Apatite as an interesting seed to remove phosphorus from wastewater in constructed wetlands

P. Molle*, A. Liénard*, A. Grasmick**, A. Iwema*** and A. Kabbabi****

*Cemagref, Research unit: Water Quality and Pollution Prevention, 3bis, quai Chauveau,CP 220, 69336 Lyon Cedex 09,France (E-mail: *pascal.molle@cemagref.fr; alain.lienard@cemagref.fr*)

**Laboratoire de Génie des Procédés, UMR Cirad 016, CC024 Université de Montpellier II, Place E. Bataillon, 34095 Montpellier Cedex 05, France (E-mail: *grasmick@univ-montp2.fr*)

***Agence de l'Eau Rhône Méditerranée Corse, 2-4 allée de Lodz, 69363 Lyon Cedex 07, France (E-mail: *Arthur.IWEMA@eaurmc.fr*)

****Laboratoire de Recherche en Eau et Environnement, CERPHOS (Centre d'Etudes et de Recherche des Phosphates Minéraux); 73, Boulevard Moulay Ismaïl, Roches Noires, 20300 Casablanca, Morocco (E-mail: *a.kabbabi@cerphos.com*)

Abstract Intensive use of phosphates has resulted in high P levels in surface waters and therefore eutrophication problems. Over the last decade many studies have revealed the advantage of using specific materials with efficient phosphorus retention capacities. Recent studies state that Ca materials are of particular interest for long-term retention of P, but can induce negative effects. To improve P retention and avoid negative counter-effects we tested the potential of natural apatites. Apatite sorption was evaluated using batch and open reactor experiments. Batch experiments identify sorption mechanisms and the influence of the ionic characteristics of the solution; open reactor experiments evaluate sorption capacities in relation to the ionic composition of the solution and biomass development. In parallel, observation of the material by electron microscopy was used to give more precision information about the mechanisms involved. This work reveals the strong chemical affinity between apatites and phosphorus. Compared to other calcareous materials apatite is better able to maintain low outlet P levels. After more than 550 days feeding, sorption was still present and low P outlet levels were still being obtained when sufficient contact time and calcium content in the solution were ensured. This work demonstrates the advantages of using apatites for phosphorus removal in constructed wetlands. The behaviour of apatite in phosphorus retention is explained and its suitability for use in such extensive systems defined.
Keywords Phosphorus retention; constructed wetlands; apatite; surface precipitation

Introduction

Intensive use of phosphates (agricultural use, food, detergents etc.), has resulted in high P levels in surface waters and therefore to eutrophication problems. Consequently P removal from municipal wastewater is of great interest and is required by European quality objectives (Urban Waste Water Treatment Directive 91/271/EEC and Water Framework Directive 2000/60/EC). In constructed wetlands (CW), P removal is mainly due to plant assimilation and biomass incorporation. However, P returns to the mobile phase during biomass death and organic matter mineralisation. Moreover, plant assimilation is small compared to the loads applied (Brix, 1997). Another way to retain phosphorus in constructed wetlands is to promote irreversible retention on a solid phase by adsorption and precipitation. The aim is to use a specific material with good retention capacities as a media. The importance of using Al, Fe and Ca materials to retain phosphorus is well known thanks to many agricultural studies on the efficiency of fertilizers in relation to soil characteristics (Barrow, 1987). Consequently, over a decade many studies have explored this way of retention to demonstrate the advantages of specific materials for long-term P retention, and so to guarantee the extensive nature of CW treatment systems (Zhu *et al.*, 1997; Brix *et al.*, 2000; Drizo *et al.*, 2002; Del Bubba *et al.*, 2003).

193

P. Molle et al.

The importance of each sorption mechanism (adsorption, precipitation) for long-term removal must be determined to predict P retention efficiency according to the characteristics of the influent. Adsorption will depend on the intrinsic characteristics of the material and its equilibrium with the solution. Isomorphic substitution of the lattice surface, and proton adsorption and desorption at the functional surface site, result in a surface charge that will permit adsorption or not depending on the pH of the solution. It is therefore possible to determine a pH of zero point of charge (pH_{ZPC}) below which phosphate adsorption will occur and above which it will not. Further adsorption types exist with different retention stabilities. The aim here is not to describe each one. For that the reader can refer to more complete studies (Sposito, 1984; McBride, 1994; Stum and Morgan, 1996). Precipitation requires the formation of stable nuclei, after which crystal growth can take place. In solution some degree of supersaturation is necessary because crystal nuclei can only be formed after an energy barrier has been overcome (Anderson and Rubin 1981). In soil, surface precipitation is much more likely to occur than homogenous precipitation because particles can catalyse the nucleation step of crystallisation by reducing the energy barrier that must be overcome to allow nucleation (Sposito, 1984). Nevertheless adsorption and precipitation of phosphorus in soil are complex mechanisms, and distinguishing between the two is not easy when one realizes that similar chemical bonds could be involved and both are surface reactions.

Very often P sorption capacities have been studied by batch and column experiments using distilled water which could introduce uncertainties in material behaviour because the chemical composition of water is of great importance in sorption mechanisms (Molle et al., 2003a). Many studies demonstrate efficient retention capacities of calcareous materials (Brix et al., 2000; Del Bubba et al., 2003) but with some limitations in sorption kinetics or chemical effluent stability that can induce problems in respect to European regulations. In previous work (Molle et al., 2003a), mechanisms involved in long-term P removal on calcareous materials were determined to be surface precipitation dependent on chemical influent characteristics. Retention on natural calcite was not efficient enough to meet European targets because it is sensitive to carbonate equilibrium. The use of natural phosphate rock to remove P from wastewater could initially seem paradoxical but is theoretically assumed to lower the energy barrier and thus promote precipitation. It also an interesting way to concentrate P on natural phosphate rock to facilitate the reuse of diffuse P (fertilizer). Moreover natural apatites are formed in a very hydrated environment with low phosphorus concentration (sea water). Apatites are also used as a permeable reactive barrier for uranium sorption (Fuller et al., 2003; Krestou et al., 2004) and in various studies on trace element and metal removal from water or soil (Boisson et al., 1999; Chen et al., 1997). Consequently, it was decided to test natural apatite as a seed to remove phosphorus. This material has the advantage of being the most stable of the calcium phosphate precipitates.

The aim of this study was therefore to demonstrate, whether adsorption or precipitation is the predominant mechanism, the limitations of P sorption (in terms of kinetics and retention capacity), the stability of retention and the role of influent characteristics. The ultimate objective was to determine the appropriateness of apatite use for P removal in order to maintain the extensive feature of CW treatment systems.

Materials and methods

Phosphorus retention capacity of apatite was studied in batch and open reactors using different apatite particle sizes.

Characteristics of apatite

Particle-size distributions were determined using dry-sieving techniques (European norm, 1997), to calculate d_{10}, d_{60} (mesh diameter allowing, respectively, 10 or 60% of the material mass to pass through) and the uniformity coefficient ($UC = d_{60}/d_{10}$). Porosity was determined from the amount of water needed to saturate a known volume of component (replicate number $n = 3$) and the bulk density was measured by the volume of water displaced by a known mass of medium ($n = 3$). The specific area of particles was evaluated from the size distribution, assuming grains to be spherical. Chemical and physical properties of apatite are shown in Table 1. Surface observation by scanning electron microscopy (SEM) showed that apatite is composed of aggregated ovoid and cylindrical grains. Ca/P and Ca/F molar ratio analyses by SEM showed the precipitate to be fluorapatite. "Clinker" aggregation of ovoid grains is mainly composed of calcium carbonates which evokes the marine origin of apatite.

With a solution free from calcium or with tap water, a fast dissolution rate of apatite particles was observed leading to an increase of calcium, hydroxyl and carbonate ions in solution. A very little phosphorus is released during the reaction (P mass dissolved <1 per 100000, Ca/P released >100). The Ca/HCO$_3^-$ molar ratio of ions released in solution is close to that observed for calcite dissolution (Molle *et al.*, 2003a). Because of this and the fact that apatite is a very stable precipitate, it can be assumed that calcium carbonate impurities are mainly dissolved.

Batch experiments

Isotherm and kinetic batch experiments were carried out with a slow rotating shaker (25 rpm) at $22 \pm 3°C$. In each test 20 g of material was injected into a 1 L glass flask containing 500 ml of distilled water, pH (7.0 ± 0.1) and conductivity ($1000 \pm 100\,\mu S\,cm^{-1}$) were, respectively, regulated with addition of NaOH and NaCl. For isotherm experiments, P solutions ranged from 0 to $500\,mg\,P\,L^{-1}$ using KH$_2$PO$_4$ (contact time $= 24\,h$). In kinetic experiments contact time varied from 1 to 48 hours. Batch experiments were also focused on the characterisation of the P/apatite reaction mechanisms by varying electrolyte concentrations (Ca^{2+}, pH, HCO$_3^-$, P). Material dissolution was studied by putting apatite in contact with a solution free from orthophosphates. P was determined using a phosphate molybdate method (NF EN 1189) on the supernatant solution obtained from centrifugation (20 mn, 2000 g).

Column experiments

Phosphorus saturation was carried out on an 85 mm diameter column filled with $570\,cm^3$ of media to attain saturation within a reasonable period and kept in hydraulically saturated conditions. A synthetic solution (P $= 20\,mg\ L^{-1}$, pH $= 7.0 \pm 0.1$; conductivity $= 1000 \pm 100\,\mu S\,cm^{-1}$) was applied continuously at a flow rate of $1.35\,L.d^{-1}$ for 550 days to evaluate P saturation which corresponds to a hydraulic retention time of 5.6 h. Effluent was collected daily and analysed for P concentration; pH and conductivity were measured continuously. Three influent solutions were studied: firstly we used distilled water, then tap water and finally filtered treated wastewater, from

Table 1 Chemical and physical characteristics of apatite

	Chemical composition % (W/W) of materials					Particle size			Porosity %	Density kg m^{-3}	Specific area m^{-1}
	Ca	Mg	P	Fe	Al	d_{10} (mm)	d_{60} (mm)	UC			
Apatite 1	37.3	1.6	16.8	<0.1	<0.1	0.5	0.9	1.9	46	2480	3500
Apatite 2						0.1	0.2	2.0			15900

an activated sludge plant, mixed with 10% of filtered untreated wastewater ($60\,mg\,L^{-1}$ COD, $35\,mg\,L^{-1}$ N-NO_3^-). At the end of the experiment, columns were fed with a P-free solution to evaluate the reversibility of the sorption.

Analytical microscopy SEM and STEM

SEM as well as scanning transmission electron spectroscopy (STEM) were carried out to characterise the microstructure and composition of the apatite surface before and after column experiments. Surface crystals were broken down by ultrasonic washing and analysed by STEM to evaluate the composition of the deposit.

Isotherm study

Prediction of maximum adsorption capacity is often determined using isotherm data via Langmuir modelisation:

$$q = \frac{bKc}{1 + Kc} \tag{1}$$

where b and K are adjustable parameters: b represents the value of q (P mass adsorbed on material in $mg\,P\,g^{-1}$) that is approached asymptotically as c becomes arbitrarily large, and is often interpreted as the maximum sorption capacity. K determines the magnitude of the initial slope that represents the affinity between ion and mineral to react. A precise way to determine these parameters is to plot the ratio $K_d = q/c$ (distribution coefficient) against q. The Langmuir equation can be written in the linear expression:

$$K_d = bK - Kq. \tag{2}$$

Such an approach should be taken with care, as it could lead to unrealistic estimates for P adsorption capacities in CWs (Drizo *et al.*, 2000). Moreover, if surface precipitation is present, adsorption could be limited by the coverage of the surface by precipitates. Nevertheless, plotting K_d against saturation progression is an interesting way to observe the evolution of reaction affinity, and determine if one or more retention mechanisms are involved.

Results

Batch experiments

Isotherms. Isotherms obtained for each particle size are presented in Figure 1. No difference is observed in P sorption capacities after a 24 hour reaction time. A good fit was obtained with the Langmuir equation (Figure 1a, $r^2 = 0.97$). K and b parameters of the Langmuir equation are, respectively, 0.15 and $4.76\,mg\,g^{-1}$. To conclude that the adsorption mechanism is the only one present could be false. Indeed, the K_d variation (Figure 1b) does not show a linear correlation with q but a convex curve. Such curves are often represented by two series of Langmuir equations for a best fit. Above all it means that at least two mechanisms are involved in P sorption on apatite. Adsorption is predominant as long as the surface state is far from a saturated state. However, this phenomenon is inhibited when a definite degree of saturation is reached allowing precipitation to become the prevailing mechanism.

Kinetics. Retention rate (ret rate) shows that reaction with apatite is very fast in batch experiments (Figure 2). This can be put in relation with isotherm results: far from a saturation condition reaction is fast. Retention rate increases with both P concentration and contact surface. A slight change of pH and conductivity is observed in the same magnitude as was observed during isotherm studies.

Unlike in previous work on calcareous materials (Molle *et al.*, 2003a) with apatite, carbonates did not show any negative impact on P sorption (in terms of capacity
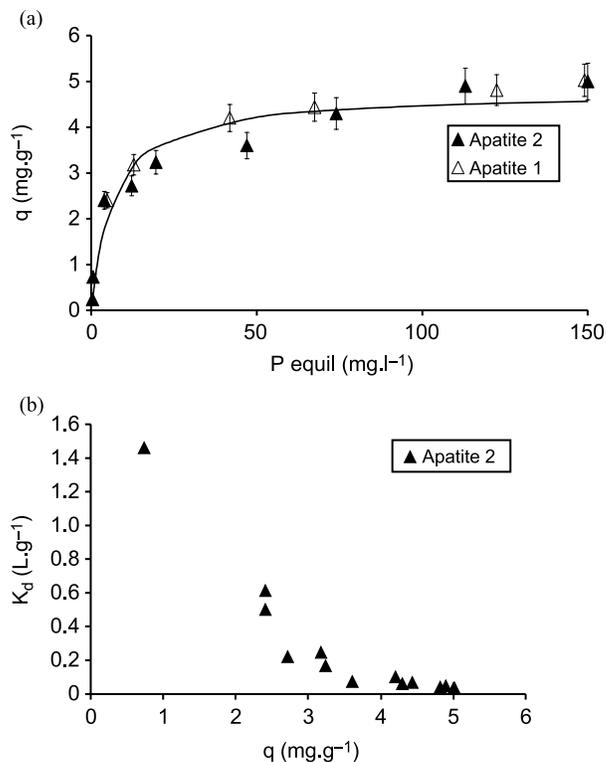
Figure 1 (a) P sorption isotherms; (b) K_d curve

and kinetics for carbonate concentrations from 0 to $350\,mg\,L^{-1}$). In the same way, no influence of calcium ions was observed on sorption capacity but only on the sorption kinetics: increasing molar Ca/P ratio in the initial solution allowed improvement in retention in the first hour of reaction. This is in accordance with work done by Song *et al.* (2001, 2002).

Column experiment

Apatite continuously fed with a phosphated solution in an open reactor shows a reasonable stability of pH and conductivity of the solution (see Table 2).

Four periods were noted in relation to the feeding solution.

- A first period of 150 days in which apatite was fed with distilled water, which allowed a partial dissolution of calcium carbonate impurities ($Ca_{outlet} = 5\,mg\,L^{-1}$). The outlet P concentration remained below $2\,mgP\,L^{-1}$, and saturation at the end of this period was about $4.9\,mgP\,g^{-1}$.
- A second period of about 200 days, also using distilled water, in which apatite dissolution was reduced and P retention became limited by the lack of calcium in solution. Retention rate decreased considerably and P outlet concentration rose to $14\,mg\,L^{-1}$.
- A third period of about 110 days in which apatite was fed with tap water. The presence of calcium (see Table 2) allowed an improvement in P retention and the P outlet concentration reached a level of less than $2\,mg\,L^{-1}$.
- A final period of 90 days in which the media was fed with treated wastewater mixed with 10% of wastewater as explained earlier. The presence of calcium still permitted efficient P retention and low outlet concentration levels. The impact of biomass development was not observable. Variations observed in retention rate were of the same order of magnitude as those measured with tap water with a similar change in ionic
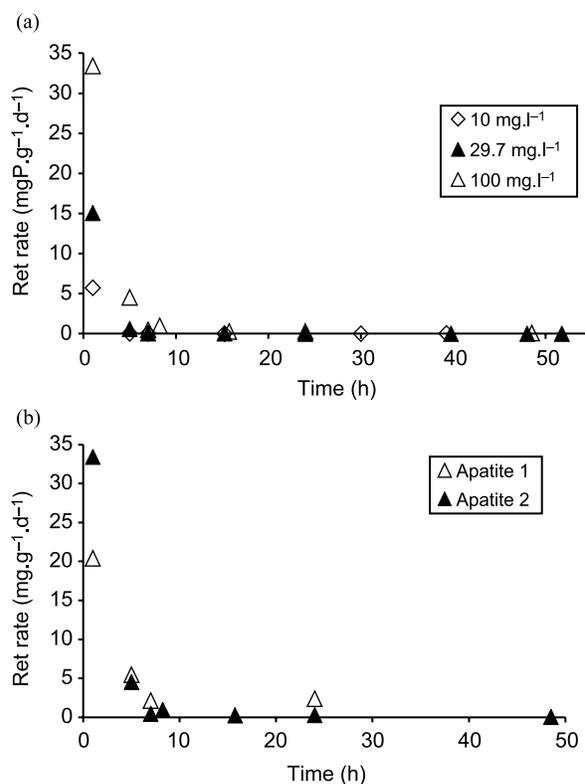
Figure 2 P sorption kinetics on apatite. (a) For different P concentrations, (b) For different material particle sizes

solution. Nevertheless, one would expect a poor biomass development over the 3 months of experiment with an inlet dissolved COD of between 30 and 70 mg L^{-1}.

At the end of the experiment P retention still occurred and no decrease in permeability due to chemical clogging was observed. After 550 days of operation saturation was about 13.9 mgP g^{-1}.

Analytical electronical microscopy

After 305 days of use in a column experiment, surface observation of apatites revealed that precipitates were formed. Ca/P precipitate ratios analysed by SEM (cf. Figure 4) and STEM were close to those for hydroxyapatite (1.67 by SEM and 1.84 (standard deviation 0.06) by STEM). No fluorine was found in these precipitates. At day 305, surface coverage appeared to be far from saturation while P outlet concentration was high (see Figure 3). This confirms that retention is limited by a decrease in kinetic reaction, due to an absence of calcium, and not by media saturation.

Discussion

Above experiments show that the identification of the prevailing mechanism involved in P removal and evolution of material potential to fix P when used in CWs, can be largely different according to experimental procedures. In batch experiments adsorption was seen to be the main mechanism involved in P removal. The ratio of P and Ca that have reacted, the equilibrium pH close to the pH_{ZPC} of apatite and the fact that Ca addition to solution did not improve retention capacity, confirm this hypothesis. Besides, the K_d curve progression in relation to material saturation indicates that, even if several mechanisms are present, adsorption is predominant at a low saturation state due to the great

Table 2 Physicochemical characteristics of solution

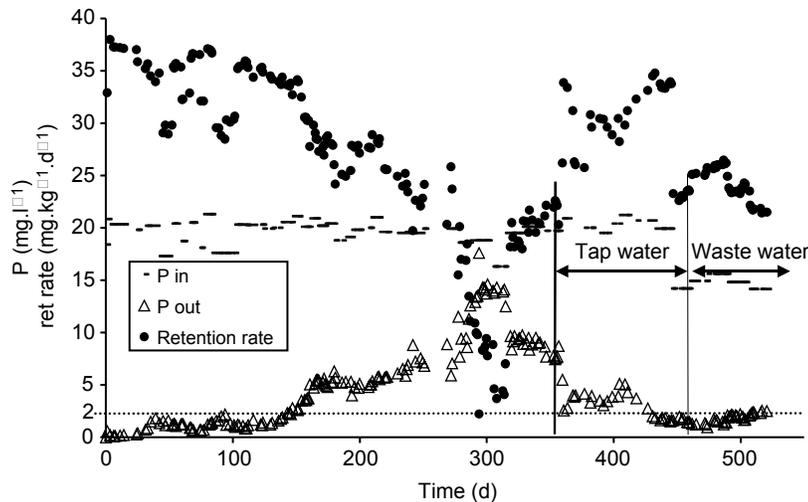|	pH		Conductivity ($\mu S\,cm^{-1}$)		Ca^{2+} ($mg\,L^{-1}$)	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Distilled water	7.0 ± 0.1	8.0 ± 0.2 (<8.0 after 150 d)	1000 ± 100	1000 ± 100	0	5 ± 2 ($n = 10$)
Tap water	7.0 ± 0.1	<8.0	1000 ± 100	1000 ± 100	55 ± 15 ($n = 10$)	45 ± 10 ($n = 10$)
Wastewater	$6.8 \Leftrightarrow 8.4$	<8.0	700 ± 100	700 ± 100	75 ± 15 ($n = 10$)	60 ± 10 ($n = 10$)

Figure 3 Breakthrough curve in column experiment

chemical affinity between P and the media. These tests are useful in defining the influence of solution characteristics on P retention. We observed that the presence of carbonates and calcium in solution did not appear to affect retention capacity, but kinetics were slightly improved by increasing calcium in the solution. Calcium is needed for P retention and, if not present in solution, reactions can occur after dissolution of calcium carbonate impurities. If calcium is present in solution reaction by complex adsorption can occur. Therefore, Ca addition increases reaction rate, but retention capacity appears to be limited by the number of functional sites in batch experiments.

In column experiments reactions were quite different due to different hydraulic conditions and reaction time. A 500-fold decrease in reaction rate was observed in comparison to batch experiments. This could be explained by a saturation front in the column which contributes to an underestimation of reaction rate (as observed in previous studies (Molle, 2003b)), but is mainly due to a different retention mechanism. In fact, surface microscopy observations revealed that precipitates were formed. Retention in the column was not governed by adsorption (for long-term removal) but by surface precipitation. Kinetics observed in batch experiments at near saturation ($q = 2.4 \, \text{mg g}^{-1}$ see Figure 1a), were only 5-fold greater than observed in column experiments for the same inlet concentration. This confirms the differences in P retention mechanism in relation to media saturation. It is of great importance to predict long-term behaviour of a filter: predictions cannot be made from batch experiments.
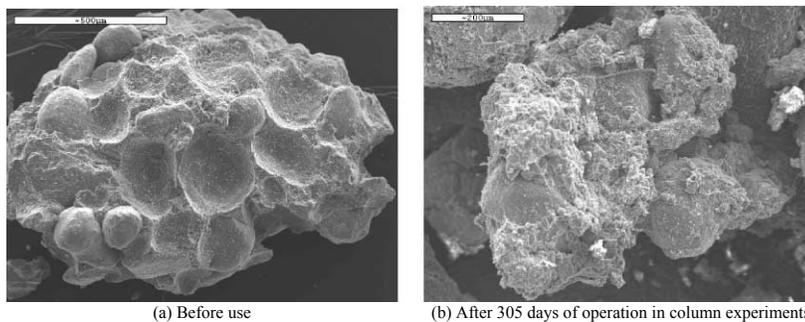
(a) Before use

(b) After 305 days of operation in column experiments

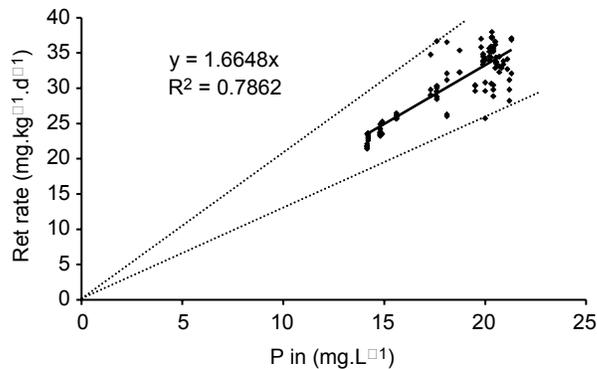**Figure 4** Low magnification SEM image of apatite

Figure 5 Surface precipitation kinetics for different P inlet concentration measured in column experiment when Ca is present

P. Molle et al.

This study aimed to demonstrate the potential of apatites as a suitable nucleation seed material in horizontal flow CWs. It can be observed firstly that apatites allow a relatively good stability of pH and conductivity of the solution. So the volume of material needed to allow a sufficient contact time in the filter to respect the $1\,mg\,P\,L^{-1}$ level in the effluent can be estimated assuming the following hypotheses: $3\,g\,P\,pe^{-1}\,d^{-1}$ and $150\,L\,pe^{-1}$ of wastewater per day (normal influent), a first-order reaction for P retention and an ideal plug flow water circulation throughout the system. However, we consider that P inlet concentration could change due to clean water intrusion into the network which would lead to a diminished contact time in the filter. These changes in concentration induce change in kinetic reaction, as presented in Figure 5, with extremes shown by dotted lines. Taking these hypotheses the volume of material needed would be about $0.6\,m^3\,pe^{-1}$ (Figure 6) for diluted influent and about $0.3\,m^3\,pe^{-1}$ for normal influent.

An essential question remains, how to know when surface crystallisation will be too great to modify water circulation through media because of chemical clogging or, in other words, how long can such a filter operate. Our study does not directly address this. However, we observed that media saturation at the end of the experiment was about $13.9\,mg\,P\,g^{-1}$ (taking the Ca in the precipitate into account there is less than 5% of material mass and it can therefore be ignored in terms of clogging), that retention still occurred and no permeability decrease was observed. This saturation state (more than 3 fold greater than the one observed in batch experiments) corresponds to a volume of $0.56\,m^3$ of material per p.e. for 10 years P retention.
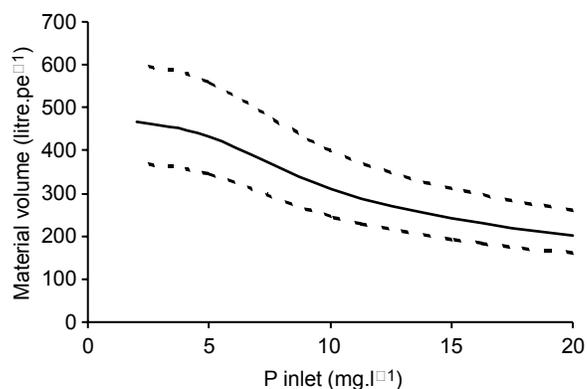

Figure 6 Estimation of volume need to respect $1\,mg\,P\,L^{-1}$ in the outlet concentration

201

Conclusions

This paper aims above all to analyse the potential of apatites to remove P from wastewater. Apatites allow a good pH stability of the solution and good kinetic crystallisation. Precipitate observation by SEM and STEM reveals the importance of using a seed close to the precipitate we want to form. The energy barrier for crystallisation is reduced and the precipitate seems to be hydroxyapatite, the most stable precipitate of calcium phosphate. The good potential of apatites, in terms of kinetics and saturation capacity, means that only a small volume of material is needed for long-term P removal. Of course more precise analysis in full scale experiments will need to be defined to verify:

- in what ways precipitation and pollutant sedimentation can affect the flow pattern and associated P removal efficiency.
- in what way a more developed biofilm could affect retention kinetics.

Nevertheless apatites appear to be attractive for use in CWs and the small volume necessary would mean that an apatite P removal filter would only need to be inserted in the last part of the horizontal flow CW thus limiting biomass development on the active media. Limitations can be stated for acidic influent (pH below 6), which can partially dissolve material and release P into solution. About 50 different types of apatite exist with more or less impurities. Other studies in our laboratory are now in progress on different apatites and later communications will be presented to show how kinetics could be considerably improved by taking the mineralogy of apatites into account.

Acknowledgements

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P. Molle *et al.*

203