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Removal of phosphorus through struvite precipitation using a by-product of magnesium oxide production (BMP): Effect of the mode of BMP preparation

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Abstract

Different modes of preparation of a by-product of a magnesite calcination process for magnesium oxide production (BMP) were compared for phosphorus removal to struvite at a molar Mg:P ratio of 1.6 mol Mg/mol P from liquor of a full-scale sewage treatment plant. Five experimental runs were performed—Run 1: raw BMP; Run 2: screened to particle size < 0.04 mm; Run 3: milled to particle size < 0.04 mm; Run 4: suspension at a concentration of 3 g of BMP/l of tap water; Run 5: supernatant after settling for 4 h after 28 h aeration. Temperature and pH were determined in liquid samples and analyses of phosphorus (P) and magnesium (Mg) were also performed in liquid and solid samples. pH increased with the reaction time (t) in Runs 1–4. However, in Run 5 there was an initial increase from 8.28 to 8.41 only to decrease to 8.34 at the end of the experiment. Phosphorus concentration decreased with t, but also with the mode of BMP preparation where the concentration of phosphorus in relation to the reaction time was lower in Runs 2 and 3 than in the other runs, the minimum being in Run 5. It was found that the smaller the particle size, the lower the concentration remained in solution. This fact was confirmed by the values obtained in the equilibrium concentrations which were: 14.7, 9.1, 13.2, 17.4 and 7.3 mg/l for Runs 1–5, respectively. The amount of struvite obtained was improved by the increase of t, the maximum being in Run 5 at 0.5 h (450 mg/l). In Runs 2 and 3, values of struvite production of 400 and 350 mg/l were achieved at t higher than 2.5 h. It was found that a first-order kinetic model fitted in well with the experimental data obtained. The values of the reaction constants were found to be: 1.2, 1.5, 1.4, 1.3 and 2.9 h^{-1} for Runs 1–5, respectively. The proposed model accurately predicted the performance of the process, showing deviations lower than 5% between the experimental and theoretical data of struvite production.

Keywords: Struvite; By-product of magnesium oxide production (BMP); Mode of preparation; First-order kinetics

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1. Introduction

Wastewater treatment plants must remove phosphorus and nitrogen compounds from the wastewater before its final disposal in order to prevent eutrophication [1,2]. Crystallization of N and P in the form of struvite ($MgNH_4PO_4 \cdot 6H_2O$) has been successfully used for nutrient removal and for obtaining a valuable fertiliser [1,3]. Since wastewaters tend to be low in magnesium ion, it is usually necessary to add this mineral so as to enhance the process [4]. Struvite precipitation for NH_4^+ removal was studied in effluents from one and two-phase anaerobic digesters of dairy manure [1]. Mg^{2+} ion was added using both $Mg(OH)_2$ and $MgCl_2 \cdot 6H_2O$. Efficiencies above 95% were obtained when $MgCl_2 \cdot 6H_2O$ was added and showed to be higher than when treated with $Mg(OH)_2$ for the same Mg^{2+} concentration. It was observed that the pH adjustment to 8.5 did not improve the NH_4^+ removal. The influence of Ca^{2+} ions on struvite precipitation was studied in aqueous solutions containing Mg^{2+}, NH_4^+ and PO_4^{3-} ions at a molar ratio of 1:2:2 and pH of 9.0 [5]. The presence of calcium significantly affected the characteristics and size of the crystal produced. Struvite precipitation and ammonia stripping were compared on anaerobically pretreated landfill leachate with NH_4^+ concentration of 2240 mg/l [6]. A stoichiometric ratio (Mg^{2+}:NH_4^+:PO_4^{3-} = 1:1:1) was

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applied and a maximum removal of 85% was observed at pH 9.2. In ammonia stripping with 2 h of aeration, removal of 72% at pH 12 was obtained while the removals were around 20% at lower pH. The authors concluded that both systems could be used as appropriate post-treatment. A bench-scale experiment was performed to remove NH_4^+–N in the leachate collected from a local landfill in Hong Kong [7]. It was found that N–NH_4^+ could be reduced from 5618 to 112 mg/l within 15 min, when $MgCl_2\cdot 6H_2O$ and $Na_2HPO_4\cdot 12H_2O$ were applied with a Mg^{2+}:NH_4^+:PO_4^{3-} molar ratio of 1:1:1. The pH range of the minimum struvite solubility was between 8.5 and 9.0. A reactor with dual functions (crystallization through aeration and separation of formed struvite by settling) for swine wastewater was evaluated [8]. It was found that struvite scraped off easily with only a light brushing, with a purity of 95%. Bench-scale studies were conducted in different streams of a full-scale sewage treatment plant [9]. It was demonstrated that centrifuge liquor with a composition of 167 mg/l phosphorus, 44 mg/l magnesium, 615 mg/l ammonium, 56 mg/l calcium, 2580 mg/l of alkalinity and a pH of 7.6 was the most suitable stream. This stream had a predicted struvite precipitation potential of 140 mg/l and up to 97% of phosphorus removal as struvite was achieved. Pilot and bench-scale studies have been carried out using anaerobic supernatants from a centrifugation sludge section of full-scale biological nutrient removal plants [10–14]. The plants used anaerobic, anoxic and aerobic processes to perform carbon oxidation, nitrification–denitrification and biological phosphorus removal, respectively. An increase in pH from 8.1 to 9.1 determined an increase in the removal efficiency from 65 to 80%, while the maximum removal efficiency was achieved before 0.4 h of contact time, remaining constant from there on. Phosphorus removal from an anaerobic supernatant through the crystallization of struvite and/or hydroxyapatite in a fluidized bed reactor at bench and half-scale was also investigated, obtaining maximum phosphorus removal of 80% and proving the suitability of the reactor used. The results showed phosphate removals higher than 80% through crystallization on quartz sand in about 100 min. Phosphate removal of anaerobic supernatant with alkalinity 3550 mg $CaCO_3$/l, PO_4^{3-} 139 mg/l, and Mg^{2+} 24 mg/l was studied using a fluidized bed reactor. Air was only used for stripping to reach the supersaturation pH and a removal efficiency higher than 90% was achieved without adding any chemicals.

Pilot-scale anaerobic treatment followed by a struvite production reactor was performed for a non-diluted whisky pot [15]. Removal efficiency in COD was 76% at a loading rate of 20 kg COD/m^3 day, while around 80% of organic nitrogen was converted into N–NH_4^+, and around 90% of the organic phosphorus into P–PO_4^{3-}. In the reactor for struvite production approximately 20% of N–NH_4^+ and over 90% of P–PO_4^{3-} were simultaneously removed. Adding $MgSO_4$ to the anaerobic batch digestion supernatant of piggery wastewater was carried out at laboratory scale [16]. The use of this reagent raised the pH to 9.0 and led to a reduction in PO_4^{3-}–P concentrations from 33 to 7 mg/l. The precipitate formed was comprised of struvite, apthitatite ($K_3Na(SO_4)_2$) and thermarite (Na_2SO_4). Anaerobic digester liquor with a concentration of 61 mg P/l from an enhanced biological phosphorus removal wastewater treatment plant was treated at pilot-scale [17]. Magnesium hydroxide slurry at 60% was used to produce struvite and phosphorus removal of 94% was obtained. There was no influence of the hydraulic residence time in the range of 1–8 h. Heavy metal concentration in the dry product was well below the legal limits and may be used as fertiliser. A low-cost source of magnesium ions, a by-product of salt manufacture (bittern), was evaluated [4]. Phosphorus removals with bittern added were 76%, compared to 75% and 81% for $MgCl_2$ and seawater, respectively. The performance of bittern for ammonia removal was 39%, compared to 53–54% for $MgCl_2$ and seawater. The use of the by-product of the magnesite calcination process (BMP) was also studied in synthetic and anaerobic digester liquor [18,19]. The effect of (Mg:P) molar ratio of pure MgO reagent and BMP was compared for synthetic liquor. The feasibility of a first-order kinetic model was demonstrated with the values of the reaction constants higher for pure MgO (2.34–2.88 h^{-1}) than for BMP (0.54–1.02 h^{-1}). Experiments with real anaerobic liquor and BMP at molar (Mg:P) in the range of 1.5–3.6 gave values of the reaction constant from 1.08 to 1.74 h^{-1}. The results obtained demonstrated that BMP was a good reagent for the removal of phosphorus and for producing struvite. The effect of pH and molar ratio Mg:P in anaerobic swine lagoon liquid was evaluated at a pH range of 7.5–9.5 and Mg:P between 1:1 and 1.6:1 [20]. It was found that struvite formation reduced the PO_4^{3-}–P concentration to as low as 2 mg/l at a pH range of 8.90–9.25 for all Mg:P ratios. Struvite precipitation lowered the PO_4^{3-}–P concentration by 85% within 20 min at pH 9.0 for a Mg:P ratio of 1.2:1. A first-order kinetic model also described the PO_4^{3-}–P decrease with rate constants of 3.7, 7.9 and 12.3 h^{-1} at pH 8.4, 8.7 and 9.0, respectively. The kinetics of the spontaneous precipitation of struvite was also investigated in aqueous supersaturated solutions containing stoichiometric concentrations of Mg^{2+}, NH_4^+ and PO_4^{3-} ions, ionic strength 0.15 M NaCl and at 25 °C in a batch stirred reactor at constant supersaturation. Kinetic analysis of the rates that depended strongly on the solution supersaturation yielded a second-order dependence suggesting a surface diffusion mechanism [21]. With this in mind, the aim of the present work was to evaluate different modes of BMP preparation on the performance of phosphorus removal and struvite production using real liquor derived from a sewage treatment plant.

2. Materials and methods

2.1. Characteristics and features of the liquor used in the experiments

The liquor used in the experiments was obtained from a domestic wastewater treatment plant at “Navalcarnero” serving a population of 70,000 inhabitants and located outside the city of Madrid, Spain. The plant is composed of the following treatment steps:

1. Preliminary treatment made up of screening and grit and grease removal.

Table 1
Characteristics and features of the liquor used in the experiments

Parameter	Values[a]
COD	675 (mg/l)
$P–PO_4{}^{3-}$	64.4 (mg/l) 2.08 (mM/l)
Mg	16.4 (mg/l) 0.68 (mM/l)
Ca	49.2 (mg/l) 1.23 (mM/l)
$NH_4{}^+$	322 (mg/l) 17.89 (mM/l)
pH	7.31
Alkalinity	2110 (mg/l)
Temperature	13.0 (°C)

Values are averages of three determinations. There was virtually no variation (less than 3%) between analyses.

2. Primary treatment by plain sedimentation.
3. Secondary treatment by a biological process composed of anaerobic-anoxic–aerobic steps.
4. Secondary settling tank.
5. Sludge treatment by gravity thickener and screw press.

The supernatant of the thickener and the liquor from the press-screw were recycled to the inlet of the plant. A sample of this liquor was used in the experiments. The characteristics and features of the liquor are summarized in Table 1.

2.2. Characteristics of the by-product of magnesium oxide production

The reagent used as a magnesium source in the experiments was obtained as a residue from a cyclone located for cleaning of the exhausted gases from the kiln used for magnesite calcinations in order to obtain magnesium oxide. This reagent was provided by Magnesites Navarra S.A. Table 2 shows the chemical composition and the particle size of the BMP used in the experiments.

2.3. Experimental procedure

Five experimental runs were performed using a molar ratio (Mg:P) of 1.6 in all cases. The reagent used in Run 1 was BMP as it is. In Run 2, the reagent used was screened BMP to obtain a particle size of ≤0.04 mm. In Run 3, the reagent used was BMP milled to a particle size of ≤0.04 mm. In all these runs, doses of 230 mg of BMP were added per litre of liquor. In Run 4, the reagent BMP as it is was added in suspension at a dose of 76 ml of suspension per litre of liquor to achieve the desired molar ratio. The suspension was obtained by mixing 3 g of raw BMP with 1 l of tap water with a magnetic stirrer at 500 rpm for half an hour. In Run 5, the suspension previously mentioned was mixed for 28 h and settled for 4 h and the supernatant obtained used in the experiment at a dose equal to that used in Run 4. Due to the small volume of the doses used in Runs 4 and 5 (76 ml/l of liquor, 7.6% of the total volume of liquor), the effect of dilution was not considered in the experiment. In addition, the initial concentration of Mg in the liquor was not taken into account when defining the doses of reagent used in the experiments.

Table 2
Particle size distribution and chemical composition of BMP as it is

Particle size (mm)	Distribution (%)	Component	Concentration (%)
<0.1	82.2	Mg–MgO	67.67
0.1–0.2	13.2	Ca–CaO	9.58
0.2–0.4	2.6	$Al–Al_2O_3$	0.37
0.4–0.5	0.7	$Fe–Fe_2O_3$	2.63
0.5–0.7	1.3	SO_3	3.95
		SiO_2	2.60
		LOI[a] at 1100 °C	13.20

[a] Loss of ignition.

The experimental runs were performed in 5 l operational volume glass vessels. After the reagent dosage the samples were mixed with magnetic stirrers at 120 rpm. Each experimental run lasted 3 h. Samples of a volume of 50 ml were taken at the beginning and every half hour of the experiment to determine the effect of time on the course of reaction. Before the start of the experiments, the liquor samples were aerated for 30 min by using a blower at a flow of 15 l per min. The experiment was initiated with the addition of the BMP reagent.

2.4. Analytical techniques

Phosphorus (P), calcium (Ca) and magnesium (Mg) were determined in the solutions by plasma emission spectrometry (PES-ICP) in a Jarrell Ash 955 ICP spectrophotometer. The rest of the determinations: chemical oxygen demand (COD), temperature, ammonia, pH and alkalinity were performed according to the recommendations of the standard methods [22]. The amount of struvite was determined in the precipitates obtained by vacuum filtration and drying the solid samples at 40 °C to prevent the loss of crystallization water. Afterwards, the samples were digested by perchloric and nitric acids. The main components examined in the acid solution obtained were phosphorus, magnesium, calcium and ammonium, which were determined as described above. Phosphorus concentrations in the liquid and precipitate samples were determined in triplicate. Given that the standard deviations of the mean values were always less than 3%, average values were considered and plotted in the related graphs.

2.5. Kinetic model

Based on the literature review, a first-order kinetic model was applied to the experimental data obtained [18–20,23–25]. The kinetic constants were determined by fitting a slightly modified first-order kinetic model to the experimental data obtained. This model relates the disappearance of a reactant ($-dC/dt$) to the rate constant (K) and the reactant concentration at reaction time t (C) minus the reactant concentration at equilibrium

(C_E) through Eq. (1):

$$-\frac{dC}{dt} = K(C - C_E) \tag{1}$$

By integrating Eq. (1) and ordering the terms, the following linear form of the first-order rate equation was obtained (Eq. (2)):

$$-\ln\frac{C - C_E}{C_0 - C_E} = Kt \tag{2}$$

where C_0 is the initial concentration of the reactant. Assuming first-order reaction kinetics, a plot of $-\ln[(C - C_E)/(C_0 - C_E)]$ against reaction time should give a straight line with slope K [20] and the intercept equal to zero.

According to Eq. (1), the value of C_E can be obtained from the intercept with the abcissa at $-dC/dt$ equal to zero when this parameter is plotted versus the phosphorous concentration remaining in the filtrate. Therefore, the value of C_E may be determined by plotting the naeperian logarithm of reactant concentration ($\ln(C)$) versus the inverse of the time of reaction ($1/t$). The value of the intercept with the abscissa equal to zero is equivalent to the naeperian logarithm of C_E.

Based on Eq. (2), the production of struvite as a function of the reaction time can be determined from the equation:

$$C_0 - C = (C_0 - C_E)(1 - e^{-Kt}) \tag{3}$$

where $(C_0 - C_E)$ is the maximum amount of struvite obtainnable per litre of wastewater treated.

3. Results and discussion

3.1. Experimental results

Aerating the liquor for 30 min caused a considerable increase in the pH, from 7.31 to 8.25–8.28, a value favourable for the reaction of struvite production [3,7,19,23,26,27]. The increase in pH may be caused by the displacement of dissolved CO_2 from the liquor solution with air as well as by an increase in the concentration of bicarbonate and carbonate [26]. Additionally, a reduction in the ammonia nitrogen concentration from 322 to 296 mg/l was appreciated. The temperature value was 14 °C and this remained constant during the sample aeration. Fig. 1 shows the effect of the reaction time on the variation of pH for the different runs carried out. A similar behaviour was appreciated in Runs 1–4, for which an increase in pH with the increase in reaction time was observed with no significant differences in the first four runs. In Run 5, the variation of pH with reaction time was completely different, increasing from 8.28 to 8.41 at half hour reaction interval and decreasing to a value of 8.34 at the end of the experiment. This behaviour could be determined by the massive removal of phosphorus and ammonia that occurred at the first 0.5 h of reaction and by the reduction of the carbonate concentration which determined the increase of the relative concentration of bicarbonate and the decrease of alkalinity. Similar results have been reported by other researchers [1,5,12].

Fig. 2 shows the effect of time (t) on the phosphorus concentration in the liquor for the different experimental runs performed. As can be seen, typical exponential curves were obtained in all cases. The concentration of phosphorus decreased as a function of t, but this decrease also depended on the mode of BMP preparation. A considerable decrease in the phosphorus concentration was achieved during the first hour of reaction, decreasing slowly at higher values of reaction time. The effect of the mode of BMP preparation was also of great importance, where the phosphorus concentration in relation to the reaction time was lower in Runs 2, 3 and 5 than in Runs 1 and 4. It was found that the minimum value of phosphorus concentration corresponded to Run 5 being 7.3 mg/l. The supernatant of BMP after 28 h of mixing and 4 h of settling had a magnesium concentration of 635 mg/l or 26.5 mM/l. This fact facilitated the reaction of struvite formation because magnesium in solution reacts faster than in other modes of preparation, which determines lower concentrations of phosphorus remaining in the solution.

The influence of reaction time and mode of BMP preparation on the percentage of phosphorus removed (η) with respect to the initial concentration of phosphorus (C_0), may be determined by the equation:

$$\eta = \left[\frac{C_0 - C}{C_0}\right] \times 100 \tag{4}$$

where η is the percentage of phosphorus removed, C_0 the initial concentration of phosphorus in the liquor (in this case, 64.4 mg/l) and C is the phosphorus concentration in the liquor at time t. As can be determined from Fig. 2, the maximum amount of phosphorus removal was achieved in Run 5 with a value of around

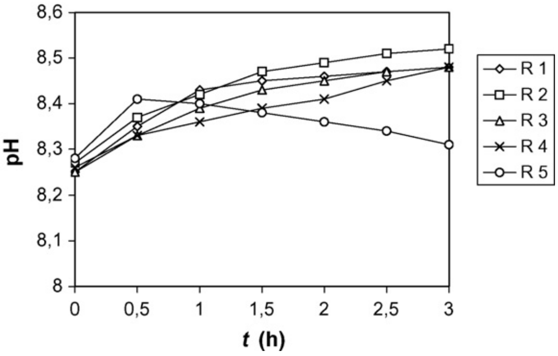

Fig. 1. Variation of pH with the reaction time for BMP at different modes of preparation.

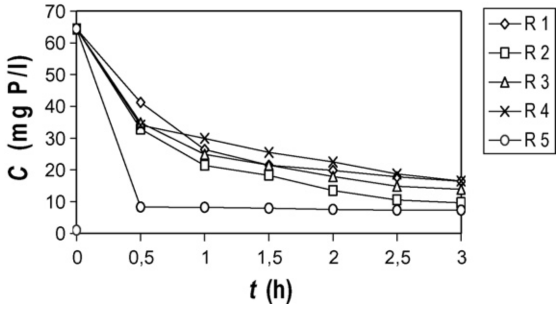

Fig. 2. Variation of the phosphorus concentration with the reaction time for BMP at different modes of preparation.

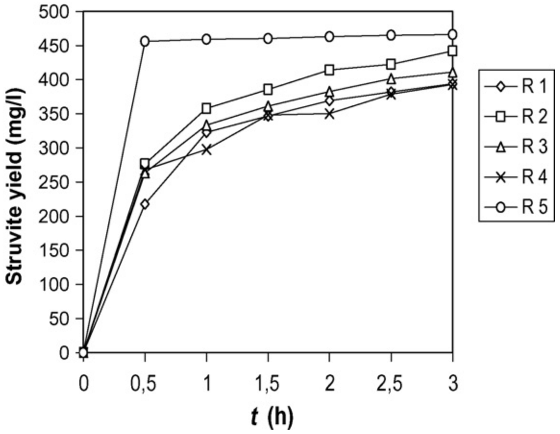

Fig. 3. Amount of struvite obtained at different reaction times and modes of BMP preparation.

90%. This percentage was achieved at a reaction time of 0.5 h. At higher values of t the percentage recovered did not increase significantly. In the case of Run 2, the maximum performance obtained was around 80% at a contact time of 2.5 h. In Run 3, the maximum amount removed was reached at a contact time of 2.5 h, but the value was slightly lower compared with Run 2. In the case of Runs 1 and 4, the results obtained were very similar with the maximum amount removed at around 70% at a contact time of 2.5 h.

Fig. 3 shows the effect of reaction time on the amount of struvite obtained per litre of liquor treated. The amount of struvite obtained increased as the reaction time increased. It was observed that in the Run 5, the maximum production of struvite – around 450 mg/l – was achieved at 0.5 h. More acceptable results were obtained in Runs 2 and 3 as compared with the rest of experiments, with values over 400 and 350 mg/l, respectively, at times higher than 2.5 h.

3.2. Kinetic modelling

Fig. 4 shows the plot of the naeperian logarithm of phosphorus concentration in liquor versus the inverse of the reaction time in order to determine the values of C_E for the different experimental runs. As can be seen, straight lines were obtained with intercepts with the abscissa equivalent to the values of C_E. It was found that the equilibrium concentration, C_E, was different in all runs and the values were found to be: 14.7, 9.1, 13.2, 17.4 and 7.3 mg/l for Runs 1–5, respectively. The values of the equilibrium concentration obtained show the capacity of the different modes of BMP to react with phosphorus present in the liquor, in such a manner that the lower the value of the equilibrium concentration, the higher the capacity of the reagent to react with the phosphorus to produce struvite, the highest being for Run 5 and the lowest for Run 4. With the corresponding values of C, C_E and t, the values of the reaction constants can be determined by using Eq. (2). Table 3 shows the values of the kinetic constants obtained for the five runs considered. The absolute values of the standard deviations were lower than 0.1 h^{-1} in all cases. The values of K obtained in the cases of R1, R2 and R3 were very similar to those obtained in a previous work in which raw screened and milled BMP were used [19]. In addition, the values of the reaction constants in Runs 1 and 4 were very close, indicating that the utilization of BMP as it is in suspension did not improve the process performance. However, in the case of R5, the value of the constant was significantly higher than that obtained in the other runs carried out and as high as that obtained in a previous work with pure MgO and real liquor [19].

By using Eq. (3), the theoretical and experimental values of phosphorus recovered as struvite were compared and the results are given in Fig. 5. As can be seen, the differences between the experimental and the theoretical data predicted with the model described by Eq. (3) were lower than 5% in all cases. These

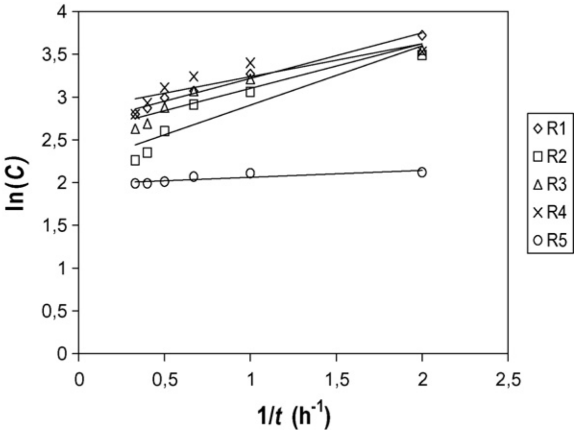

Fig. 4. Determination of the equilibrium concentration at different modes of BMP preparation.

Table 3
Values of the kinetic constants, K, obtained by Eq. (2)

Run	K (h^{-1})	Standard Deviation (h^{-1})
R1	1.2	0.07
R2	1.5	0.07
R3	1.4	0.05
R4	1.3	0.06
R5	2.9	0.09

Probability level of 95% ($p < 0.05$).

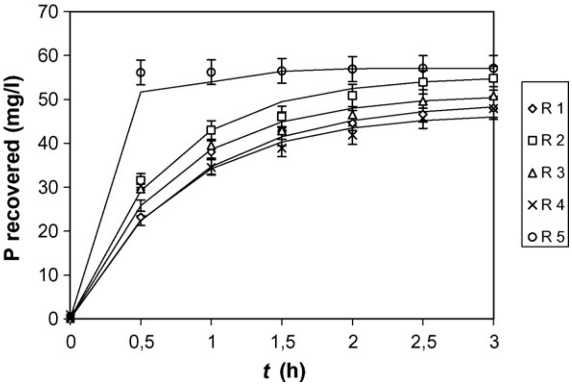

Fig. 5. Comparison of the experimental data of phosphorus removed and those predicted by the model for different reaction times and modes of BMP preparation.

results strongly suggest the validity of the first-order model proposed for describing the kinetics of struvite production. The experimental results obtained also show that the best results were achieved when supernatant of BMP (Run 5) were employed with a maximum removal of 90% in phosphorus at 0.5 h of reaction time. Additionally, in this case the removal of ammonia was around 30%, obtaining an effluent with a phosphorus concentration of 6 mg/l and an ammonia concentration of 200 mg/l.

4. Conclusions

The experimental results obtained show that BMP treated by mixing for 28 h and settling for 4 h (R5) was the best mode of preparation and the most effective procedure for phosphorus removal and struvite production. The mode of BMP preparation had a considerable influence on the process performance. The reagent in solution or in fine suspension reduced the reaction time and increased the quantity and quality of the final product obtained. A first-order kinetic model adequately fitted in with the experimental data obtained. A maximum value of K and minimum value of C_{E} were obtained when supernatant of BMP previously mixed and settled was used. According to the values of the reaction rate constant, the following order was obtained in the runs: R5 > R2 > R3 > R4 > R1. It was found that the proposed model based on the first-order kinetics predicted the process performance very accurately showing deviations lower than 5% between the experimental and theoretical values of struvite production. The results obtained will be very useful for the application of this process at pilot-scale.

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