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Reusing industrial by-products to enhance phosphorus removal in waste stabilization ponds: laboratory approach

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ABSTRACT

Waste stabilization ponds (WSP), in spite of being a suitable technology for wastewater treatment, present low phosphorus removal. This study aimed at evaluating the net increase on phosphorus removal efficiency in microcosm WSP in which sludge was conditioned with an adsorbent (industrial by-product) having a high phosphorus retention capacity. In order to determine the best candidate to condition the sludge, four different industrial by-products (granular bentonite; fly ashes from a municipal solid waste incineration plant; and two types of fly ashes from power plants) were tested for their phosphorus adsorption capacity. Experimental results were fitted to Langmuir and Freundlich models. All adsorbents showed a high phosphorus adsorption capacity. Maximum phosphorous adsorption capacity estimated from Langmuir equations ranged between 34.7 and 74.0 mgP/g adsorbent, being fly ashes from a power plant and granular bentonite the adsorbents with the highest and lowest adsorption capacity, respectively. Microcosms WSP were set up and the sludge conditioned with fly ashes from a municipal solid waste incineration plant. Results showed that phosphorus removal efficiency increased up to 90% by adding 5% of adsorbent (in terms of weight of adsorbent to weight of sludge). Main conclusion is that of industrial by-products may be a low-cost solution for enhancing phosphorus removal in WSP.

Keywords: Bentonite; Fly ashes; Isotherm; Waste stabilization ponds; Phosphorus

1. Introduction

Phosphorous (P) is widely used either in the production of fertilizer for agricultural activities or in explosives, pesticides and detergents [1]. Human activities have significantly affected natural phosphorous cycles, which have resulted in some serious ecological problems [1]. Excessive phosphorous in surface water is unhealthy for aquatic ecosystems, since it causes eutrophication [2–4]. According to that, wastewater needs proper treatment for the removal of phosphorous to levels that are acceptable for natural systems [5]. Conventional processes proposed for phosphorus removal from domestic wastewater are mostly based on chemical precipitation (such as electro-coagulation, struvite precipitation and the use of iron, aluminium and calcium salts); biological accumulation (based on operational processes enhancing the growth of phosphate accumulating organisms) and adsorption (by aluminium hydroxide, ferric oxides among others) [6]. Chemical and biological processes

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are expensive and increase the amount of sludge generated during wastewater treatment. However, technologies based on adsorption processes are easy to operate, generate little sludge and allow phosphorous recycling processes to be applied [6,7]. Phosphorus removal in conventional wastewater treatment systems has been widely addressed by applying either physical–chemical precipitation or by enhanced biological. However, phosphorus removal in natural wastewater treatment systems for the sanitation of small communities or rural areas is a critical issue that still remains under improvement [4].

Waste stabilization ponds (WSP) have been considered as an effective and low-cost technology for wastewater treatment in small communities [8,9]. WSP are shallow basins in which wastewater is treated by bacteria (anaerobic/aerobic) and algae. Pollutants removal in WSP is carried out by means of physical, chemical and biological processes, which naturally occurs in aquatic systems. Furthermore, phosphorus removal in WSP depends on a wide variety of environmental conditions such as temperature, pH, E_H, and dissolved oxygen concentration and involves complex physical and bio-chemical processes (i.e.: adsorption, precipitation, uptake by microorganisms and sedimentation). Accordingly, phosphorus can be adsorbed by sediment, or it can be released into water from the sediment [10] leading to a net phosphorus removal, which is usually less than 50% [11]. The amount of phosphorus adsorbed depends on the capacity of the sediment to adsorb and desorb phosphorus from solution [8,12]. To this regard, several authors proved that phosphorus adsorption could be enhanced by the presence of iron (Fe), aluminium (Al) and calcium (Ca) at high concentration in the sediment [8,13]. Some industrial by-products are rich in Fe, Al and Ca (such as fly ash, furnace slag and alum sludge) and may be a low-cost solution to enhance the phosphorus adsorption/removal capacity of WSP sediments [2,4,13,14]. In spite of the potential use of industrial by-products to enhance phosphorus removal in WSP, there is scarce information in current literature.

This study aimed at evaluating the net increase on phosphorus removal efficiency in microcosm WSP in which sludge was conditioned with an adsorbent (industrial by-product) having a high phosphorus retention capacity. In order to determine the best candidate to condition the sludge, four different industrial by-products (granular bentonite; fly ashes from a municipal solid waste incineration plant and two types of fly ashes from power plants) were tested for their phosphorus adsorption capacity. Afterwards, a microcosm WSP experiment was conducted at laboratory scale in order to determine the net enhancement of phosphorus removal in WSP by conditioning the sediment with fly ashes from a municipal solid waste incineration plant.

2. Materials and methods

2.1. Industrial by-products

Adsorbents studied were a granular residue of commercial granular bentonite (GB) provided by the AEB group, fly ashes from the municipal solid waste incineration plant of Mataró (MSW FA) and two types of fly ashes from two different power plants (named under PP FA I and PP FA II). More precisely, PP FA I and PP FA II were provided by the power plants of Cercs and Andorra (Teruel), respectively.

Adsorbents were characterized in terms of total solids (TS) and volatile solids (VS) following standard methods [15].

Chemical composition of adsorbents was analysed by X-ray fluorescence spectrometry in order to determine the concentration of Fe, Mn, Ti, Ca, K, P, Si, Al, Mg and Na oxides. Physical and chemical characteristics of adsorbents are shown in Table 1.

Table 1
Chemical characteristics of industrial by-products

(%)	GB	MSW FA	PP FA I	PP FA II
TS	97.91	100	99.29	97.74
VS	2.09	0	0.71	2.26
Fe_2O_3	2.04	1.35	20.08	6.36
MnO	0.02	0.05	0.04	0.07
TiO_2	0.24	0.96	0.87	0.71
CaO	1.00	31.25	7.35	27.40
K_2O	0.86	4.33	1.32	1.54
P_2O_5	0.03	1.41	0.44	0.36
SiO_2	73.66	11.97	41.90	38.63
Al_2O_3	13.44	5.30	25.22	16.36
MgO	2.27	1.66	1.11	1.37
Na_2O	2.10	4.01	0.33	0.22

Note: GB: granular bentonite; MSW FA: fly ashes from the municipal solid waste incineration plant; PP FA I and PP FA II two types of fly ashes from two different power plants; TS: total solids; VS: volatile solids.

2.2. Isotherm experiments

2.2.1. Bach adsorption experiments

Batch adsorption experiments were carried out in duplicate following the procedure described by Nair et al. [16]. Briefly, Erlenmeyers of 250 mL were filled up with 50 mL of distilled water to which KH_2PO_4

was added to reach the desired phosphorus concentration (see below for the specific phosphorus concentrations tested). Furthermore, adsorbents were pre-screened (<2 mm) and 1 g was added to each Erlenmeyer. Electrolyte solution ($CaCl_2$) was added to a molar concentration of 0.01 mols/L, and bacterial growth was inhibited by adding chloroform at a final concentration of 20 g/L. Phosphorus initial concentration (C_i) for each tested by-product is summarized in Table 2. Five equal C_i (from 0 to 900 mgP/L) were used for GB, MSW FA; whereas for PP FA I and PP FA II, one higher concentration was also considered to obtain a good correlation with Langmuir model and to obtain the Q parameter.

Flasks were continuously stirred for 24 h, as suggested by Nair et al. [16]. Experiments were carried out at room temperature (25°C). pH of the solution was measured by means of a Crison pH-meter at the beginning and at the end of the experiment. After 24 h, samples were collected from the flasks and analysed for orthophosphate concentration according to [15].

2.2.2. Adsorption isotherm models

Data obtained from the batch tests were fitted to both Freundlich and Langmuir adsorption isotherm equations.

The Freundlich isotherm model, which is an empirically equation more appropriated for heterogeneous substrates [17], is expressed as:

$$q = K_d C_e^{1/n} \tag{1}$$

where q is the amount of adsorbate per unit of adsorbent (mgP/g of adsorbent in this study); C_e is the equilibrium solution concentration (mgP/L); K_d ($\mathrm{mg}^{1-1/n}\ \mathrm{L}^{1/n}/\mathrm{g}$) is the Freundlich adsorption capacity parameter; and $1/n$ (dimensionless) is the Freundlich adsorption intensity parameter. K_d and $1/n$ are obtained empirically and considered constant for each adsorbent [18].

Freundlich isotherm can be also expressed as:

$$\log q = \log K_d + \frac{1}{n}\log C_e \tag{2}$$

A linear plot of log q and log C_e provides the value for K_d and n in the form of intercept and slope [2].

The Langmuir equation can be written as:

$$\frac{C_e}{q} = \frac{C_e}{Q} + \frac{1}{bQ} \tag{3}$$

where Q is the maximum adsorption capacity (mgP/g of adsorbent in this study) and b (l/mg) is a measure of the sorption energy. Langmuir adsorption allows to calculate the maximum phosphorous adsorption capacity of the substrates, and consequently, to estimate substrate longevity [13].

Q and b are determined from the slope and intercepts generated from a linear plot of C_e/Q vs. C_e [2].

2.3. Microcosm waste stabilization ponds

Microcosms WSP consisted of nine 400-mL glass bottles filled up with 250 mL of a 5 mg PO_4-P/L solution, which is in the range of phosphorus concentration of domestic wastewater in Spain [19], 15 mL of sludge from a municipal wastewater treatment plant located in Barcelona and 0.1 or 5% of fly ashes from a municipal solid waste incinerating plant (percentage expressed as weight of adsorbent to weight of sludge). Percentages of fly ashes applied in this study were selected according to previous and simplified trials in which phosphorus adsorption was qualitatively detected under sludge to by-product mixtures applied in this study (percentage expressed as weight of adsorbent to weight of sludge) (results not shown). Each experimental condition (control, 0.1% and 5% of industrial by-product) was tested in triplicate. Moreover, sludge layer thickness within glass bottles was that of 2 cm, while water column height was that of 9 cm. This sludge-to-water height ratio is comparable to that of a WSP after 5 to 10 years of operation [20]. Sludge employed was characterized in terms of TS, VS, chemical oxygen demand (COD), total P and total Nitrogen (N) according to [15]. A summary of sludge characteristics is given in Table 3.

Microcosms WSP worked under batch conditions. Samples withdrawal and feeding were carried out on a daily basis for one week. To this aim, 40 mL of overlaying water was daily collected from the bottles and analysed for orthophosphate concentration, dissolved oxygen and pH. Furthermore, in order to maintain the water level constant, 40 mL of phosphate solution at a concentration of 5 mg PO4-P/L was carefully added after sampling. Therefore, the microcosms WSP were operated at a HRT of ca. 6 d (which is in the range of that described in the literature for full-scale WSP [20]).

2.4. Statistical analyses

Differences among time for each experimental condition considered were determined by means of one-way repeated-measures ANOVA with three levels (control; 0.1% of industrial by-product and 5% of

Table 2
Estimated isotherm parameters and correlation coefficients (R^2) for P adsorption on all by-products

						Langmuir model					Freundlich model				
	C_i (mgP/L)	Real C_i (mgP/L)	C_e (mgP/L)	pH (initial)	pH (final)	q (mgP/mg media)	C_e/q	b (L/mgP)	Q (mgP/g of adsorbent)	R^2	log C_e	log q	n	K_d (mg$^{1-1/n}$ L$^{1/n}$/g)	R^2
GB	0	0	0	8.66	7.88	0	0	0.02	34.72	0.93			2.76	4.71	0.70
	100	103.08	0.57	6.68	7.45	5.10	0.11				−0.24	0.71			
	200	202.68	32.15	6.25	6.94	8.31	3.87				1.51	0.92			
	600	602.05	164.79	6.22	6.64	21.86	7.54				2.22	1.34			
	900	1,015.01	388.27	6.70	6.97	31.23	12.43				2.59	1.49			
MSW	0	0	0	12.10	12.10	0	0	0.16	42.55	0.69			1.29	3.08	0.72
FA	100	100.65	3.97	9.50	12.05	4.78	0.83				0.60	0.68			
	200	202.68	2.51	13.25	11.65	10.01	0.25				0.40	1.00			
	600	587.47	23.89	12.55	11.85	27.82	0.86				1.38	1.44			
	900	966.43	19.03	12.05	11.65	47.25	0.40				1.28	1.67			
PP	0	0	0	10.90	8.48	0	0	0.03	40.65	0.98			5.51	11.38	0.95
FA	100	94.68	0	6.50	7.85	4.71	0					0.67			
I	200	186.58	0.41	5.95	7.65	9.07	0.04				−0.39	0.96			
	600	550.45	32.34	6.56	6.86	25.90	1.25				1.51	1.41			
	900	901.80	338.65	6.82	7.01	28.07	12.06				2.53	1.45			
	1,800	1,838.74	1,014.32	6.99	7.24	40.92	24.79				3.01	1.61			
PP	0	0	0	11.7	8.9	0	0	0.06	74.07	1.00			2.45	7.41	0.82
FA	100	100.65	1.54	9.50	8.95	4.79	0.32				0.19	0.68			
II	200	210.46	1.54	7.60	9.05	9.99	0.15				0.19	1.00			
	600	592.33	14.17	7.85	8.85	28.08	0.50				1.15	1.45			
	900	908.13	19.03	7.45	8.90	43.85	0.43				1.28	1.64			
	2,000	2,059.58	606.91	7.70	9.10	72.25	8.40				2.78	1.86			

Note: GB: granular bentonite; MSW FA: fly ashes from the municipal solid waste incineration plant; PP FA I and PP FA II two types of fly ashes from two different power plants; C_i: initial concentration; C_e: equilibrium solution concentration; q: amount of adsorbate per unit of adsorbent; b: measure of the sorption energy; Q: maximum adsorption capacity; n: empirical constant of Freundlich isotherm model; K_d: empirical constant of Freundlich isotherm model.

Table 3
Physical and chemical characteristics of sludge used for microcosm WSP experiment

Parameters	Values
TS (%)	2.9
VS (% TS)	65.9
COD (g O_2/kg_{TS})	968.2
P (mgP/g_{TS})	3.9
P (% TS)	0.40
N (% TS)	0.06

Note: TS: total solids; VS: volatile solids; COD: chemical oxygen demand; P: total phosphorous; N: total Nitrogen.

industrial by-product). Homogeneity of variances and normality of data were tested by performing a Levene test and a Kolmogorov–Smirnov test, respectively. All statistical analyses were performed using SPSS v.19. Differences among levels of the same factor were determined by means of a Tukey's test. Analyses were considered statistically significant at p values below 0.05.

3. Results and discussion

All adsorbents tested showed high phosphorus adsorption capacity (Table 2). Moreover, data obtained fitted quite well for all adsorbents studied ($R^2 > 0.70$) (Table 2), regardless the model considered (Freundlich or Langmuir) (Fig. 1). However, the Langmuir model always showed a better correlation than the Freundlich model for almost all of the adsorbents tested (except for the MSW FA where phosphorus adsorption was described to a similar extent for both models) (Table 2 and Fig. 1). Our results are in agreement with the current literature since Langmuir equations have been described to provide a better fitting to data obtained from phosphorus adsorption experiments conducted on waste materials such as fly ashes [21] or dried sludge and cement dust [2]. Moreover, using the Langmuir model over other models is of special interest since it allows us to estimate the maximum (theoretical) phosphorus sorption capacity (Q) [13], which may be useful to compare among different adsorbents in terms of adsorption capacity and longevity. In line with this, GB showed the lowest value of Q (34.72 mgP/g of bentonite) compared to the rest of tested adsorbents. The highest value of Q was that of the PP FA II (74 mgP/g). The Freundlich coefficient K_d ranged between 3.08 and 11.38 $mg^{1-1/n}$ $L^{1/n}$/g, being the highest value that of PP FA I (11.31 $mg^{1-1/n}$ $L^{1/n}$/g). Fig. 1 shows a summary of all the experimental data obtained for all the industrial by-products analysed. It was observed that for low equilibrium concentrations (up to 30 mgP/L) all adsorbents had the same phosphorous removal. The ranking order in terms of maximum phosphorus adsorption capacity was that of the following: PP FA II > MSW FA > PP FA I > GB. Maximum sorption capacity (Q) ranged from 34 to 74 mgP/g of adsorbent. These values were very much higher than the values obtained from natural soils and sediments in treatment ponds (<2 mgP/g of sediment) [22] and natural materials such as gravel (<0.05 mgP/g of gravel) [23], sands (<0.2 mgP/g of sand) [24] or bentonite (0.93 mgP/g) [13]. Values of Q reported in this study were also higher than those reported for other waste materials such as alum sludge [2], furnace slag [21] or even fly ashes from China power plants (between 8 and 45 mgP/g) [13]. However, it is important to point out that maximum adsorption capacity of a material calculated from isotherms experiments (as it is carried out in this study) may lead to an overestimation (up to 3 times overestimation) of the true adsorption capacity of the material tested [25]. Therefore, authors suggest that longer study periods, closer to materials lifespan, are required to truly assess the phosphorus retention capacity of the tested materials. The magnitude of overestimation depends on several factors such as the methodology employed (batch or column studies) [26], initial phosphorous concentration and absorbents tested. Therefore, it is of difficult predictability under the conditions here considered and should be experimentally addressed.

According to [27], phosphorus is adsorbed and precipitated by the reaction with Ca, Fe, Al and Mg, which are released from the adsorbent to the solution. In this study, the ranking order in terms of maximum phosphorus adsorption capacity (PP FA II > MSW FA > PP FA I > GB) seemed to be more correlated to the amount of calcium oxides than other compounds. Indeed, the high P sorption capacity of PP FA II and MSW FA obtained corresponded to the adsorbents having a high content of CaO (27 and 31% for PP FA II and MSW FA, respectively), while Fe_2O_3, Al_2O_3 and MgO content was lower or equal to other absorbents (Table 1). In line with this, the high values of pH observed in the solution using PP FA II and MSW FA (Table 2) evidenced the presence of Ca^{2+} ions that favoured the adsorption [28]. The pH in the GB and PP FA I solutions (Table 2) indicated that phosphorous adsorption was caused mainly by Al_2O_3 content, which is relatively high in these by-products (Table 1) [13].

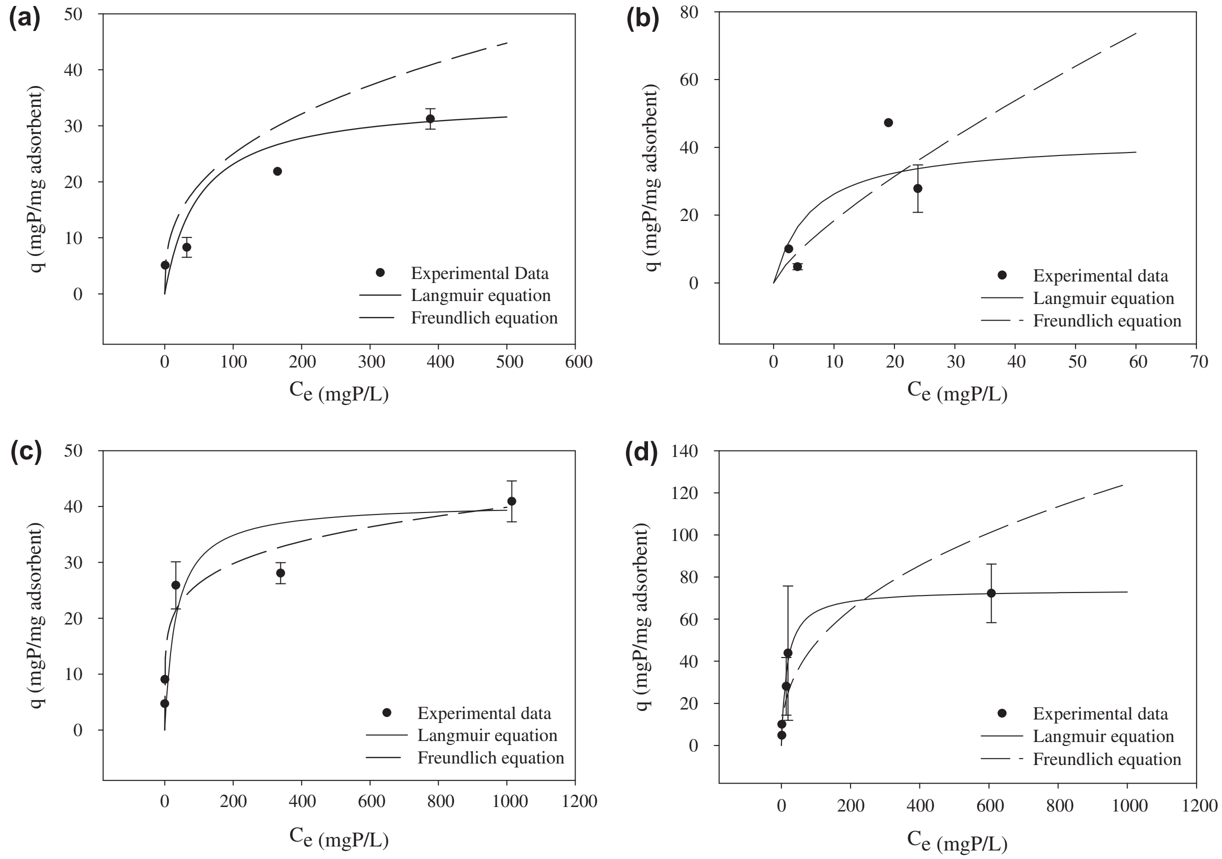

Fig. 1. P adsorption isotherms for GB (a), MSW FA (b), PP FA I (c), and PP FA II (d).

3.1. Enhancement of phosphorus removal in microcosm waste stabilization ponds

Fig. 2 shows the phosphorus concentration in overlaying water for all the experimental conditions considered. Phosphorus removal in control experiments was far lower than that observed in other experimental lines (around 10%), which suggested that phosphorous uptake by microorganisms was negligible. Therefore, chemical processes were the main responsible pathway for phosphorous removal in microcosms WSP. Phosphorus concentration in overlaying water for the control and the experimental lines conditioned with the lowest amount of adsorbent ranged from 3 to 6 mg PO_4-P/L. However, the phosphorus concentration was much lower for the experimental line with the highest content of adsorbent (from <0.1 to 0.5 mg PO_4-P/L) and statistically lower when compared to the rest of experimental conditions (Fig. 2). Phosphorus removal in the experimental line with sludge conditioned at a 5% with the MSW FA was close to 90%. On the other hand, the control line (without conditioning) showed a phosphorus removal, in average, close to 10%.

Concerning the pH, it ranged from 7.2 to 7.6 for the control and the experimental line with the lowest content of adsorbent, whereas it was significantly higher (ranging from 7.8 to 8.3) for the experimental line with the highest content of adsorbent. As mentioned above, a high content of Ca^{2+} ions in the experimental line having the highest content of fly ashes, which favoured phosphorus adsorption and precipitation.

Finally, oxygen concentration in the overlaying water ranged from 8.5 to 9.5 mg/L, regardless the experimental condition considered. Sediments strongly affect the performance of WSP during wastewater treatment [29]. The experiment carried out in this study proved that phosphorus removal in WSP can be enhanced by adding ashes from municipal incinerating plants. However, it is worth mentioning that harmful compounds present in such kind of wastes (such as heavy metals or chlorides) might leach from the sediments [29]. The fate of such harmful pollutants is, however, unclear under the circumstances here considered since it has been also demonstrated that phosphorus adsorption can be used as a chemical

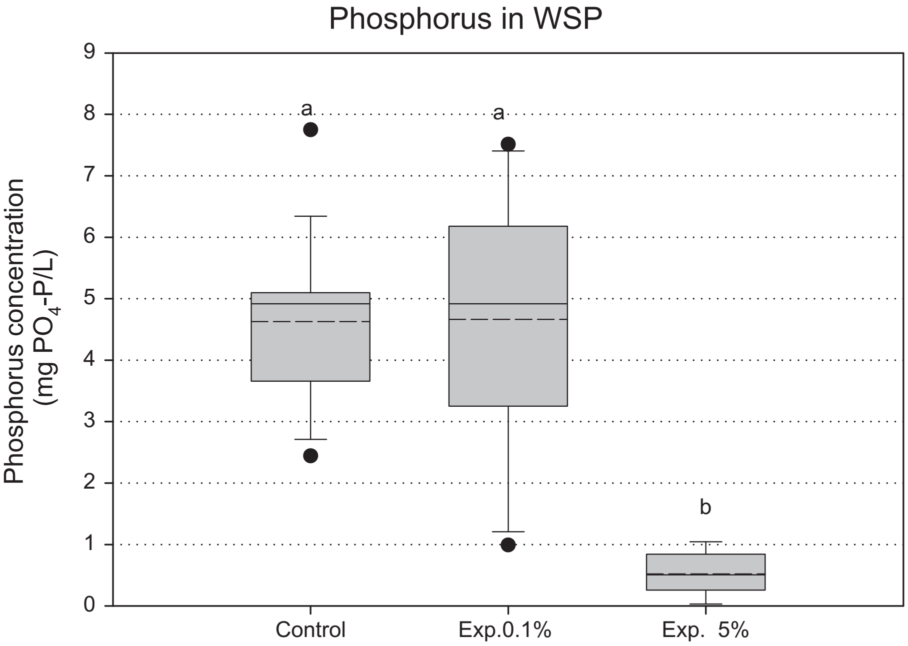

Fig. 2. P concentration in overlaying water for each microcosm WSP.
Note: *a* and *b* are groups of statistical significance determined by a post hoc test (Tukey's test).

process to stabilize heavy metals from bottom ash combustion of municipal solid waste plants [30]. Overall, authors believe that in spite of the potential use of industrial by-products for increasing the phosphorus treatment efficiency of WSP, further studies must address the potential negative effects of heavy metal leaching during treatment.

4. Conclusions

All adsorbents tested showed high phosphorus adsorption capacity and were generally better described by the Langmuir model. Maximum adsorption capacity ranged from 34 to 74 mgP/g of adsorbent, being fly ashes either from municipal solid waste treatment plants or power plants the materials with the highest capacity for phosphorus adsorption.

Results presented in this study proved that sludge conditioning with fly ashes from a municipal incinerating plant can substantially increase (up to 90%) the phosphorus removal capacity of waste stabilization ponds.

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