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Nutrient Recovery and Recycling from Human Urine: A Circular Perspective on Sanitation and Food Security

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Abstract

Closed-loop nutrient cycling is a simple, persuasive and elegant approach to realize efficient natural resource management, improved human well-being and long-term food security. In the spirit of sustainable sanitation through nutrient cycling, this study proposes a new pathway to realize resource recovery from anthropogenic waste fractions by the application of physico-chemical separation processes. Microwave Activated Carbon (MAC) prepared from coconut shells (agro-waste) were immobilized on etched glass bead supports and utilized within a continuous flow packed-bed column. Physical adsorption experiments were performed by passing human urine through the column to strip and recover more than 80% of the intrinsic urea. Backwashing of the column was performed to demonstrate the ease of urea-N desorption, reusability of MAC over multiple cycles and the reversible nature of the process. Further nutrient recovery was realized by dephosphatizing the column overflow with MgO to allow phosphate precipitation (>90%) as struvite. Sorption kinetics, behaviour and influence of process parameters were studied by testing the experimental data against Yoon-Nelson, Thomas and Adams-Bohart models. The column adsorption was also numerically optimized using Response Surface Methodology to determine the optimal parameters as: sorbate flow rate – 8.5 L.h⁻¹, urea concentration – 100%, support size – 1 cm and consequently, column capacity of 21.58 g. Nutrient recovery, concentration and recycling from diverted human urine can be seen as a synergistic and circular solution to the issues of sanitation, hygiene, water, and food security.

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1. Introduction

The manifestations of rapid population growth, urbanization, improved standards of living and concurrent intensification of socio-economic activities on the overall environmental health are well-recognized and acknowledged today [1-4]. However, technological solutions that are aimed at addressing this deteriorating state of our life support systems fail to recognize the dynamic interconnectedness between its various aspects such as, food security, hygiene, water scarcity, sanitation, resource conservation, waste management, etc. and fall short of satisfying the imperative of sustainable development. Moreover, in various instances, despite conceptual realization, the implemented solutions to overcome our issues do not achieve systemic synergy; conventional models have failed to demonstrate sustainability in their operation [5] and hence, the search for holistic solutions that simultaneously address several thematic concerns continue.

The water, sanitation and hygiene nexus in the developing world offers a promising platform to develop and implement sustainability-centric solutions. Further integration can be achieved within this nexus by looking at it through the lens of sustainable agriculture. The notion of closed-loop fertility cycle claims to provide a simple, persuasive and elegant approach to realize efficient natural resource management, improved human well-being, and long term food security [5,6,7]. It entails the use of urine diversion toilets that provide not only access to sanitation but more importantly, segregation of human waste fractions at source [8]. Recent research effort has been focused on developing cost-efficient and implementable technologies to concentrate, recover and recycle nutrients from the waste fractions to shift towards a more circular way of resource management [9-13]. Given the variability in effectiveness of nutrient recovery demonstrated by these studies, it thus becomes necessary to investigate alternative resource recycling pathways.

Through previous batch investigations on urine and urea recovery therein, we have demonstrated that nutrient recovery can be attained by the application of agro-waste sourced activated carbon [14-18]. Specifically, *Cocos nucifera* shells were demonstrated as an effective adsorbent to recover more than 90% urea from human urine [15,18]. In the present study, to investigate the ease of implementability and scale-up of this process, we employ a continuous packed-bed column that treats a larger amount of influent urine using a predefined quantity of the adsorbent. Further, an initiative was undertaken to reduce the process energy requirements by minimizing the systems' adsorbent loading. To do this, immobilization procedures were performed on the prepared carbon using etched glass beads as the support. Immobilized column adsorption is well suited for non-destructive resource recovery, is resistant to chemical toxicity and has been shown to enhance sorption selectivity and effectiveness [19]. Hence, the objectives of this study are to (i) examine the urea and phosphate removal efficiency using a continuous, immobilized packed-bed column; (ii) analyze the effect of various process parameters, viz., initial concentration, flow rate and size of sorbent support on the removal efficiency (iii) model the column breakthrough and sorption kinetics (iv) optimize the process parameters by using Response Surface Methodology (RSM).

2. Experimental

2.1. Sorption raw materials and setup

Human urine was obtained from 40 healthy male volunteers (aged 18-21) for a period of 45 days. The samples were collected in polypropylene containers and stored at -20°C to inhibit NH_3 volatilization. Before use, the urine was thawed to room temperature ($23\pm 1^{\circ}\text{C}$), mixed and characterized; the initial urea and phosphate concentration were determined to be $18750\text{ mg}\cdot\text{L}^{-1}$ and $595\text{ mg}\cdot\text{L}^{-1}$, respectively. Microwave Activated Carbon (MAC) was prepared from waste *Cocos nucifera* shells and immobilized onto etched glass beads as described in earlier studies [14,15,18]. All reagents and chemicals (analytical grade) were purchased from Sigma Aldrich Chemicals, Mumbai India. The sorption experiments were performed in a Pyrex glass column (ϕ 4 cm and height 80 cm), randomly packed with immobilized MAC beads. The effect of the following process parameters on urea uptake capacity of MAC was investigated for sorbate flow rate ($1\text{-}10\text{ L}\cdot\text{h}^{-1}$), initial urea concentration (20-100%), and size of MAC support (ϕ 1.5, 2 and 2.5 cm). Effluent urea concentration was measured at different time intervals by analysing 3 mL aliquots through UV-vis Spectrophotometry (Shimadzu UV-1601, Japan) at 430 nm [19]. Upon saturation of the column, the MAC was regenerated by pumping deionized water at $2\text{ L}\cdot\text{h}^{-1}$ in up-flow mode to recover the adsorbed

urea. Regeneration was terminated when a 10% loss in sorption capacity of the regenerated MAC was recorded. For P recovery, after each experimental run, the urine was dephosphatized by addition of 0.25, 0.75 and 1 g MgO.L⁻¹ to precipitate struvite [Mg(K,NH₄)(PO₄)·6H₂O] as described by Ganrot et al. [10].

2.2. Sorption design, optimization and data analysis

In a dynamic packed-bed column, the adsorption zone moves down with time until it attains breakthrough where, exit concentration equals that of the feed [20]. In order to understand the performance of the column, the time required for column breakthrough (t_b) was observed and breakthrough curves were plotted to express the relationship between inlet (C_0 ; mg.L⁻¹) and outlet urea concentration (C ; mg.L⁻¹). Urea adsorbed by the column (C_{ad} ; mg.L⁻¹) was calculated as the difference between the inlet and outlet concentration. The column capacity (q_c ; mg.g⁻¹) was estimated by Eq. 1 where A is the area under the curve and Q (mL.min⁻¹) is the urine flow rate. The equilibrium sorption (q_e ; mg.g⁻¹), length of mass transfer zone (Z_m ; cm), Empty Bed Residence Time (EBRT; min) and total urea fed to the column (m_{total} ; g) were calculated by Eq. 2-5, respectively. Moreover, the sorbate-sorbent interaction was determined by non-linear regression of the column sorption data against Adams-Bohart, Thomas and Yoon-Nelson models [21]. To ensure accuracy of the results and their reproducibility, experiments were done in triplicate with statistical analysis performed using MATLAB[®]. The coefficient of determination (R^2), Root Mean Square Error (RMSE) and chi-square (χ^2) were the primary criteria to test the goodness of fit of the sorption data for all the models [22].

$$q_c = \left(\frac{Q A}{1000} \right) = \frac{Q}{1000} \int_0^{t_{total}} C_{ads} dt \quad (1)$$

$$q_e = \left(\frac{q_c}{X} \right) \quad (2)$$

$$Z_m = Z \cdot \left(1 - \frac{t_b}{t_e} \right) \quad (3)$$

$$EBRT = \frac{\text{Bed volume}}{\text{Volumetric flow rate of the liquid}} \quad (4)$$

$$m_{total} = C_0 \cdot Q \cdot \left(\frac{t_{total}}{1000} \right) \quad (5)$$

Further, nutrient recovery from the column was optimized by Response Surface Methodology (Design-Expert[®] V.9, MN, USA) by using a three-factor and three level (-1, 0, +1) central composite design. The following variables were considered during optimization – sorbate concentration: X_1 (20, 40 and 100 mg.g⁻¹), sorbate flow rate: X_2 (2, 6 and 10 L.h⁻¹), and MAC support size: X_3 (1.5, 2 and 2.5 cm) and the experimental design followed is presented in Table 1 and Table 2. The quadratic polynomial regression model employed in this study is presented in Eq. 6.

$$Y = \beta_o + k \sum_{i=1}^3 \beta_i X_i + \sum_i^3 \beta_{ii} X_i^2 + \sum_{i=1}^2 \sum_{j=i+1}^3 \beta_{ij} X_i X_j \quad (6)$$

β_o , β_i , β_{ii} and β_{ij} are the regression coefficients for intercept, linear, quadratic and interaction terms, respectively; X_i and Y_j are the coded values of the process variables while k is the number of tested factors (See Table 1). Analysis

of variance (ANOVA) and Fischer-Test were performed using Minitab® (V. 15.1, State College, PA, USA) to check the statistical significance and the goodness of fit was expressed by coefficient of determination (R^2) [23].

3. Results and Discussion

3.1. Effect of process parameters on urea sorption

The column breakthrough curves are presented in Fig. 1 and were studied at various inlet urea concentrations (20-100%) and sorbate flow rates (1-10 L.h-1). At all conditions examined, the sorption demonstrated a smooth sigmoid-shaped breakthrough from the column [24]. The effect of initial concentration (Fig. 1(a)) was examined at an inlet flow rate of 5.5 L.h-1, bed height of 65 cm and MAC support size (ϕ) of 1.5 cm. The EBRT in the column, estimated using Eq. 4 was found to be 8.9 min. The curves depicted a relatively early breakthrough with breakthrough time (t_b) varying inversely with concentration; t_b decreased from 45 to 22 min as urea concentration was increased from 20 to 100%. Similarly, the curves were sharper at higher concentration pointing towards a strong dependence of MAC's urea uptake capacity on the initial sorbate concentration [21]. This inverse dependency is possibly due to the availability of a larger concentration difference or lesser mass transfer resistance that subsequently reduces the length of the adsorption zone [25]. Further, the urea sorption capacity of MAC also increased from 71.27 to 333.95 mg.g-1 with corresponding increase in concentration. Fig. 1 (b) is representative of the experimental breakthrough for the five flow rates investigated. As seen in the figure, t_b shifted towards the origin at higher flow rates as the gradient of the curves became steeper. Higher inlet flow rates allowed for lesser contact time of urine with the immobilized MAC and hence, resulted in earlier breakthrough; t_b decreased from 68 to 45 min as flow rate increased from 1 to 10 L.h-1. A corresponding increase in sorption capacity of MAC from 40.78 to 382.08 mg.g-1 was also observed. This can be attributed to an increase in fluid turbulence within the column that allows the sorption to overcome its mass transfer resistance. However, increasing the flow rate from 1 to 10 L.h-1 results in decrease of EBRT from 49 min to 9 min and hence, lowers the urea removal efficiency of the immobilized carbon [26]. In addition, the effect of glass bead support size was investigated at three levels (ϕ 1.5, 2 and 2.5 cm) with constant inlet flow of 10 L.h-1 and urea concentration of 60% (Fig. not shown). Relatively early breakthrough and shorter t_b was noticed for the smaller beads; the supposition here is that smaller beads allow for greater sorbate-sorbent interaction as they have higher surface area per unit volume of MAC and hence, result in better urea uptake. Similar observations have also been made by Ko et al. [27] in their study of metal adsorption onto char.

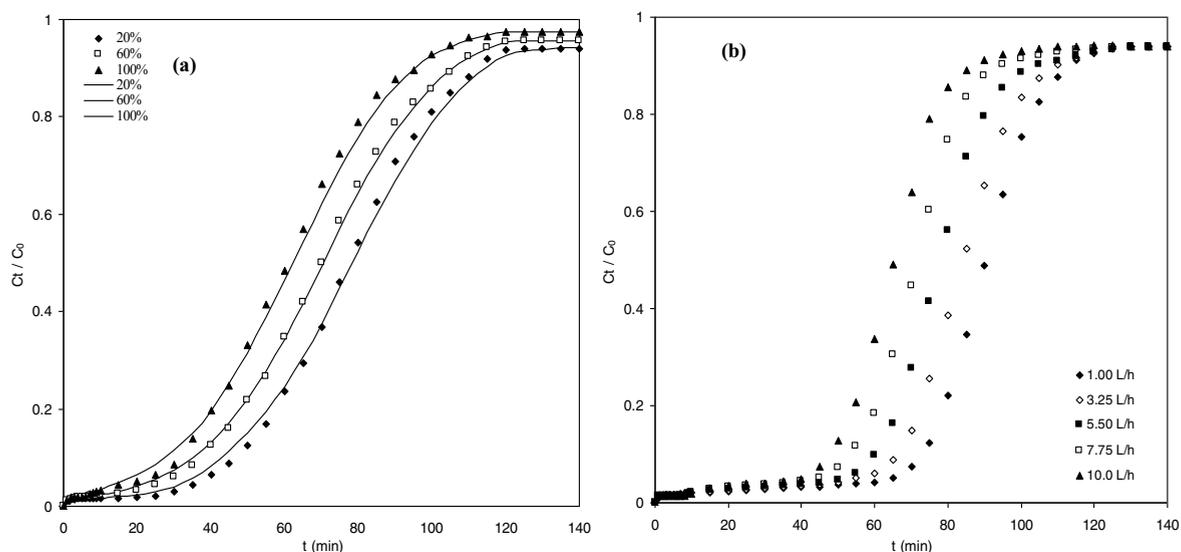


Fig. 1: Effect of initial concentration (a) and flow rate (b) on column breakthrough; Symbols: experiment and lines: Yoon-Nelson model

3.2. Kinetic modeling of the column breakthrough

To describe the sorption behavior of MAC in the column, experimental data was subjected to non-linear regression against three well-known empirical models. While a good fit was seen for all the tested models, based on the R^2 and χ^2 values, Yoon-Nelson was found to better represent the urea sorption onto immobilized MAC. To illustrate this, Fig. 2 was plotted to depict the goodness of model fit for a column of bed height 65 cm, flow rate of 10 L.h⁻¹, inlet urea concentration of 60% and MAC support size of ϕ 1.5 cm.

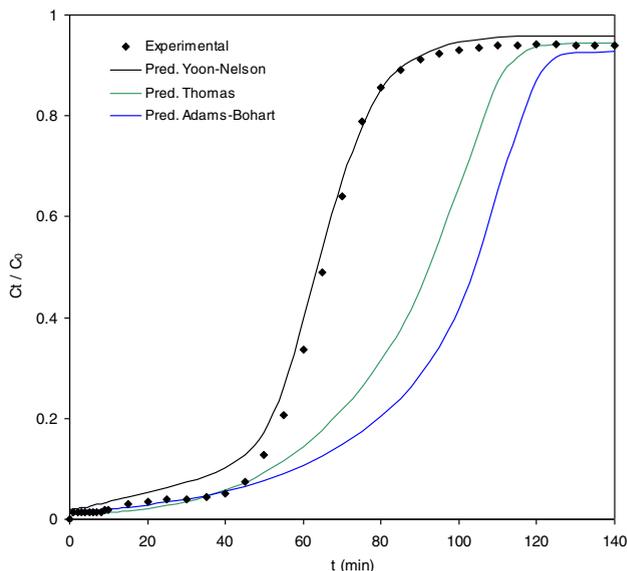


Fig. 2: Comparison of experimental (symbols) and predicted breakthrough (lines) for Yoon-Nelson, Thomas and Adams-Bohart models

As seen through Table 1, the rate velocity constant (k_{YN}) decreased with sorbate flow rate and increased as inlet concentration was increased. A good agreement of the experimental breakthrough time (t_b) and τ estimated by the Yoon-Nelson model was also observed. Similarly, k_{Th} and k_{AB} were also found to decrease at higher flow rates to the column. The application of the Adams-Bohart model indicated that the maximum saturation capacity (N_0) increased with flow rate as well as initial concentration. N_0 was estimated to be 297.5 mg.L⁻¹ for Run 7 wherein inlet concentration was 100%, feed flow rate was 10 L.h⁻¹ and support size was 1.5 cm. Aksu and Gönen [19] as well as Ahmad et al. [21] reported similar findings in their kinetic studies on immobilized activated sludge adsorption.

Table 1. Kinetic model parameters and goodness of fit for the experimental column data; *Experimental runs are presented in Table 2

Run*	Yoon-Nelson Model					Thomas Model					Adams-Bohart Model				
	k_{YN}	τ	R^2	RMSE	χ^2	k_{Th}	q_0	R^2	RMSE	χ^2	k_{AB}	N_0	R^2	RMSE	χ^2
1	0.055	67.60	0.959	5.92	5.99	6.1E-05	303.5	0.888	10.8	11.1	3.5E-05	40.13	0.904	9.55	10.1
2	0.060	42.11	0.964	4.74	5.13	5.6E-05	362.1	0.949	6.55	6.94	3.2E-05	43.66	0.919	8.88	8.93
3	0.054	72.10	0.921	10.1	10.2	2.5E-05	265.2	0.820	14.0	14.7	3.7E-05	39.55	0.906	9.61	10.0
4	0.071	46.39	0.966	4.65	9.16	5.8E-06	470.0	0.882	9.98	10.3	3.3E-06	234.1	0.901	7.18	8.31
5	0.062	55.63	0.947	6.47	6.90	4.2E-06	376.0	0.743	15.9	17.1	3.1E-06	148.4	0.885	10.9	11.2
6	0.052	72.15	0.933	8.11	8.70	3.3E-06	338.0	0.863	7.54	7.78	3.0E-06	29.12	0.940	7.94	8.01
7	0.046	21.38	0.924	9.11	9.43	2.2E-05	584.2	0.824	13.5	14.2	3.0E-06	297.5	0.868	7.55	7.82
8	0.063	25.22	0.931	7.15	7.62	3.3E-06	548.4	0.911	9.12	9.55	3.0E-06	239.3	0.877	7.41	7.51

3.3. Column regeneration and phosphate recovery

The saturated MAC in the column was regenerated through urea desorption and backwashing with deionized water at 2 L.h⁻¹. The cut off value for backwashing was fixed at 10% of the maximum urea uptake capacity of MAC and it was found that this occurred at the end of 8th adsorption-desorption cycle. Elution with deionized water allowed for more than 90% recovery of the adsorbed urea in less than 30 minutes of operation pointing towards the reversible nature of the sorption. Further, the urea-N stripped urine, collected as column overflow at the end of each experimental run was subjected to struvite precipitation by addition of MgO for phosphate recovery [10]. Addition of MgO led to a corresponding increase in urine pH (ca 9.1–10.2) as well as P removal efficiency. Maximum precipitation was seen in the urine collected at the end of run 7 and 8, indicating that higher inlet flow rate and initial urine concentration allow for better post-adsorption, P recovery. Similar to the observations made elsewhere [28], 95% of the phosphate was removed as precipitated struvite.

3.4. Process optimization

The complex interactive effects of the investigated process variables on urea uptake capacity of the column were studied using RSM. Multiple regression analysis and fitting of the experimental data from Table 2 against Eq. 2 followed by an adequacy check allowed for the development of the adjusted model equation (Eq. 7) wherein, only statistically significant variables ($P < 0.05$) were considered [23]. The accuracy of this model was verified through coefficient of determination ($R^2 > 0.9$) and lack of fit (Probability $> F$ was < 0.001) values [29]. The model suggested that initial urea concentration – X_1 , sorbate flow rate – X_2 , MAC support size – X_3 , two-level interaction of flow rate and support size – X_2X_3 and the quadratic effect of concentration – X_1^2 strongly influenced the column capacity. Further, the 3-D response surfaces illustrated in Fig. 3 depicted the contribution of any two variables on the uptake while the third was kept at zero level; the sensitivity of the sorption to X_1 and X_2 was evident in the plots. Moreover, the relatively insignificant effect of support size in comparison to the other parameters was also evident (Fig. 3 (b)).

$$Y = 12.007 + 0.2062X_1 + 3.9854X_2 - 2.8614X_3 - 1.2442X_2 \cdot X_3 - 7.6349X_1^2 \quad (7)$$

Table 2. Optimization of urea sorption onto MAC using Central Composite Design: predicted and experimental column capacities

Run	Coded Variables			Real Variables			Y: Column Capacity (q_c)	
	X_1	X_2	X_3	X_1	X_2	X_3	Predicted	Observed
1	+1	-1	-1	10.0	20	1.5	3.951	3.636
2	0	-1	0	5.50	20	2.0	4.128	4.276
3	-1	-1	+1	1.00	20	2.5	1.395	1.112
4	+1	0	-1	10.0	60	1.5	16.95	17.92
5	0	0	0	5.50	60	2.0	12.01	12.44
6	-1	0	+1	1.00	60	2.5	4.231	4.166
7	+1	+1	-1	10.0	100	1.5	18.23	18.11
8	0	+1	-1	5.50	100	1.5	16.65	16.09

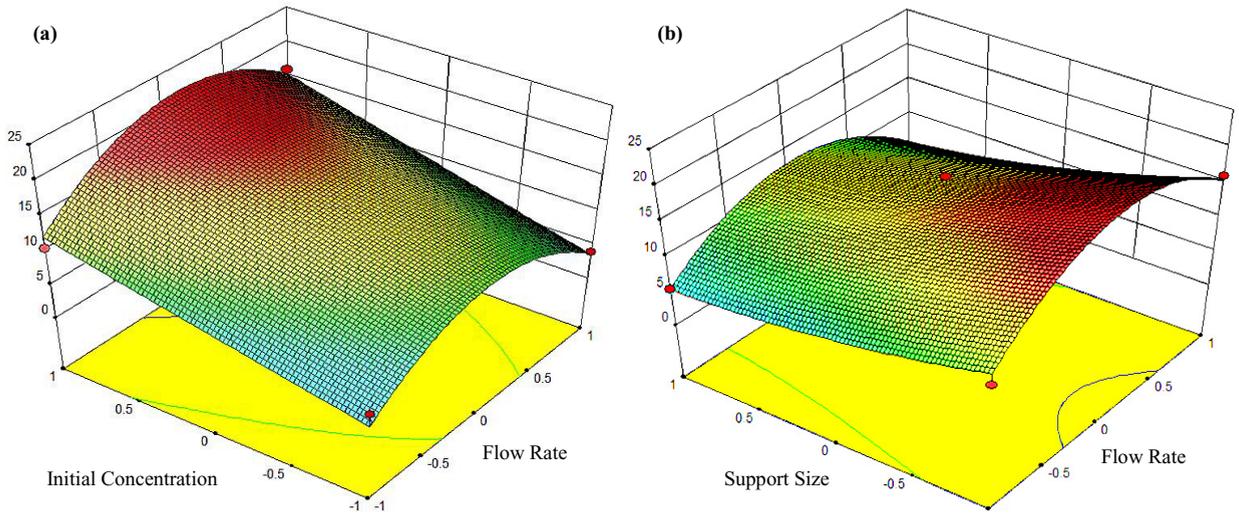


Fig. 3: Comparison of experimental (symbols) and predicted breakthrough (lines) for Yoon–Nelson, Thomas and Adams–Bohart models

4. Conclusions

The present investigation demonstrated both the applicability and effectiveness of agro–waste sourced activated carbon towards nutrient recovery from human urine. Based on the results of a continuous column adsorption study it was found that, the urea–N recovery and column breakthrough were strongly influenced by the initial urea concentration, urine flow rate and support size of the immobilized MAC. Further, following urea–stripping from the column, dephosphatization with MgO allowed P recovery as precipitated struvite ensuring greater efficiency of nutrient recovery in the process. Kinetic modeling of the process indicated that it could be best described by the Yoon–Nelson model. Lastly, the sorption was numerically optimized through central composite design in RSM to yield the following parameters: sorbate flow rate – 8.5 L.h⁻¹, urea concentration – 100%, support size – 1 cm and subsequently, column capacity of 21.58 g. Nutrient recovery, concentration and recycling from diverted human urine can thus be seen as a sustainable and circular solution to the issues of sanitation, hygiene, water, and food security.

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