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Gastropod shell column reactor as on-site system for phosphate capture and recovery from aqua system

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ABSTRACT

Gastropod shell (GS), whose distribution is worldwide and mineralogical assemblage is aragonite, was packed in a glass column and evaluated for phosphate capture and recovery from agua system. The column reactor was operated in a batch-continuous mode, considered as a close approximation to a practical water treatment system, for 30 days. The time-concentration profile parameters were derived at different influent phosphate concentrations and hydraulic residence time (HRT) to determine the performance efficiency of the reactor column. The mechanism of phosphate removal was investigated and phosphate fractionation protocol was employed to elucidate the form and pattern of phosphate distribution in the spent reactor. Variations in influent phosphate concentration and HRT had minimal influence on the time-concentration profile parameters and the performance efficiency of the column reactor within the process variables studied. The first order rate constant (k_1) showed that increase in the HRT of the influent in the SS column reactor does not enhance the phosphate removal. The value of the saturation index (SI) obtained over the 30 days period were all positive and the thermodynamic parameter (ΔG) was <0 which is an indication of supersaturation, the significant role of precipitation as a mechanism of phosphate removal in the reactor and spontaneity of the precipitation reaction. The order of distribution of each fraction of P, relative to the total phosphorus (T-P) in the phosphate laden SS, is as follows: dicalcium phosphate (Ca₂-P) (42.5%)>ten-calcium phosphate (Ca₁₀-P) (40.50%)>octacalcium phosphate (Ca₈-P) (16.65%) > occluded phosphate (O-P) (0.35%). The simple appraisal of the fertilizer value of the spent reactor showed that it would preferentially serve as a soil conditioner cum fertilizer in acidic soil.

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1. Introduction

The negative environmental impact of euthrophicated water bodies has been the impetus for the continuous enterprise for the development of strategies for the removal of the causative agents. Phosphorus (P) is a limiting nutrient for eutrophication, thus, its enhanced removal from waste streams before discharge into receiving waterways is of considerable significance (Hecky and Kilham, 1988). Chemical precipitation and enhanced biological phosphorus removal (EBPR) are the two conventional approaches for phosphate removal in the water industry. Unfortunately, the recently published costs for some widely used cationic salts, required for chemical precipitation (Chemical Market Reporter, 2006), showed astronomical increase which might limit the continuous use of this approach. Furthermore, the procedure is encumbered with additional costs for the purchase and installation

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http://dx.doi.org/10.1016/j.ecoleng.2014.03.077 0925-8574/© 2014 Elsevier B.V. All rights reserved. of dosing equipment and high operational costs for power, disposal of additional sludge and manpower (Oladoja et al., 2012). EBRP can remove up to 97% of the total phosphorous load, but the process is highly unpredictable, due to fluctuations in operational variables (Clark et al., 1997). Rittmann et al. (2011) opined that the aforementioned conventional wastewater treatment techniques were developed for removing phosphate from municipal wastewaters, which contain modest concentrations of P (i.e. 5–10 mg/L). While they are effective at the job they were designed to do, they are not necessarily going to be effective for the job that our society needs to have done in the future: capturing the lost P from its large P flows, which have different characteristics than domestic wastewater. Besides, the primary goal needs to shift to capturing P so that it can be reused.

Four major approaches (precipitation, adsorption, ion exchange, and biological uptake) have been identified for removing inorganic P from the water stream in a form that can be reused. Phosphorus separation by adsorption during coagulation, followed by removal through sedimentation and filtration, was first used in the 1950s and has since been used to treat water and wastewater using a







wide variety of sorbents (Morse et al., 1998; Westholm, 2006; Goh et al., 2008; Lan et al., 2006; Mortula et al., 2007; Xiong and Peng, 2008). Premised on the perspectives of waste minimization and cost reduction in the treatment of phosphate contaminated water, a Gastropod shell (African land snail, *Achatina achatina*) was evaluated in our laboratory as a low cost sorbent in batch treatment of phosphate contaminated water and reported (Oladoja et al., 2012). The theoretical basis for the choice of Gastropod shell was predicated on the global pervasiveness and the chemical and mineralogical assemblage (Oladoja et al., 2012).

Over the years, batch adsorption studies have been the most popular procedure employed for screening and evaluating lowcost sorbents in the removal of pollutants from aqua system. Batch adsorption experiments can easily be used in the laboratory for the treatment of small volume of effluents, but are less convenient to use on an industrial scale, where large volumes of wastewater are continuously generated (Oladoja et al., 2008). Gravity percolation of wastewater through one kind or another of percolator material is a common on-site wastewater treatment technology that holds promises and is being continuously studied for pollution abatement (Oladoja and Ademoroti, 2006). During the transit through the percolator, the wastewater is purified by physico-chemical (filtration, adsorption) and biological (microbial degradation) processes (Lens et al., 1994). Recently, the use of reactive alkaline based filtration media for phosphate removal and recovery from aqua stream (Johansson and Gustafsson, 2000; Drizo et al., 2006; Shilton et al., 2006; Adam et al., 2007) has shown promise as a pivot on which future strategies for eutrophication mitigation is to be based. The striking feature of this reactive filtration technique is the potential application of the nutrient-loaded filter media in agriculture as P fertilizer and soil conditioner (Oladoja et al., 2012; Hylander et al., 2006a,b).

Premised on the promising results obtained from our laboratory on the use of Gastropod Shell, in a batch system, as a low cost material for the removal and recovery of phosphate from aqua system (Oladoja et al., 2012), the present studies aimed at the use of gastropod shell as a reactive filtration medium in an on-site system for the removal and recovery of phosphate from agua system. On-site treatment of waste is a decentralized wastewater treatment system that requires simple, reliable, low-energy consuming and low-cost technology that private homeowners with little skills for operations can afford (Schudell and Boller, 1989). The reactive columns shall be charged with polluted water and discharged at fixed hydraulic residence time (HRT) and then recharged, instead of a continuous flow column protocol with fixed flow rates. Nath and Dutta (2012) posited that this column reactor could be considered as a close approximation to a practical water treatment system where the water can have a long contact time with the filtration medium.

In the present studies, the time-concentration profile parameters of the phosphate removal process in a Gastropod shell column reactor shall be studied. The effect of the influent HRT on the phosphate removal process and the time-concentration parameters shall be evaluated, the mechanism of phosphate removal shall be elucidated and the phosphate moieties in the reactive medium shall be fractionated to understand the form and pattern of the phosphate distribution in the reactor.

2. Materials and methods

2.1. Gastropod shell preparation and characterization

African land snail shell (SS), whose some of the physicochemical parameters have been determined and reported in our earlier exposition was the Gastropod shell used and was prepared as previously enunciated (Oladoja and Aliu, 2009; Oladoja et al., 2011). The surface functional groups of the SS were determined using FTIR spectrophotometer (Thermo Scientific, USA) while the surface architecture and surficial elemental composition were determined using scanning electron microscope (SEM), equipped with energy dispersive analysis of X-ray (EDAX). The textural characteristics of the SS were determined using nitrogen absorption procedure, to determine the specific surface area and the pore diameter using an ASAP Micromeritics instrument by Brunauer–Emmett–Teller (BET) method (1938), and the Barrett–Joyner–Hanlenda (BJH) method (1951) was used to calculate the mesopore distribution. The thermogravimetric analyses of the SS were carried out in Perkin Elmer model TG analyzer.

2.2. Column reactor treatment procedure

Potassium dihydrogen phosphate (KH_2PO_4) was used as the source of phosphate and the stock orthophosphate solution of 2000 mg/L was prepared with laboratory grade water while working solutions of different orthophosphate concentrations (25–500 mg/L) were prepared, as required, from the stock by serial dilution.

The phosphate removal experiments were carried out in a glass column (Scheme 1) of length 54 cm and diameter 3 cm. Granular SS particles, whose particle was retained in a 2.36 mm sieve were packed in the glass column via the dry packing technique. The phosphate contaminated water was put into the SS column reactor (column height = 35 cm, SS weight = 372 g) to fill up the vacant space up to the top level of the SS column. The total volume of water in the SS column each time was 120 ml. The columns were charged with water and discharged completely at fixed influent HRT (6, 12 and 24 h), subject to the variable under investigation and then recharged instead of a continuous flow column procedure with fixed flow rate. The recharge and discharge of the column were repeated continuously until a fixed volume of phosphate contaminated water was treated, with the same SS column. The phosphate concentration and pH at each sampling time were determined.

The effluent phosphate concentration was determined by the molybdenum-blue ascorbic acid method, with a UV–vis spectrophotometer (at λ_{max} = 880 nm), while the effluent pH was determined using a laboratory pH meter (Hanna, pH211 microprocessor).

2.3. Fractionation of the P-saturated SS

The forms and pattern of phosphate distribution in the SS reactor was studied via a protocol, initially proposed by Chang and Jackson (1957) and subsequently modified by Gu and Jiang (1990). This method is normally used to fractionate P in calcareous soil, which includes different operationally defined phosphorus fractions.

Phosphate saturated SS was obtained by weighing 10.0 g of SS into a 1 L Erlenmeyer flask, and 500 ml of phosphate solution (500 mg/L) was then added, and agitated for 24 h. The phosphate adsorbed SS was washed with distilled water until no phosphate can be detected in the filtrate and dried at 40 °C for 8 h.

P-saturated SS (0.50 g in dry weight) was added into 50-ml polyethylene centrifuge tubes containing 25 ml of 0.25 M NaHCO₃ (pH 7.5). The mixture was agitated on an orbital at 25 °C for 1 h, and the supernatant was separated by centrifugation. The supernatant was filtered and then analyzed for dicalcium phosphate (Ca₂-P).

The residue was washed twice with 95% ethanol (each wash was 12.5 ml). The supernatant was centrifuged and discarded before 25 ml of 0.5 M NH₄ acetate (pH 4.2) was added. The mixture was left

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