal by coagulation/flocculation. Regarding phosphates, the phase separation after the precipitation reaction is performed after flocculation and sedimentation. The phosphorus removal efficiency obtained with the use of Fe or Al is considered high. Usually, iron or aluminum sulfate or chloride are used. Another advantage of using Fe and Al in phosphorus removal is the wide application range since precipitation occurs at any phosphorus concentration range, different from what occurs in the struvite formation process.

Fe³⁺ has been used due to its lower cost. The ideal pH for the use of Fe³⁺ ions is above 7. The colloidal characteristic of FePO₄ requires an excess of Fe³⁺ for the colloidal precipitate formation which, in turn, will aggregate other FePO₄ colloids and adsorb other chemical P species (Loehr et al., 1973).

Satisfactory results have been reported up to pH 7.5 when using Al3+ although the ideal pH for the AlPO4 formation reaction is around 6.3, which in many cases avoids the need to adjust the pH of the digestate before the treatment.

The main disadvantage of phosphorus precipitation with Fe or Al is related to the possibility of phosphorus reuse. The formed compounds are not suitable for use as a fertilizer, as they prevent the solubilization of phosphorus in the soil, making it unavailable to plants. The use of tannin, a natural polymer, as a coagulation aid, can increase the sedimentation rate of precipitates and also enable the reuse of the generated sludge as fertilizer (Zhou et al., 2008).

Types of reactors used in chemical phosphorus removal processes

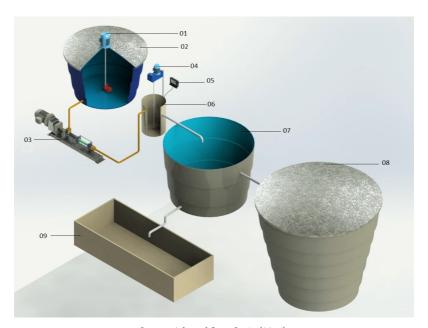
The crystallization reactions of orthophosphate with metal ions occur quickly and hence fast-mix, continuous, or batch reactors, equipped with a stirring system, are normally used.

The mixing time is greatly reduced depending on the reaction speed, but a complete homogenization needs to be ensured. Therefore, a hydraulic retention time of one minute is sufficient for the mixture between the effluent to be treated and the Ca(OH), solution to be carried out.

The added reagent volume for crystallization with slaked lime can be determined by controlling the pH (Fernandes et al., 2012; Vanotti et al., 2009), that is, crystallization will occur keeping the pH above 9 and, consequently, precipitation.

Although pH is the main control parameter, Suzin (2016) demonstrated that the use of 7.3 mL.L⁻¹ of a 10% (m/v) Ca(OH), solution in effluent from a nitrifying reactor with alkalinity below 300 mg.L⁻¹ of CaCO, allowed maintaining phosphorus removal above 90%. Therefore, the injection of 8 mL of solution per liter of effluent to be treated can be safely indicated for effluents with low alkalinity and ammonia.

The separation of phases (sludge and supernatant) can be conducted in a slow mixing unit or even a settler, which can be installed after the fast mixing unit, as shown in Figure 2. Studies have shown that three hours of hydraulic retention time at this stage are enough for phosphorus precipitation and elimination of several pathogens that may be present in the effluent (Viancelli et al., 2015; Suzin, 2016).



Source: Adapted from Suzin (2016).

Figure 2. Representation of a phosphorus removal system. 1 and 4: mechanical stirrer; 2: tank for preparation of Ca(OH), solution; 3: Ca(OH), metering pump; 5: pH controller; 6: quick mixing unit; 7: settler; 8: reuse water reservoir; 9: sludge drying bed.

Case study: dimensioning of a phosphorus removal system by chemical precipitation

A covered lagoon digester (CLD) is fed with a flow of 220 m³·d⁻¹ and 8 m³.sludge.d⁻¹ is discarded. The digestate is treated by the nitrification/denitrification process, from which 6 m3.sludge.d-1 is discarded. The effluent from the nitrogen removal process should be sent to a phosphorus removal system by chemical precipitation through the calcium hydroxide (Ca(OH), addition. Considering that the CLD has a total phosphorus removal efficiency of 86% and nitrification/denitrification has a phosphorus removal efficiency of 75%, dimension a phosphorus removal system as shown in Figure 3. Assume that the total phosphorus concentration in the CLD feed is 1.33 g.L⁻¹ and the alkalinity and ammoniacal nitrogen concentrations at the outlet of the nitrification/ denitrification system are negligible. Calculate the demand for Ca(OH), (assuming 90% purity) in kg.d⁻¹ so that the phosphorus removal efficiency is 90% in the phosphorus removal module.

Initially, the concentration of total phosphorus (P) at the outlet of the nitrification/denitrification system needs to be determined. Therefore, the phosphorus concentration at the digester outlet is estimated considering that 86% of the P is naturally transferred to the CLD sludge.

$$[P]_{outlet-dig} = [P]_{inlet-dig} - ([P]_{inlet-dig} \cdot Ef_{Premoval-dig})$$

$$[P]_{outlet-dig} = 1.33 \frac{g}{L} - \left(1.33 \frac{g}{L} \cdot 0.86\right)$$

$$[P]_{outlet-dig} = 0.186 \frac{g}{L} = 0.186 \frac{kg}{m^3}$$

$$Equation 11$$

Considering that the total phosphorus concentration at the digester outlet is equal to the concentration on the inlet of the nitrification/ denitrification system.

$$[P]_{outlet\text{-}dig} = [P]_{inlet\text{-}nit\text{-}denit}$$
 Equation 12
 $[P]_{inlet\text{-}nit\text{-}denit} = 0.186 \frac{\text{kg}}{\text{m}^3}$

The concentration at the outlet of the nitrification/denitrification system is calculated considering a total phosphorus removal efficiency of 75% in the nitrification/denitrification system.

$$[P]_{outlet-nit-denit} = [P]_{inlet-nit-denit} - ([P]_{inlet-nit-denit} \cdot Ef_{P removal-nit-denit})$$

$$Equation 13$$

$$[P]_{outlet-nit-denit} = 0.186 \frac{kg}{m^3} - \left(0.186 \frac{kg}{m^3}.0.75\right)$$
$$[P]_{outlet-nit-denit} = 0.0466 \frac{kg}{m^3}.1000$$
$$[P]_{outlet-nit-denit} = 46.6 \frac{mg}{L}$$

The final concentration of total phosphorus can be estimated from the total phosphorus concentration at the outlet of the nitrification/denitrification system, based on the phosphorus removal efficiency of the removal system of 90%.

$$[P]_{final} = 46.6 \frac{mg}{L} - \left(46.6 \frac{mg}{L}.0.90\right)$$
$$[P]_{final} = 4.66 \frac{mg}{L}$$

The calculation of the quick mixing unit and settler volumes is carried out by the outflow of the nitrification/denitrification system and HRT of one minute for the quick mixing unit and six hours for the settler. For this, the outflow of the nitrification/denitrification system needs to be known.

$$Q_{outlet-dig} = Q_{inlet-dig} - Q_{sludge\ disposal-dig}$$

Equation 14

$$Q_{outlet\text{-dig}} = 220 \frac{m^3}{d} - 8 \frac{m^3}{d}$$

$$Q_{outlet\text{-dig}} = 212 \frac{m^3}{d}$$

 $Q_{inlet-nit-denit} = Q_{outlet-dig}$

$$Q_{outlet\text{-}nit\text{-}denit} = Q_{inlet\text{-}nit\text{-}denit} \ - \ Q_{sludge\ disposal\ nit\text{-}denit}$$

$$Q_{outlet\text{-nit-denit}} = 212 \frac{m^3}{d} - 6 \frac{m^3}{d}$$
$$Q_{outlet\text{-nit-denit}} = 206 \frac{m^3}{d}$$

 $Q_{outlet\text{-}nit\text{-}denit} = \ Q_{inlet\text{-}P\ removal}$

Quick mixing unit volume $(m^3) = Q_{inlet-P removal}$. $HRT_{quick \ mixing}$

Equation 15

Quick mixing unit volume
$$(m^3) = 206 \frac{m^3}{d}$$
. 1 min

Quick mixing unit volume (m³) =
$$206 \frac{m^3}{d}$$
 . min . $\frac{1 \ d}{1440 \ min}$. $\frac{1000 \ L}{m^3}$

Quick mixing unit volume $(m^3) = 143 L$

Settler volume
$$(m^3) = Q_{inlet-P removal}$$
. $HRT_{settler}$

Equation 16

Settler volume
$$(m^3) = 206 \frac{m^3}{d} \cdot 6 \text{ h} \cdot \frac{1 \text{ d}}{24 \text{ h}}$$

Settler volume $(m^3) = 51.5 \text{ m}^3$

The dosage of slaked lime is a function of the stoichiometry of the reaction between Ca²⁺ and PO₄³⁻ ions to form Ca₃(PO₄)₂. Thus, 3 moles of Ca^{2+} are required to precipitate 2 moles of PO_4^{3-} . Thus, 3 moles of $Ca(OH)_2 = 222$ g and 2 moles of P = 62 g, considering the atomic masses of these chemical species and that we are working with P concentration and not with the orthophosphate ion. The Ca(OH), dosage at 10% (m/v) (equivalent to 100 g.L⁻¹) can be calculated:

$$Ca(OH)_2 \, dosage \, \left(\frac{L}{m^3}\right) = \, \left(\frac{\frac{mass \, 3 \, moles_{Ca(OH)_2}. \, \, [P_{effluent}]}{mass \, 2 \, moles_p}}{100 \, \frac{g}{L}}\right). \, 1000 \, L$$

Equation 17

$$\mbox{Ca(OH)}_2 \mbox{ dosage} \left(\frac{L}{m^3} \right) = \left(\frac{\frac{222 \mbox{ g. } 0.0466 \mbox{ g}}{62 \mbox{ g}}}{100 \mbox{ g}} \right) . \mbox{ 1000 L}$$

$$\mbox{Ca(OH)}_2 \mbox{ dosage} \left(\frac{L}{m^3} \right) = \mbox{ 1.7 } \frac{L}{m^3}$$

The quicklime dosage allows calculating the daily requirement for Ca(OH), considering the Ca(OH), purity of 90%.

$$\textit{Daily Ca(OH)}_2 \ \textit{requirement}\left(\frac{\textit{kg}}{\textit{d}}\right) = \frac{\textit{Q}_{\textit{inlet-P}\ \textit{removal}} \cdot [\textit{solution}] \cdot \textit{dosage}}{\textit{purity}}$$

Equation 18

$$\begin{split} \text{Daily Ca(OH)}_2 \text{ requirement} \left(\frac{kg}{d}\right) &= \frac{206 \frac{m^3}{d} \cdot 10\% \cdot 1.7 \ \frac{L}{m^3}}{0.9} \\ \text{Daily Ca(OH)}_2 \text{ requirement} \left(\frac{kg}{d}\right) &= \frac{206 \frac{m^3}{d} \cdot 100 \ \frac{kg}{m^3} \cdot 1.7 \frac{L}{m^3} \cdot \frac{m^3}{1000 \ L}}{0.9} \\ \text{Daily Ca(OH)}_2 \text{ requirement} \left(\frac{kg}{d}\right) &= 38.91 \frac{kg}{d} \end{split}$$

Phosphorus removal by physical processes

Some physical processes have been studied for phosphorus removal. The main ones consist of the use of membranes (Bolzonella et al., 2018), electrodialysis, and adsorption processes (Kunaschk et al., 2015). However, their high costs still limit the full-scale application despite the good efficiency in phosphorus removal.

Phosphorus capture and recovery with biochar have also been studied (Shepherd et al., 2017). In this case, phosphorus adsorption occurs through the interaction with Fe or Mg. There is the possibility of reusing the adsorbed material, but no information on the phosphorus bioavailability can be found in the literature.

Bolzonella et al. (2018) compared different nutrient recovery systems (P and N) used on a commercial scale in northern Italy. These systems combine physical and chemical processes: drying followed by acid recovery, stripping followed by acidic recovery, and membrane separation. The authors observed that the use of membranes allowed obtaining a high purity liquid effluent (reuse water) although it is the treatment with the highest cost among those studied. The digestate in the treatment with membranes and stripping initially undergoes a centrifugation process and most of the phosphorus remains in the solid fraction at this stage. The solid residue undergoes an acid treatment to recover nitrogen in the form of ammonium sulfate in the drying and stripping processes. Moreover, the digestate is subjected to a water evaporation process at drying.

Phosphorus removal by biological processes

Biological phosphorus removal is performed by microorganisms or some types of aquatic plants. The process known as EBP has been used to recover phosphorus, mainly in sanitary sewage effluents. This process consists of the intracellular bioaccumulation of polyphosphates by phosphate-accumulating organisms (assimilation and disassimilation processes) under aerobic and anaerobic conditions. In the anaerobic phase, bacteria assimilate volatile fatty acids, which are stored as polyhydroxyalkanoates and then metabolized in the aerobic phase to provide

the energy needed for the phosphate accumulation process. In addition to bacteria, microalgae have also been investigated for their phosphate assimilation capacity (Melia et al., 2017). The EBPR process has also been used to concentrate phosphorus in the sludge for further chemical treatment for struvite crystallization, in the case of effluents with lower phosphorus concentrations. P concentration must be between 100 and 200 mg.L⁻¹ for struvite crystallization.

Phosphate-accumulating microorganisms are heterotrophic and, therefore, a sufficient amount of bioavailable carbon is required for the operational success of a treatment system that uses the EBPR process. The availability of an electron acceptor, which can be nitrate or oxygen, is another determining factor for the process. Furthermore, the presence of cations, such as magnesium and potassium, is also important to facilitate phosphate assimilation and disassimilation by microorganisms. Phosphorus is released together with cations of magnesium and potassium in the anaerobic phase, while the assimilation of these chemical species occurs in the aerobic phase

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