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Low-cost material as active substrates for the removal of phosphorus in synthetic effluents: a proposal for social treatment technology

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ABSTRACT

Considering the importance of the development of simplified technologies and social control in sanitation actions, this study investigated the use of laterite for phosphorus removal in synthetic effluents, through adsorption, as a low-cost alternative with the possibility of reusing the generated effluent, for communities where access to sanitation is limited. In the experimental design, the variables pH, contact time, granulometry and laterite dosage were used. Factorial planning was used for processing, for optimization and desirability. It was observed that the removal efficiency did not have significant interference in relation to the pH and contact-time variables. The kinetics of the batch experiments showed that the ideal contact time was 6.4 hours and pH of around 4. The adsorption capacity was plotted against equilibrium concentration for the Freundlich and Langmuir isotherms. The Langmuir isotherm was more suitable for phosphorus adsorption. The results show that laterite was effective in phosphorus adsorption in the order of removal of 87%, showing itself to be a potential adsorbent material.

Keywords: laterite, phosphate adsorption, simplified effluent treatment.

Material de baixo custo como substrato ativo para remoção de fósforo em efluentes sintéticos: uma proposta de tecnologia social para o tratamento

RESUMO

Considerando a importância do desenvolvimento de tecnologias simplificadas e de controle social nas ações de saneamento, este estudo investigou a utilização da laterita para remoção de fósforo em efluentes sintéticos, por adsorção, como alternativa de baixo custo com possibilidade de reuso do efluente gerado, para comunidades onde o acesso ao saneamento é limitado. No delineamento experimental, foram utilizadas as variáveis pH, tempo de contato, granulometria e dosagem de laterita. O planejamento fatorial foi usado para processamento, otimização e conveniência. Observou-se que a eficiência de remoção não interferiu significativamente em relação às variáveis pH e tempo de contato. A cinética dos experimentos em batelada mostrou que o tempo de contato ideal foi de 6,4 horas. A capacidade de adsorção foi plotada contra a concentração de equilíbrio e a isoterma de Freundlich e Langmuir. Apresentando-se a isoterma de Langmuir como a mais adequada para adsorção de fósforo. Os resultados mostram que a laterita foi eficaz na adsorção de fósforo na ordem de remoção de

87%, mostrando-se um potencial material adsorvente.

Palavras-chave: adsorção de fosfato, laterita, tratamento simplificado de efluentes.

1. INTRODUCTION

The lack of sanitation services is a global problem that affects about 4.5 billion of the world population, as 663 million people worldwide still consume water from unsafe sources (UNICEF and WHO, 2015); these are located mainly in small towns, peri-urban areas, and rural areas.

Due to the low economy of scale in many of these locations, there is little interest in promoting sanitation in these areas, resulting in a precarious infrastructure from capture to distribution. This primarily impacts the most vulnerable populations, such as rural areas and regions with less political and economic power (Murtha *et al.*, 2015). This in turn contributes to the proliferation of waterborne diseases (Ercumen, 2014; Kronenberger *et al.*, 2011) that also primarily affect vulnerable groups (Prüss-Üstün *et al.*, 2008; Ostro, 2004), causing injustice in terms of public policies.

Phosphorus is among the compounds present in domestic effluents that have an important impact on water bodies. It is a limiting nutrient for primary production, and for this reason it can deteriorate receiving water bodies (Pen *et al.*, 2017; He *et al.*, 2016; Withers *et al.*, 2014; Choi *et al.*, 2012;). The removal of this compound should therefore receive special attention (Jensen *et al.*, 2015; Barca *et al.*, 2014; Spears *et al.*, 2013; Ismail, 2012; Jyothi *et al.*, 2012).

Given the health importance of controlling phosphorus requirements in water, which may affect the quality of public health and the environment, regulations for human supply and surface water are presented that consider this parameter. Ordinance 888/2021 (Brasil, 2021) recommends that collective alternative water supply systems and solutions for human consumption, from surface and underground sources, must conduct an analysis of the total phosphorus parameter. Ordinance 357/2005 (Conama, 2005) establishes phosphorus limits according to the hydrodynamic conditions for each class. In lentic environments, the following concentrations of Total Phosphorus are permitted: Class 1 (0.025 mgP/L), Class 2 (0.05 mgP/L), Class 3 (0.15 mgP/L).

A waste management system must be environmentally effective, economically viable, and socially acceptable (McDougall *et al.*, 2007). Inadequate infrastructure and limited management systems increase stress on resources and can lead to a water crisis in many locations (Pandey *et al.*, 2010; 2012). As it is difficult to universalize sanitation actions, proposing simplified and low-cost systems can offer alternatives to provide opportunities for locations with little or no access to this service.

Different technologies have been used for the removal of phosphorus, such as chemical precipitation, biological treatment, and adsorption (Bashar *et al.*, 2018; Hupfer *et al.*, 2016; Tchobanoglous *et al.*, 2014). Among these, adsorption has gained prominence due to the ability to remove and recover phosphorus, as well as the possibility of applying low-cost and available materials for use as active substrates (Loganathan *et al.*, 2014; Lu *et al.*, 2009).

A wide range of potential active filter substrates, including natural materials, were tested, such as calcium carbonate (Li *et al.*, 2017), clay minerals (Lürling *et al.*, 2014), dolomite, and hydroxyapatite (Boeykens *et al.*, 2017), mesoporous materials (Huang *et al.*, 2017), lanthanum modified zeolites (Dong *et al.*, 2017; He *et al.*, 2016), natural zeolite pretreated with calcium hydroxide (Gypser *et al.*, 2018; Mitrogiannis *et al.*, 2017), laterite and sandstone (Coulibaly *et al.*, 2016), and have been reported to perform well in phosphate adsorption.

The most studied iron oxides for phosphorus adsorption are goethite and hematite, as they are abundant in oxidic soils (White and Dixon, 2002). However, laterite has also been used for phosphorus adsorption (Coulibaly *et al.*, 2016; Huang *et al.*, 2013; Mansing and Raut, 2013).

Its mineralogical characteristics express its potential for use as an adsorbent. It stands out for being an effective and low-cost adsorbent with high adsorption capacity for removing organic pollutants, providing an efficient treatment (Luo *et al.*, 2011; Zhang *et al.*, 2010; Zhao *et al.*, 2010). As a well-known separation process, adsorption has been widely applied to remove chemical pollutants from water. It has numerous advantages in terms of cost, flexibility and simplicity of design, operation and resistance to toxic compounds (Rafatullah *et al.*, 2010; Ahmad *et al.*, 2009; Zeng *et al.*, 2007). Although the challenges in operating in sanitation are fundamentally of a technical nature, overcoming these depends not only on technological and infrastructure innovation, but also on the development of technologies that correspond to the demands and how they are manifested locally.

Alternative actions are needed to identify the vulnerability in which the community finds itself, valuing cultural conditions, contributing to the transformation of tacit knowledge into explicit, and recognizing that public participation is effective in sanitation actions. Using the knowledge acquired by experiences in a formal and non-technical language, through a technology, will be easily understood by the population that receives it. In this study, an alternative post-treatment technology of easy operation and reduced cost is presented, which can be implemented on small scales, including isolated communities and in communities where access to sanitation is limited. Therefore, this study investigated the use of laterite in natura, for the removal of phosphorus in domestic effluents, as a low cost alternative, with the possibility of reusing the effluent generated after adsorption, for communities where access to sanitation is limited or where there is difficulty in implementing conventional systems.

2. MATERIAL AND METHODS

2.1. Sample collection and processing

The material used to test phosphorus adsorption in synthetic effluent were lateritic concretions from cerrado soil. The collected material was washed in tap water to remove impurities and then dried in an oven for 24 hours at 105ºC. The samples were then ground and sieved to particle sizes of 0.150 mm, 2 mm and 4 mm. The material already sieved was dried in a hot air oven at 105ºC (Mansing and Raut, 2013). The prepared samples were subjected to physical, chemical and mineralogical analyses.

Analytical data of pH, exchangeable bases (SB: Ca^{2+}, Mg^{2+}, K^{+}), extractable acidity ($Al^{3+} + H^{+}$), aluminum (Al^{3+}) and hydrogen (H^{+}) were used for calculating the cation exchange capacity (CEC), base saturation (V%), aluminum saturation and the organic carbon content (Embrapa, 1997).

The methodology used to determine the organic matter content was carried out by obtaining the organic carbon wet via potassium dichromate in a sulfuric medium, followed by titration with a standard solution of ferrous ammonium sulfate - Mohr salt - Embrapa (1997). The percentage of organic matter was calculated by multiplying the carbon result by 1.724. This factor is used because it is assumed that the participation of carbon in the average composition of humus represents 58% (Embrapa, 1997). The pH was determined by the potentiometric method, according to the Manual of Sampling Procedures for Physical-Chemical Analysis of Water (Parron *et al.*, 2011). The measurement was performed using a combined electrode immersed in soil: liquid (Potassium Chloride - KCl 1M) and soil: deionized water.

All variables at level zero constitute the central points, while the combination of variables that constitute a lower level (-1.673) or the highest level (+1.673) constitute the axial points. For the test of optimization and desirability of the results the program, Statistic Version 7.0 was used, applying the test of desirability, this tool makes it possible to identify better conditions of adjustment of a process that makes possible the simultaneous optimization of multiple responses, providing the best conditions and the most convenient way of processing.

2.2. Experimental conditions

For the adsorption tests, 3 granulometries (0.15mm, 2mm, 4mm) were adopted (Mansing and Raut, 2013). The pH values and contact time studied were defined based on studies on phosphorus adsorption (Coulibaly *et al.*, 2016; Mansing and Raut, 2013), adopting a pH range between 1.5 to 8, contact from 2 to 18 hours. The concentration of phosphorus used in the tests was determined taking as a reference the concentration of phosphorus found in domestic effluents subjected to conventional treatments (Aslan and Kapdan, 2006).

The phosphorus solution was prepared using KH_2PO_4, adopting the final concentration of 10mgP/L. The pH was adjusted using 1M HCl (hydrochloric acid) and 1M NaOH (sodium hydroxide) solutions. The percentage of solute absorbed was obtained by Equation 1:

$$R(\%) = \left(\frac{C_0 - C_e}{C_e}\right) V \tag{1}$$

Where: R (%) is the removal rate between the initial and equilibrium concentration, %; C_0 is the initial concentration of phosphorus, mg/L; Ce is the concentration of the solute after the time of contact with the soil in mg/L.

Adsorption is the mass of solute adsorbed per gram of soil, determined by Equation 2.

$$q_e\,(\%) = \left(\frac{C_0 - C_e}{Ms}\right) V \tag{2}$$

Where: q_e is the adsorption capacity, mg/g; C_0 is the initial concentration of phosphorus, mg/L; Ce is the concentration of the solute after the time of contact with the soil in mg/L, V is the volume of the solution used, L; Ms is the mass of soil used (kiln dried) in g.

The adsorption isothermal curve was obtained by plotting the weight of the adsorbed solute per unit weight of the adsorbent (qe) against the balance of solute concentration (Ce). The balance isotherm data were adjusted following the Langmuir and Freundlich models (Huang *et al.*, 2013; Kumar *et al.*, 2010), given by Equations 3 and 4, respectively. The parameters for each model were obtained from a non-linear statistical adjustment, and the evaluation of the correlation coefficients (r2).

$$q_e = \left(\frac{q_0 K_L\, C_e}{1 + K_L\, C_e}\right) \tag{3}$$

$$q_e = K_f\, C_e^{\,1/n} \tag{4}$$

Where:

q_e: adsorption capacity (mg/g);

q_0: maximum adsorption capacity (mg/g);

C_e: equilibrium adsorbate concentration (mg/L);

K_L: constant related to the solute binding energy/adsorbent surface (mg/L);

K_f: Freundlich constant (mg/g);

n: soil affinity parameter for the solute (admensional).

The effect of the phosphorus dose was studied at room temperature, using 4g of dry soil in an oven, to which was added 40 mL of the P solution, prepared in a 0.01M $CaCl_2$ solution with concentrations of 5, 10, 100, 150, 250, 400 and 1000 mL of P in the form of KH_2PO_4. Being placed under agitation of 100 rpm, temperature 25ºC, the minimum equilibrium time was chosen for this stage, in which the changes in the concentration of the solute in the solution were equal to or less than 5% in the interval of 24 hours, as recommended by the USEPA (1992). The separation of solid/liquid phases was done through centrifugation (Digital Centrifuge DAIKI), and later filtering through a paper filter, retaining the remaining soil particles. For phosphorus determination, the single-beam spectrophotometer (HACH DR 6000) was used. All the experiments were performed on a laboratory scale.

3. RESULTS AND DISCUSSION

The laterite used for the development of the study presents iron and aluminum oxides (Table 1), which allow the phosphorus (P) ions to react with the exchangeable cations and soluble ions on the internal surface of the oxides and hydroxides of Fe and Al, present in the substrate (Arai and Sparks 2001; Weng *et al.* 2011). The availability of these elements positively influences the phosphorus adsorption process, due to the ability of interaction between them (Coulibaly *et al.*, 2016; Huang *et al.*, 2013; Mansing and Raut, 2013; Vilar *et al.*, 2010).

Table 1. Physical and chemical characteristics of lateritic concretion used as an adsorbent material.

Parameters	Concentration
Physical	
Granulometry (%)	Clay: 21 Slime: 19.4 Sand: 59.6
Chemical	
pH ($CaCl_2$)	7.30
Organic matter content (%)	0.57
Macronutrients ($cmol_c/dm^3$)	Ca: 3.86 Mg: 0.58 Al: 0.00 H: 1.20 K: 0.23
Macronutrients (mg/dm^3)	P: 10.20
Micronutrients (%)	SiO_2: 37.4 Al_2O_3: 14.8 Fe_2O_3: 35.7 CaO: 0.22 MgO: 0.13 TiO: 0.75 P_2O_5: 0.16 Na_2O: <0.1 K_2O: 0.18 MnO: 0.03
CTC ($cmol_c/L$)	5.87

*Fusion with lithium tetraborate and quantification by XRF.

The iron content (35.7 mg/dm^3) found in laterite characterizes it as ferric soil (Embrapa, 2011), with a low organic-matter content (0.57%), which allows for an increase in the sorption capacity of P on the surface of the material once the humic substances competing for the adsorption sites are few. The organic matter influences phosphorus adsorption by the formation of organomineral complexes with the constituents of the clay fraction, reducing the exposure of the adsorption surfaces (Donagemma *et al.*, 2008). The cationic exchange capacity (CTC) of laterite was considered medium, with good adsorption capacity because it has a load of 8.78 cmolc/L (Anghinoni *et al.*, 2013).

In order to determine the appropriate particle size for adsorption (Figure 1), the behavior of adsorption as a function of the independent variable (Figure 1a) and the desirability for adsorption (Figure 1b) were observed. The value zero (0) represents the maximum desirability, for the minimum adsorption of 25.92% and 1 represents the maximum desirability, for the maximum adsorption of 87.45%.

The overall desirability was 0.8 (Figure 1c), obtained by the geometric means of all desires, being very close to 1, which is the most desirable value. Thus, the optimization process points the 0.15mm granulometry as the most satisfactory (Figure 1c) in the adsorptive process, confirming that the smallest particle sizes have a greater adsorption capacity (Fischer *et al.*, 2019; Sekar *et al.*, 2004).

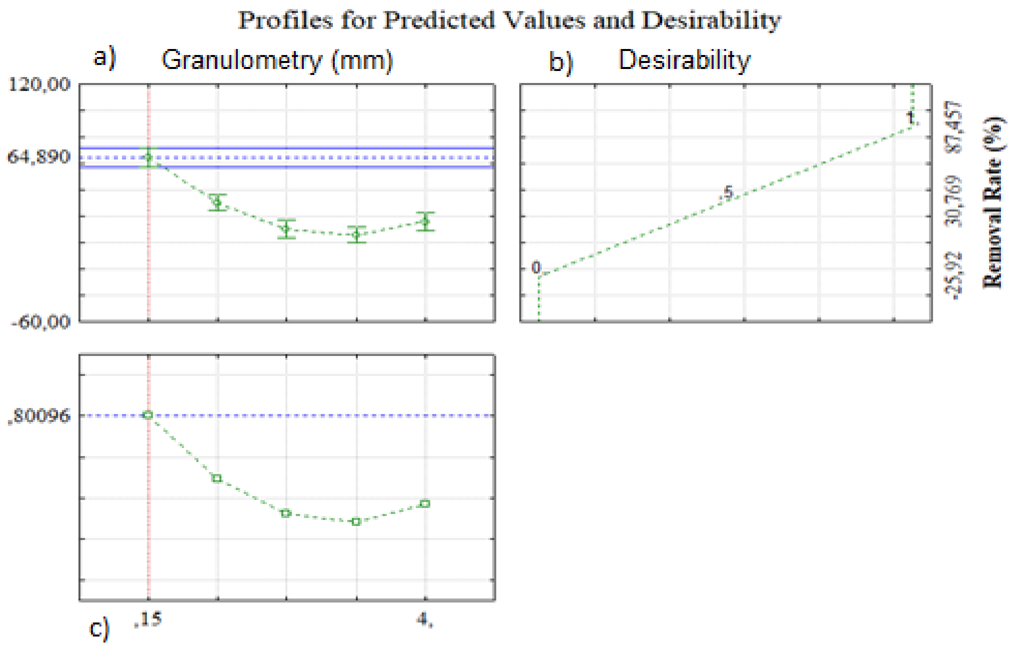

Figure 1. Profiles of predicted values and desirability for the adsorption capacity among laterites with 0.150mm, 2mm and 4mm grain sizes. Adsorption as a function of the independent variable (a); desirability for adsorption (b) and the overall desirability (c).

The interactions between the independent variables for the adsorption rate response (Figure 2) demonstrated the effect of the interaction between pH and adsorbent dosage: the best adsorption occurred at pH 4 (Figure 2a). The adsorptive processes tend to occur better in solutions with low pH (Coulibaly *et al.*, 2016; Mansing and Raut, 2013; Sato and Comerford, 2005), due to its influence on the availability of aluminum and iron ions present in the laterite, to react with phosphorus, by the electronegativity of the charges on the surface of the colloids of the adsorbent material, in this case, the oxides (Al_2O_3, Fe_2O_3).

A high pH conditions a deprotonation of the functional groups, affecting the surface load of the adsorbent (Sims and Pierzynski, 2005), decreasing the ability to exchange binders and decreasing the adsorption rate. In the interaction of the independent variables pH and contact time for the response variable, the removal rate (Figure 2b) did not show statistical significance as shown by the Pareto diagrams (Figure 3). The interactions of laterite dosage and contact time (Figure 2c) showed an adsorption that increased rapidly as the amount of laterite was increased, due to the greater availability of surface area. The highest adsorption occurred when the dosage was 15g of laterite and a contact time of 6 hours.

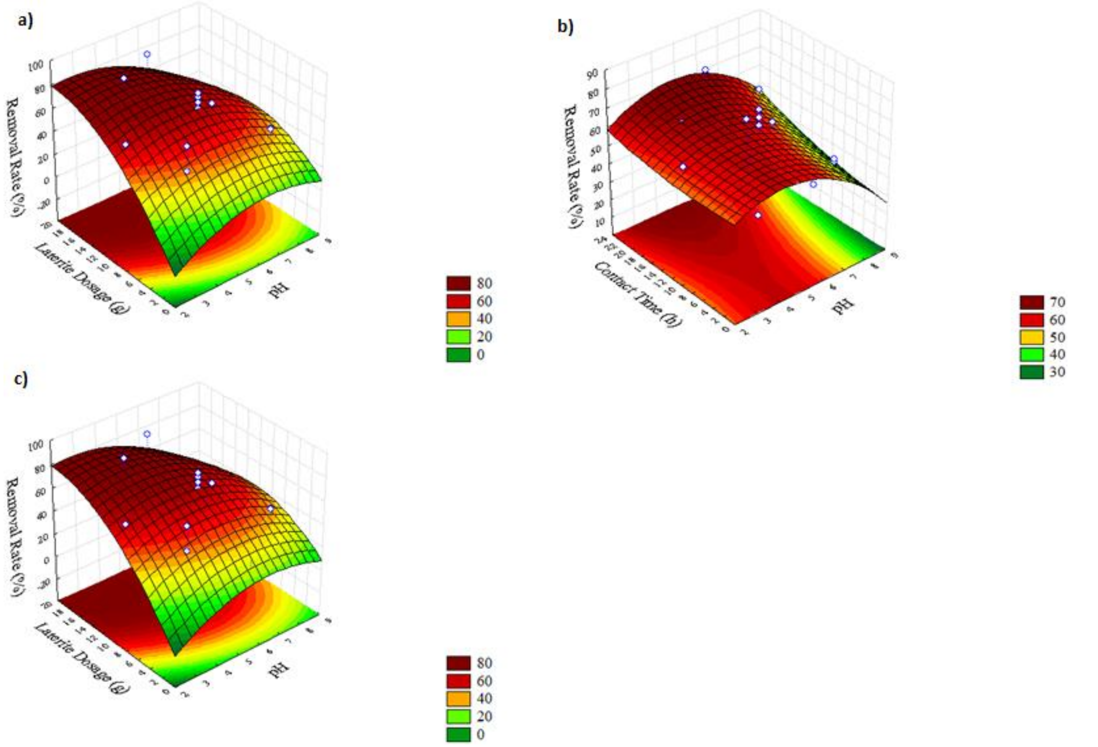

Figure 2. Response of influence of the interaction of the variables: a) dosage of adsorbent material and pH; b) pH and contact time; and, c) dosage of adsorbent material and contact time in relation to the removal rate of phosphorus.

The phosphorus adsorption efficiency was dependent on the particle size of the adsorbent material, since the smaller the particle, the greater the adsorption capacity, due to the greater availability of surface area susceptible to pollutant removal (Mansing and Raut, 2013; Worch, 2012; Tchobanoglous *et al.*, 2003). The smaller the adsorbent material particle, the greater its ability to adsorb (Fischer *et al.*, 2019; Sekar *et al.*, 2004).

The data from the Pareto diagrams (Figure 3) show that among the studied levels only the variable absorbent dosage had a significant effect on phosphorus removal. The laterite independent variable (Figure 3a and c), proved to be statistically significant, approaching 95% confidence. Still, for the removal of phosphorus, the variable contact time (Figure 3b) was not statistically significant, not showing relevance in the phosphorus adsorption process.

To determine the predicted and desirable profiles and values for this adsorption process, these interactions were studied: dosage of adsorbent and pH, contact time and dosage of adsorbent and pH, and contact time. The best conditions for phosphorus adsorption were obtained by simultaneous optimization, in which the most satisfactory responses will occur under conditions of pH 4, contact time of 2h and dosage of 18.36 g of laterite. The predictions assume that by performing an adsorption with these values, the adsorption rate will reach 85.5% of phosphorus removal (Figure 4). The maximum experimental adsorption capacity occurred with a removal rate of 87.9%, under the conditions of pH 5.3, contact time 18h and dosage of 15g laterite.

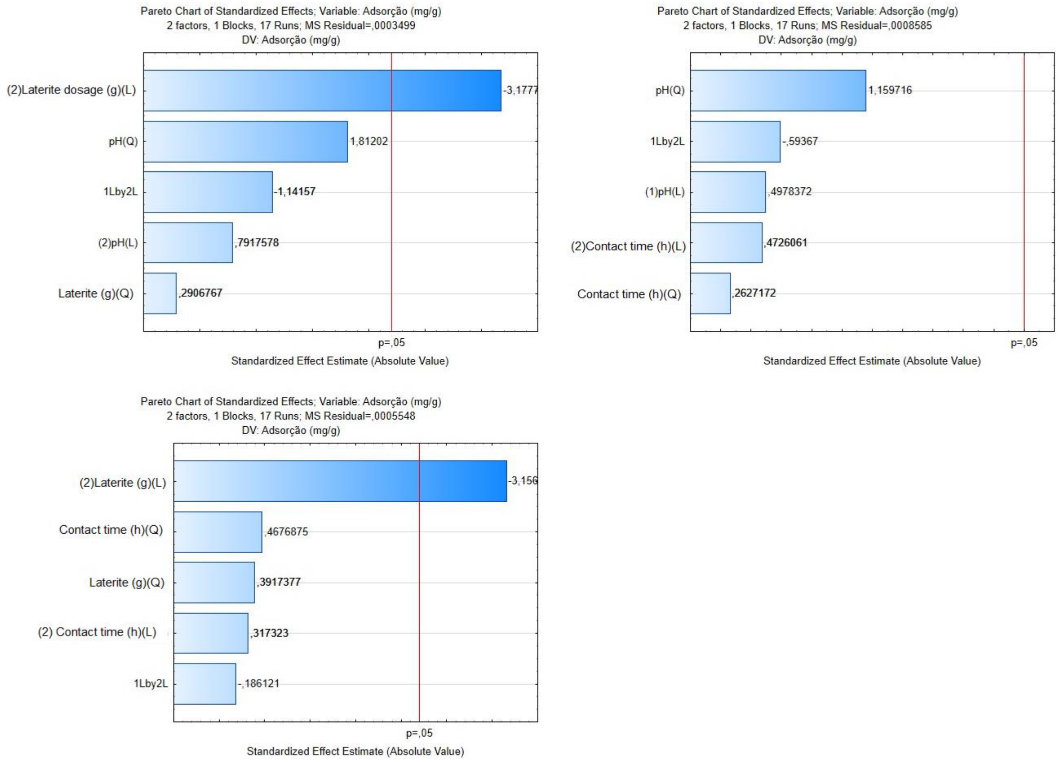

Figure 3. Pareto diagrams for the influence of the parameters: a) dosage of adsorbent and pH; b) pH and contact time; and, c) dosage of adsorbent and contact time in the adsorptive process.

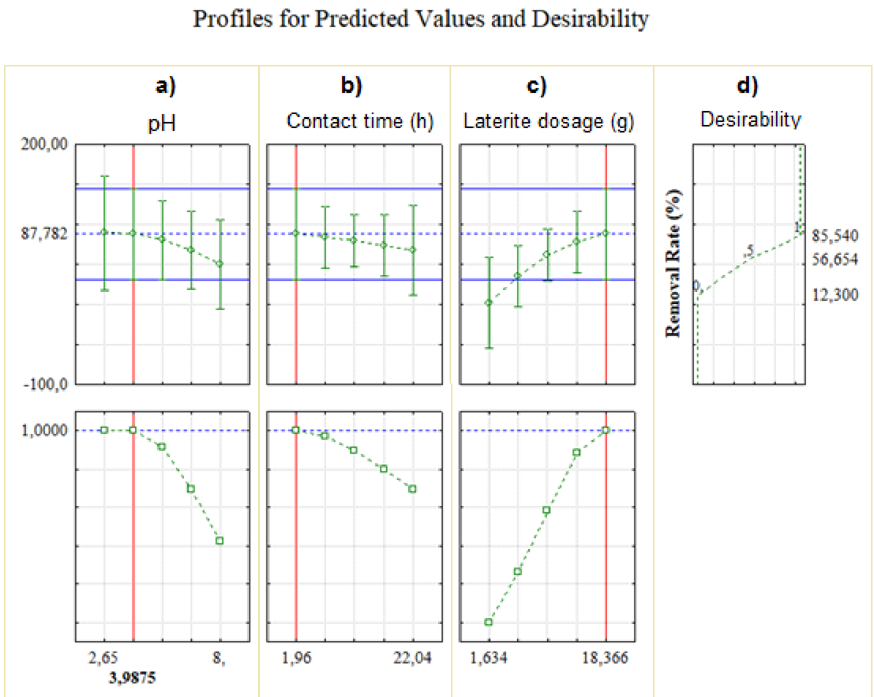

Figure 4. Predicted values for the desirability of the adsorption capacity (d) between the variables: pH (a), contact time (b) and dosage of adsorbent (c) in the adsorptive process.

The use of substrates rich in iron, aluminum and calcium increase the phosphate removal rate. Lateritic soils used as adsorbent material had a phosphate removal rate of 89% (Mansing and Raut (2013), 90.12% (Huang *et al.*, 2013) and 92.5% (Coulibaly *et al.*, 2016).

Regarding the adsorption isotherms (Figure 5), a very strong correlation was observed for the Langmuir adjustment r = 0.98 (Fig. 5a) and an average correlation for the Freundlich adjustment r = 0.71 (Figure 5b). The Langmuir model provided the best fit representing phosphorus adsorption on laterite to determine the coefficient of distribution to sorption. Langmuir's model is based on the assumption that a fixed number of sites available on the surface of the adsorbent have the same energy, and the adsorption is reversible (Bohn, *et al.*, 1979).

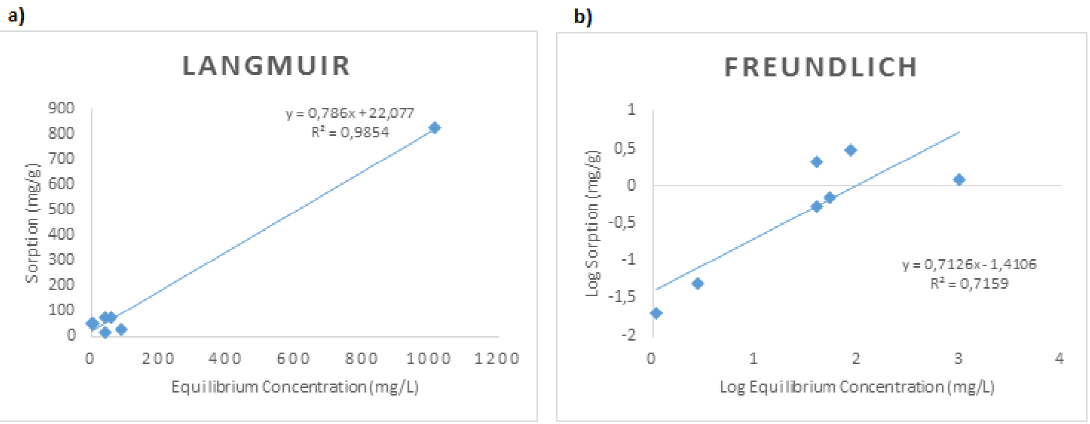

Figure 5. Phosphorus adsorption isotherm using the model of a) Langmuir and b) Freundlich.

In the Langmuir Equation, KL is the constant that presents the theoretical adsorption capacity of the monolayer and bL is the constant related to the adsorption energy. The values obtained by non-linear regression were 0.0325mL.g^{-1} and 0.0319mg.g^{-1} (Table 2), respectively, with favorable adsorption (RL), close to linearization.

The Freundlich constants, KF and bF, are empirical constants, where KF and bF are related to the adsorption capacity and binding energy of the adsorbent solute-surface, respectively, obtained by non-linear regression analysis, with values of 0. 00088mL.g^{-1} and 1. 3661mg.g^{-1} (Table 2), respectively. It expresses favorable adsorption, but is easily dissociated from the phosphate ions in the aqueous solution.

Table 2. Langmuir and Freundlich constants obtained from phosphorus adsorption results.

Adsorbent	**Langmuir**			**Freundlich**		
	K_L (mL.g^{-1})	b_L (mg.g^{-1})	RL	K_F (mL.g^{-1})	b_F (mg.g^{-1})	n
Laterite	0.0325	0.0319	0.99	0.00088	1.3661	1.36

It should also be noted that, in this study, a strong correlation was obtained for the Langmuir adjustment. The Langmuir Model presented an ideal type of adsorption, which implies that the molecules are adsorbed on the surface and that the energy of the sorbed species is the same at any point, regardless of the neighboring molecules, which represents an energetically uniform surface (Kumar *et al.*, 2008), so that the adsorbed phosphorus will be retained in a monolayer and does not have secondary adsorption sites, with a predominance of chemisorption.

The use of non-conventional and low-cost adsorbents, such as those obtained from the agricultural segments, household waste, by-products, natural materials, soil and ore, has been an alternative for wastewater treatment (Gisi *et al.*, 2016).

The laterite used, when saturated with phosphorus, can be used as a plant fertilizer, and only the adsorption process has the potential to recover P as a usable fertilizer, since P is a nonrenewable resource and is obtained by extraction from rocks (Sengupta and Pandit, 2011). The adsorbed phosphorus can be reused after the process of desorption, reuse or regeneration (Nguyen *et al.*, 2014).

Technologies that recognize logical factors, including community participation, public involvement, social perception, attitudes and public acceptance can lead to improvement in practical quality and wastewater management (Saad *et al.*, 2017).

Although there are many technologies for treating effluents, the sector still faces difficulties in implementing projects where social participation occurs, both due to the lack of initiatives on the part of local and regional governments and due to the lack of knowledge of the population on the subject, which inspires little interest in applied techniques (Rosenquist, 2005). Therefore, the use of simplified technologies will make it easier for the target community to connect with and understand these useful alternative technologies.

The use of laterite is highlighted as an alternative treatment that assists in fulfilling the needs of vulnerable populations through the management and use of technologies of lesser operational complexity (Saad *et al.*, 2017). The effectiveness of sanitation actions depends on the collaboration and participation of individuals or the community, from the definition of the principles and guidelines of a sanitation policy to the planning and execution of these actions (Silva and Naval, 2015).

4. CONCLUSION

Acidic pH favored adsorption, influencing the availability of ions present in laterite. The adsorption of phosphorus was dependent on the particle size of the adsorbent material; the smaller the particle, the greater the adsorption. The molecules were adsorbed on the surface and retained in a monolayer, with a predominance of chemisorption.

Laterite, without chemical modifications, was tested as an adsorbent material and proved effective for the removal of phosphorus in synthetic effluents, and can be used in filtering units to remove pollutants.

It must be taken into account that urban, rural or isolated spaces are heterogeneous, made up of different communities and specificities, which requires particular forms of intervention in basic sanitation, both for environmental and educational and technological issues. Simplified techniques are evaluated as a proposal for social technology in order to meet the demand of the population without access to water treatment and distribution.

5. ACKNOWLEDGMENTS

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