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Continuous versus batch contacting for nutrient recovery from human urine upon processing with clinoptilolite I: surface capacities and operation times

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ABSTRACT

Processing source separated urine with clinoptilolite is one effective method for nutrient recovery from urine where ion exchange/adsorption is the predominating process. Although this method can be applied in either batch or continuous mode, almost all work in the literature has been focused on the batch mode. The aim of this work was to determine and to compare surface capacities and operation times for both of these two modes to identify the more effective one for nutrient recovery under similar conditions. Using initial ammonium loadings between 10–40 mg ammonium/g clinoptilolite for the batch mode and empty bed contact times between 30–120 min for the continuous mode, it was observed that higher surface capacities and lower operation times were achieved in the continuous mode while higher efficiencies and lower residual concentrations were observed for the batch mode. From the stand point of surface capacities and operation times, continuous mode seems preferable over batch operation for nutrient recovery from human urine with clinoptilolite through ion exchange/adsorption.

Keywords: Nutrient recovery; Human Urine; Fertilizer; Clinoptilolite; Ion exchange/adsorption; Batch and continuous modes

1. Introduction

A recent approach to domestic wastewater management is stream segregation at the source which enables the reuse of each fraction, and therefore helps sustainability of resources. One of those streams, yellow water, is human urine, which is a renewable and recyclable material. Its rich nutrient content in terms of nitrogen, phosphorus and potassium makes it a valuable alternative source of fertilizers through either direct or indirect use. Processing it with the natural zeolite clinoptilolite is an effective method of recovering plant nutrients for further use as fertilizer. In this process, nutrients in urine are transferred onto zeolite, subsequently to be released from the surface and made available for plant use.

As clinoptilolite is highly selective towards ammonium, ammonium removal with different clinoptilolites was discussed in the literature from various liquid phases like pure/synthetic solutions, domestic wastewater, landfill leachate, etc. using Bulgarian [1–4], South African [5], Australian [6,7], Mexican [8], Chilean [9], Turkish [10–15], etc. clinoptilolites. The highest values of surface concentrations reported in these papers are 27.9 mg ammonium/g clinoptilolite (1.55 meq/g clinoptilolite) for the Mexican [8] and 27.36 mg ammonium/g clinoptilolite (1.52 meq/g clinoptilolite) for the Turkish clinoptilolite [10].

There are various experimental results to justify that the mechanism of ammonium removal is ion exchange when ammonium containing solutions are contacted with clinoptilolite. As examples, it was demonstrated by Yurtoglu [12] that as an average, 86% of ammonium removal occurred through ion exchange, while Allar [16] provided quan-

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titative data involving ionic concentrations to show that ammonium ions were exchanged for sodium ions at a ratio of 98% when the sodium form of conditioned clinoptilolite was used as the solid phase with human urine.

Hedstrom and Amofah [17] used a Turkish clinoptilolite from Gordes region for phosphate removal, and reported a maximum phosphate removal of 16% for filtered domestic wastewater with an initial concentration range of 1.8–2.4 mg PO_4-P/L. Sari [13] observed 20% phosphate removal from domestic wastewater for 1.0–2.0 mg PO_4-P/L initial concentration with clinoptilolite from the same region upon conditioning with sodium chloride. The maximum phosphorus removal capacity was reported for a Slovakian clinoptilolite by Chmielewska et al. [18] as 12 mg/g clinoptilolite for 1400 mg/L liquid phase concentration. It is to be noted that surface capacities in ion exchange/adsorption are dependent upon initial concentrations and Chmielewska et al. [18]'s initial concentration is extremely high in comparison with domestic wastewater as well as human urine which has a typical concentration range of 250–415 mg/L [19,20] when freshly excreted. Furthermore, Chmielewska et al. [18]'s work was done using a pure solution of phosphorus as the liquid phase in the absence of competition with others.

Although ion exchange/adsorption was used successfully in some pieces of work for removal of phosphorus only as exemplified by O'Neal and Boyer [21,22] with anion exchange resins, it is an effective method of recovering both nitrogen and phosphorus simultaneously for further use when the natural zeolite clinoptilolite is employed as the solid phase. In this case, both nitrogen and phosphorus in urine are transferred onto clinoptilolite, to be subsequently made available to plants through desorption upon contact with water either through irrigation or precipitation [20,23,24].

The mechanism of phosphorus removal through the use of clinoptilolite does not seem to be well established at this time. Since phosphorus occurs mostly as an anion, i.e. orthophosphate in human urine, ion exchange with clinoptilolite will not be possible since it is a cation exchanger [23,24]. Adsorption is a probable mechanism for phosphate removal. Clinoptilolite could possibly adsorb phosphorus either as phosphate or as a surface adsorbing phosphorus containing precipitate. Using pure solutions as the liquid phase, Chmielewska et al. [18] reported that precipitation of phosphate with calcium and magnesium ions on clinoptilolite surface was the predominant mechanism, with minor amounts of phosphate removal through physical adsorption. Hedstrom and Amofah [17] postulated that phosphate removal could occur either through chemical sorption or surface precipitation with domestic wastewater as the liquid phase. However, they indicated that phosphate removal could not be explained by chemical sorption because chemical sorption is not easily reversible but phosphate on clinoptilolite could easily be desorbed with water. Therefore they concluded that adsorption of calcium-phosphorus compounds having low solubility is more probable. With her experimental work involving ionic concentrations using clinoptilolite and human urine, Allar [16] concluded that surface precipitation of phosphates upon reacting with calcium ions seems to be a probable mechanism, based on the observation that surface concentration of phosphate increased with increasing calcium ions released into the solution. No work in the literature was encountered with a concrete conclusion regarding the mechanism of phosphate removal when urine was used as the liquid phase.

Nutrient removal/recovery from human urine through ion exchange/adsorption using clinoptilolite had also been the subject of several papers in the literature. A comparative investigation involving clinoptilolite and three other ion exchangers were recently presented by Tarpeh et al. [25] for nitrogen recovery from source separated urine, while some earlier pieces of literature [26–28] investigated the use of adsorption onto zeolites, wollastonite and activated carbon in combination with freezing/thawing and struvite precipitation for nutrient recovery from urine. Beler Baykal and Dogan [29] suggested the use of ion exchange/adsorption with clinoptilolite as one of the alternative stages in two stage processing of source separated human urine for nutrient recovery.

In other pieces of work, nutrients in urine were first transferred from the liquid phase and concentrated on clinoptilolite, subsequently they were recovered upon contact with water through desorption from the solid phase to be made available to plants. Application of nutrient enriched clinoptilolite thus produced is suggested as fertilizer [20,23,24,29–32]. In their work regarding nutrient removal from human urine Kocaturk and Beler Baykal [23] achieved 8.7–9.5 mg ammonium/g clinoptilolite surface capacity for 10 mg ammonium/g clinoptilolite initial loading in the batch mode with typical values of 90% for ammonium, 89% for potassium and 97% for phosphate removals. Other values reporting values from work along similar lines went as high as 98% for ammonium [30] and 99% for potassium removal [31]. All experiments mentioned here were carried out in the batch mode.

Within that context, human urine is contacted with clinoptilolite to load nutrients onto zeolite through ion exchange/adsorption to produce nutrient enriched clinoptilolite which can be used as the fertilizer product. After loading, nutrient enriched clinoptilolite is applied onto soil as fertilizer, and nutrients on loaded clinoptilolite are released upon contact with water, either through irrigation or upon precipitation, and are made available for plant growth. The nutrient loading phase of this application can be carried out in two modes as batch or continuous. When human urine is contacted with clinoptilolite with 100% recycle for batch mode operation, initial ammonium loading is the main parameter to determine the outcome of the process [31]. For continuous operation, human urine passes through clinoptilolite column continuously and empty bed contact time becomes the main parameter [33].

Majority of the work on this subject matter cited above is focused upon batch-wise operation to load nutrients in urine onto clinoptilolite [20,23,24,29–32]. Considering that the expected surface capacity in a continuous ion exchanger/adsorber should be higher as compared to its batch counterpart due to always being in contact with the highest possible liquid phase concentration, this paper aims to investigate the characteristics of system behavior under continuous flow conditions in an attempt to compare it to the more widely used batch-wise contacting. Source separated human urine is used as the liquid phase and the focus is on recovery of nitrogen and phosphorus. The paper addresses breakthrough profiles, surface concentrations/capacities and operation times in particular.

2. Material and methods

Source separated human urine was stored at least two months after collection. As urea is the most common form of nitrogen in fresh human urine, storage is significant for completion of its hydrolysis to provide the ionic form, NH_4^+, which can be removed through ion exchange. pH of stored urine was adjusted to around 7 at the beginning of the loading experiments to ensure that nitrogen is in the form of NH_4^+. Natural clinoptilolite from Gordes of Turkey was used as ion exchanger/adsorbent with a particle size of 1–2 mm. The chemical composition of Gordes clinoptilolite as given by the supplier is presented in Table 1 [34]. The clinoptilolite used was preconditioned with 1 M NaCl to attain the sodium form for increasing ammonium exchange capacity. The same batch of urine and the same batch of clinoptilolite were used throughout the entire experimental work under both modes of contact.

Batch loadings were made in columns of 3.6 cm diameter with 100% recycle for 10–40 mg NH_4/g clinoptilolite initial loadings. The flow rate was the same for all loadings as 10 mL/min. Samples were taken from the feed tank in time to monitor system behavior as it reached the final constant value.

Continuous loadings were also carried out in similar columns with a diameter of 3.6 cm for a range of 30–120 min contact times (t_c) and samples were taken from the column effluent continuously. Influent concentrations were measured at the beginning and at the very end of the experiments to observe that no changes have occurred.

Table 1
Chemical composition of Gordes clinoptilolite [34]

Compound	%
SiO_2	68.30
Al_2O_3	12.55
Fe_2O_3	1.03
TiO_2	0.07
CaO	2.33
MgO	1.09
Na_2O	0.88
K_2O	2.32

The exhaustion time was taken as the time when the concentration in the feed tank was practically constant for batch contact and the time when effluent concentration reached 93–96% of the influent concentration with continuous operation.

The basic experimental setup is shown in Fig. 1 and experimental conditions are given in Table 2 for the batch mode and in Table 3 for the continuous mode. All samples were analyzed for ammonium with an Orion ion meter/Orion ammonia probe and orthophosphate with the Stannous Chloride Method of Standard Methods [35].

Surface capacities (q) for batch mode were calculated using initial (C_I) and final concentration (C_F) in feed tank (mg/L), urine volume (V) (L) and the amount of clinoptilolite (M) (g) used.

$$q = \frac{C_I - C_F}{M} xV$$

Surface capacities for continuous mode were calculated using influent and effluent concentration (mg/L), flow rate (mL/min), time of sample taken from the effluent of column (min). Using these values, effluent concentration versus throughput (flow rate × time) graphs were drawn. The area under the curve was calculated to find the amount of nutrient leaving the column and it was subtracted from the total amount of ammonium/phosphorus that was fed into the system, which was calculated by multiplying the influent concentration by the flow rate and the total time of experiment.

3. Results and discussion

This work was undertaken with the primary aim of determining surface capacities and operation times in the batch and continuous modes for nutrient recovery from

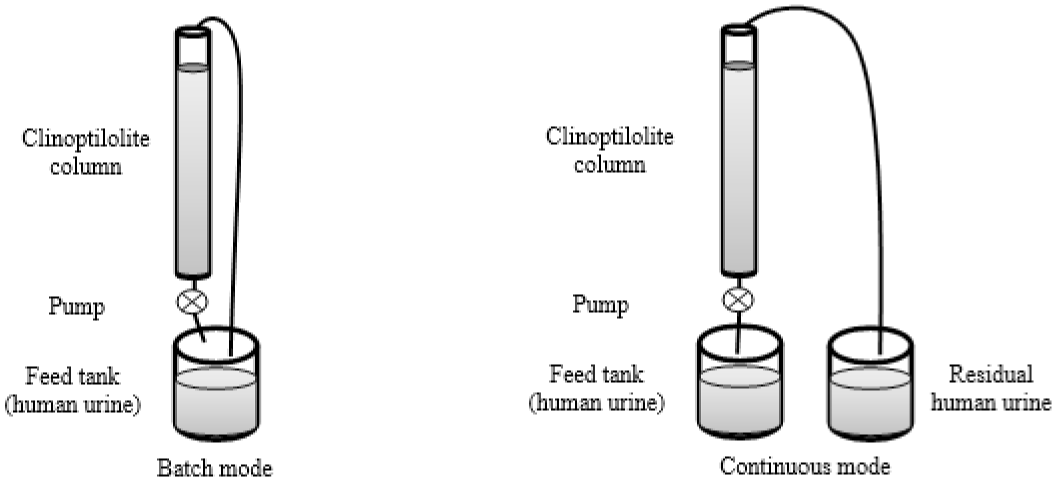

Fig. 1. The basic experimental setup for batch and continuous mode.

Table 2
Experimental conditions for batch loadings

Initial loading	Bed height	Amount of clinoptilolite	Volume of urine	Initial concentration	
mg NH_4/g clinoptilolite	cm	g	mL	mg NH_4/L	mg PO_4-P/L
10	91.5	750.318	1046	7175	261
15	92.2	750.242	1631	6900	220
20	98.0	801.168	2003	8000	263
25	89.8	750.672	2615	7175	261
30	89.0	750.358	3170	7100	257
40	29.0	202.507	1000	8100	274

Table 3
Experimental conditions for continuous loadings

Contact time	Bed height	Flowrate	Amount of clinoptilolite	Influent concentration	
min	cm	mL/min	g	mg NH_4/L	mg PO_4-P/L
30	15.3	5.2	100.092	7800	234
65	15.2	2.4	100.092	7750	246
90	15.9	1.8	100.096	8125	227
120	15.8	1.3	100.092	7800	234

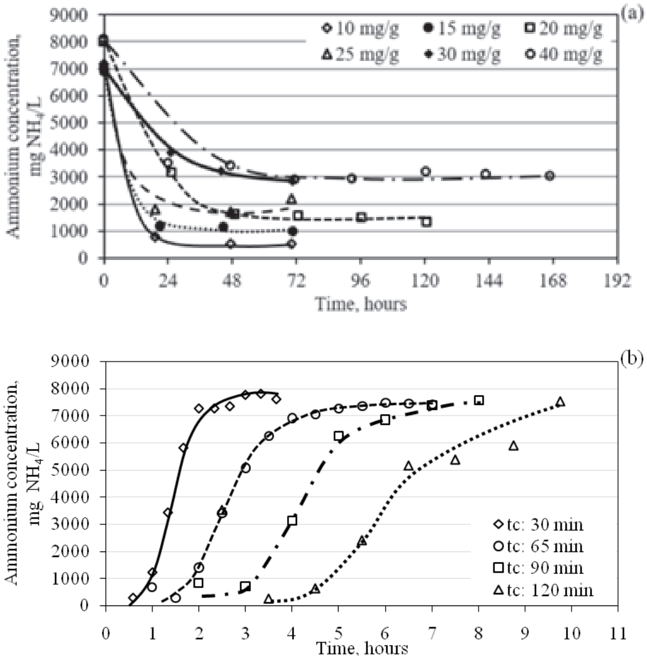

Fig. 2. Liquid phase ammonium concentration in time for batch operation (a), the breakthrough profile for continuous operation (b).

human urine with clinoptilolite through ion exchange/adsorption. Experimental conditions were so set up as to enable a comparison between two modes of contact, i.e., batch vs. continuous, using the same batch of urine and the same batch of clinoptilolite. The results are presented in Figs. 2–3, and Tables 4–5.

The exhaustion time, which marks the end of the loading cycle under each option, was taken as that time when the concentration in the feed tank was practically constant for batch operation and the time when effluent concentration reached 93–96% of the influent concentration with continuous contact which may be determined from the breakthrough profiles. The liquid phase concentrations in time in the batch mode and the breakthrough profiles for continuous operation are given in Fig. 2 to show that two days were necessary for batch loading as operation time while a maximum of 10 h was enough for continuous loading when the same batch of urine and clinoptilolite were used.

The surface capacities calculated from Fig. 2 are presented in Fig. 3 and Table 4. Surface capacities were 30.1–38.5 mg NH_4/g clinoptilolite and 0.44–0.66 mg PO_4-P/g clinoptilolite for the continuous system, while it was 9.3–25.0 mg NH_4/g clinoptilolite and 0.33–1.33 mg PO_4-P/g clinoptilolite for the batch mode. As can be observed, the highest surface capacity was 38.5 mg NH_4/g clinoptilolite with 90 min contact time for the continuous mode. On the other hand, the highest surface capacity for the batch operation was 25.0 mg NH_4/g clinoptilolite with 40 mg NH_4/g clinoptilolite initial ammonium loading. This shows that under all circumstances surface capacity was lower in the batch mode as compared to the continuous one, as in the continuous mode all were higher than 30 mg NH_4/g clinoptilolite. The results indicated that while for phosphorus batch operation gave slightly better outcomes, surface capacities were remarkably higher for nitrogen, which is removed through ion exchange, when continuous mode was employed. The reason for the difference between surface capacities for batch and continuous modes is that clinoptilolite is in contact with the same high influent concentration in the continuous mode throughout the entire operation while the liquid phase concentrations keep decreasing in time in the batch mode. Therefore, surface concentrations with higher liquid phase concentration as in the case of continuous operation will be expected to be higher.

Regarding a comparison between maximum surface concentrations attained in this work and those that were reported in the literature, the highest surface capacity observed in this work for nitrogen was 25 mg NH_4/g clinoptilolite, while the maximum in the literature for the Turkish clinoptilolite was 27.36 mg NH_4/g clinoptilolite [10], both performed in the batch mode. It has to be noted that this study uses human urine as opposed to synthetic solution in the work of Sirkecioglu and Senatalar [10]. With regard to phosphorus, the highest surface capacity attained in this work was 1.33 mg/g with an initial P-concentration of urine between 220 and 274 mg P/L, as opposed to 12 mg/g attained with an extremely high P concentration at 1400 mg/L in a pure solution [18]. For both nutrients, the results seem to be in line with expectations considering the differences in liquid phase characteristics and concentrations.

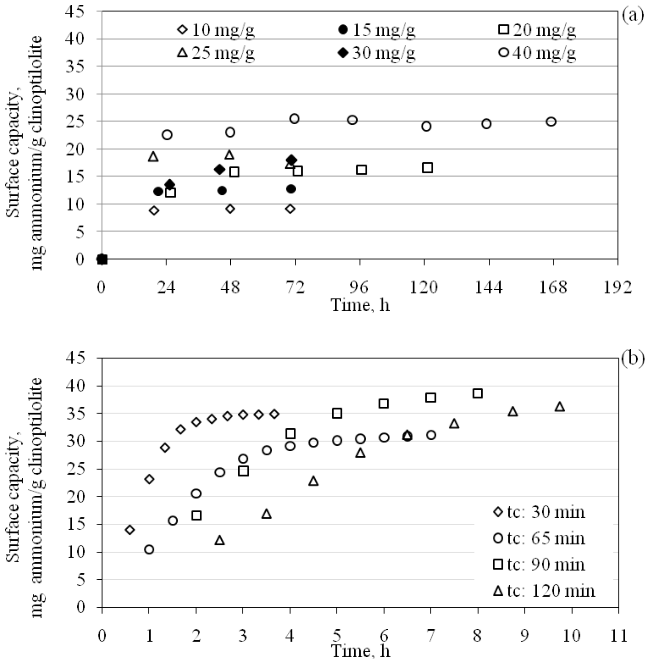

Fig. 3. Surface ammonium concentrations/surface capacities in batch operation (a) and surface capacity in continuous operation (b).

It was previously stated that an operating time of two days was necessary for batch loading while a maximum of 10 h was enough for continuous loading as operation time. The lowest surface capacity, 9.3 mg NH_4/g clinoptilolite, was observed with 10 mg NH_4/g clinoptilolite initial loading in the batch mode in one day, and this capacity could be attained in an operation time of 24 min with 30 min contact time in the continuous mode. While a maximum of 25 mg NH_4/g clinoptilolite surface capacity with 40 mg NH_4/g clinoptilolite initial loading in the batch mode could be achieved in 3 d, 2 h was enough to attain the same value in the continuous mode with 30 min contact time. The results show that operation times were remarkably lower for the continuous mode, which will provide advantages in terms of sizes and operational costs.

From the stand point of surface capacities and operation times, it is clear that continuous mode seems preferable over batch operation for nutrient recovery from human urine with clinoptilolite through ion exchange/adsorption. However, when residuals, in this case residual liquid phase, i.e. human urine, are considered, continuous operation may be disadvantageous. This may be an important issue when pollution control is considered rather than or in addition to nutrient recovery.

Previous work on the impact of initial ammonium loading had shown that although surface concentrations increase with increased initial loadings, removal efficiencies from the liquid phase are lower and residual liquid phase concentrations are higher for higher initial loadings for batch systems [31]. This present work provides support for these findings. Ammonium removal efficiencies were 60-93% for the batch mode and 48–60% for the continuous one. The lowest ammonium removal efficiency for the batch mode was achieved as the highest value in the continuous mode and phosphorous removal efficiencies in the batch mode were at least 3 times higher than those in the continuous mode.

Additionally, the results of this work presented in Table 5, have shown that residual liquid phase concentrations were 3086–4188 mg NH_4/L and 170–194 mg PO_4-P/L on a collective basis for the continuous mode, and 524–3020 mg NH_4/L and 2–57 mg PO_4-P/L for the batch mode,

Table 4
Surface concentrations on clinoptilolite for batch and continuous operation

Batch			Continuous		
Initial NH_4 loading	Surface capacity		Contact time	Surface capacity	
mg NH_4/g clinoptilolite	mg NH_4/g clinoptilolite	mg PO_4-P/g clinoptilolite	min	mg NH_4/g clinoptilolite	mg PO_4-P/g clinoptilolite
10	9.3	0.33	30	33.4	0.44
15	12.8	0.39	65	30.1	0.61
20	16.6	0.63	90	38.5	0.66
25	17.4	0.71	120	36.2	0.66
30	18.0	0.90			
40	25.0	1.33			

Table 5
Summary of liquid phase NH_4 and PO_4 concentrations and removal efficiencies (I-initial, R-residual concentrations)

	Batch system Initial loadings, mg NH_4/g clinoptilolite						Continuous system Contact times, min			
	10	15	20	25	30	40	30	65	90	120
C_I, mg NH_4/L	7175	6900	8000	7175	7100	8100	7800	7750	8125	7800
C_R, mg NH_4/L	524	1010	1330	2196	2832	3020	3086	3907	4188	3852
Removal, %	93	85	83	69	60	63	60	50	48	51
C_I, mg PO_4-P/L	261	220	263	261	257	274	234	246	227	234
C_R, mg PO_4-P/L	26	42	12	57	44	2	194	176	170	174
Removal, %	90	81	95	78	83	99	17	28	25	26

indicating that residual nutrient concentrations are higher in the continuous mode.

In summary, despite lower removal efficiencies and higher residual liquid phase concentrations, continuous mode is more preferable than batch mode in terms of surface capacities of nitrogen and operation times. The benefits of continuous operation in terms of sizes and costs in an actual application are addressed elsewhere [36]. As the main target of this work was to attain higher surface capacities to recover higher quantities of plant nutrients as fertilizer, the continuous system provided a distinct advantage.

4. Conclusion

In conclusion, continuous systems approach exhaustion in a much shorter time for nitrogen and phosphorous recovery from source separated human urine. Under no circumstances were operation times higher than 10 h in the continuous mode as opposed to 2 d in the batch operation. Moreover, surface capacities of ammonium reflecting the amount which could be recovered per 1 g of clinoptilolite were higher when the mode of contact was continuous. This is expected to reflect into smaller sized columns and thus lower costs as advantages of the continuous operation. However, removal efficiencies were higher and the cumulative concentrations in the remaining liquid phase were lower in batch operation indicating that it is more advantageous for those aspects. All in all, continuous contact seems more plausible when the primary aim of processing is nutrient recovery for further use as fertilizer, especially when production of nitrogen fertilizers is intended.

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