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Preliminary results for start-up and adaptation of an EGSB reactor for valorization and treatment of fresh urine

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ABSTRACT

Processing of the liquid phase remaining after nutrient recovery from source separated urine using clinoptilolite is an important issue regarding environmental protection. Possibility of anaerobic treatment using an expanded granular sludge bed reactor for this purpose is investigated as an option to remove organic matter together with possible biogas generation. To start this investigation, an anaerobic granular sludge from a confectionery industry was adapted to source separated fresh urine, which is highly saline with an electrical conductivity of 8,000 μS/cm, has a high ammonium concentration (900 mg NH_4^+–N/L) and relatively high organic content of 1,200 mg COD/L. In a period of observation which exceeded 2 months, 75% and 60% of removal could be achieved for soluble and total COD, respectively, together with minor amounts of NH_4^+ release into the liquid phase. The amount of CH_4 to be produced was calculated from COD removal as 1.3 L CH_4/d and 0.24 L CH_4/L urine. The preliminary results obtained in this work were motivating for further investigation upon the subject matter with emphasis on the effect of high salinity content.

Keywords: Source separation; Fresh human urine; Anaerobic treatment; Expended granular sludge bed (EGSB) reactor; Salinity; Adaptation/start up; COD removal; Biogas production

1. Introduction

One of the most effective ways of recycling nutrients in domestic wastewater is segregation of human urine at the source and processing it further to produce fertilizers to be returned to the food chain. Human urine is a nutrient-rich stream containing over 80% of nitrogen, and over 50% of both phosphorus and potassium in domestic wastewater, in addition to highly concentrated organic matter content, and high salinity [1]. Ion exchange/adsorption is one of the successful methods of recovering nutrients from urine which may be recycled to agricultural use [2–7]. This may be done through contacting urine with clinoptilolite upon which nutrients are concentrated. Contacting urine with clinoptilolite leaves a liquid residue which has a high COD content, typically over 4,000 mg COD/L, and high salinity over 20,000 μS/cm [8] which has to be treated before disposal for environmental protection. COD concentration of this residual liquid phase is fit for anaerobic processing, which may act not only to treat the organic fraction but also can generate biogas that may possibly be used as energy. Yet the high salinity of the liquid phase is a challenge which needs attention.

There are very limited studies in the literature where anaerobic processing is employed to handle organic matter in human urine. Previous studies in the literature used urine only in co-digestion rather than using it by itself to feed an anaerobic reactor. In one study [9], urine was used as a co-substrate in anaerobic digestion of manioc wastewater to improve biogas production. The results of that study revealed

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that addition of human urine helped to increase the biogas production from 14.63 dm^3 to 60–80 dm^3. In another study [10], where anaerobic treatment of kitchen waste in combination with brown water (mainly source separated human feces), and yellow water (source separated human urine) was investigated, the results showed that the addition of yellow water had improved the performance of the batch reactor systems used. This was attributed to the micronutrients present in yellow water. According to the same study, the percentage of urine in the reactor was observed to be significant due to free ammonia inhibition at higher percentages. With 50% urine with an ammonia concentration around 2,000 mg $N–NH_3$/L, free ammonia inhibition was observed. No publications were encountered in the literature which investigated anaerobic treatment for processing/revaluating urine directly as its focus.

Additionally, several pieces of work in the literature [11,12] reported possible ammonium release during anaerobic processing. The ammonium release was attributed most probably to anaerobic hydrolysis of the organic nitrogen in the feed.

The purpose of this paper is to present the preliminary results from an expended granular sludge bed (EGSB) reactor performing anaerobic treatment to remove organic matter from urine. The main focus of this particular investigation was the adaptation of the anaerobic sludge with a feed solution of 25% fresh urine, in an attempt to adapt the anaerobic granular sludge from a confectionary wastewater treatment plant to highly saline urine feed. The adapted granular sludge was to be used later to handle the liquid phase remaining after the ion exchange process for nutrient removal, in an attempt to treat the residual organic matter for environmental protection. The success of this proposal will lead to the production of biogas from this waste stream which may be used as a source of energy. The EGSB reactor was investigated not only for organic matter removal but also to observe if anaerobic processing will have an effect on NH_4^+ concentrations.

Table 1 shows characteristics of fresh urine as well as the confectionery wastewater upon which the granular sludge was produced in this work. It may be observed that the pH of fresh urine falls within the range of the confectionary wastewater while the COD content is at the lower end of the range for the latter. However, conductivity and nitrogen forms in fresh urine are greater than that of their counterparts in confectionery wastewater, specifically about 35-fold for conductivity and over 45-fold for nitrogen, indicating the main challenges in this investigation as salinity and possible ammonium inhibition.

2. Materials and methods

Urine was collected from men's toilet at Istanbul Technical University, Department of Environmental Engineering, originating from urinals that use flush water. Partial hydrolysis of urine was observed as it flows through the collection pipes while it is transported into the collection tanks. The feed solution was 25% fresh urine diluted with tap water. Characteristics of the feed solution measured in this work are shown in Table 2. The pH of the feed solution was controlled with hydrochloric acid to keep it at around 7.5 to maintain a suitable pH for anaerobic sludge as well as to keep the nitrogen in ammonium form so that it will not be lost into the atmosphere as ammonia.

The granular sludge to be adapted to urine feed was originally provided from the anaerobic processing unit of a confectionary plant treating confectionary wastewater whose characteristics are summarized in Tables 1 and 2.

The experiments were performed in a lab-scale EGSB reactor made of plexiglass. The EGSB reactor had an effective working volume of 9.2 L, with a total height of 120 cm. The diameter of the lower column where the granular sludge was located was 8.0 cm, while the diameter of the upper part was 20 cm, which is designed for biogas collection as shown in Fig. 1. The column had five sample outlets, the top one located at 88 cm from the bottom was used for circulation, the one directly below it was used to take samples for checking the pH of the reactor. 30% of the reactor volume was filled

Table 1
Characterization of fresh urine and confectionery wastewater available in literatures

Parameter	Fresh urine [8]	Fresh urine [13]	Confectionery WW [14]
pH	6.0	5.6–6.8	3.83–6.48
E. conductivity, μS/cm	14,000	14,800–25,400	252–689
COD, mg/L	7,120	3,400–6,550	6,540–37,180
Ammonium, mg/L	344	780	0.22–15.4
TKN, mg/L	5,700	4,700	16–95

Table 2
Characterization of fresh urine used in the experiment

Parameter	25% Fresh urine, average	100% Fresh urine, average	Confectionery WW [14]
pH	7.5	9.3	3.83–6.48
E. conductivity, μS/cm	8,100	20,000	252–689
COD, mg/L	1,200	4,550	6,540–37,180
Ammonium, mg/L	906	3,550	0.22–15.4
TKN, mg/L	2,660	9,800	16–95

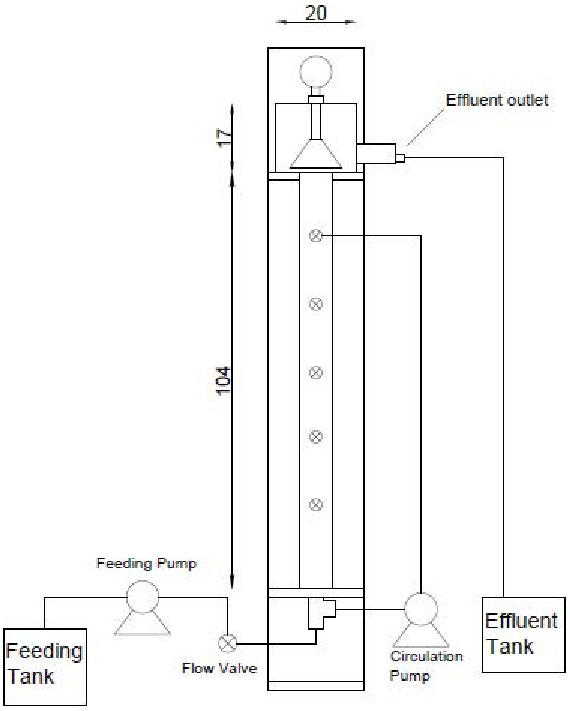


Fig. 1. EGSB reactor set up.

with granular sludge from the confectionary plant. Two pumps were connected to the column, one for feeding, and the other for circulation. The flowrate was adjusted using a circulation pump until the granular sludge was in suspension, as it is one of the main characteristics of EGSB reactors. The EGSB reactor was fed with 5.5 L/d at a hydraulic retention time (HRT) of 40 h and an organic loading rate (OLR) of 0.72 kg COD/m^3d. The feeding of the column was done intermittently to provide 40 h of HRT. The reactor was located in a constant temperature room adjusted to 35°C–40°C to maintain mesophilic temperature conditions.

The influent samples were collected from the feeding tank while the effluent samples were collected from the hose connected to the effluent outlet. Parameters which were monitored continuously on a daily basis were pH, conductivity, COD and N as ammonium and total Kjeldahl nitrogen (TKN). pH was determined by using a pH probe (Hanna instruments HI 1332, USA), and conductivity by a conductivity probe (HACH CDC 401, USA). Nitrogen analyses as ammonium and TKN were made by using ion meter/ammonia probe (Thermo Orion 9512) and Macro Kjeldahl Nitrogen method, respectively. COD as soluble and total COD were measured by open reflux method. Samples were filtered using 0.45 μm filters for soluble COD analysis [15].

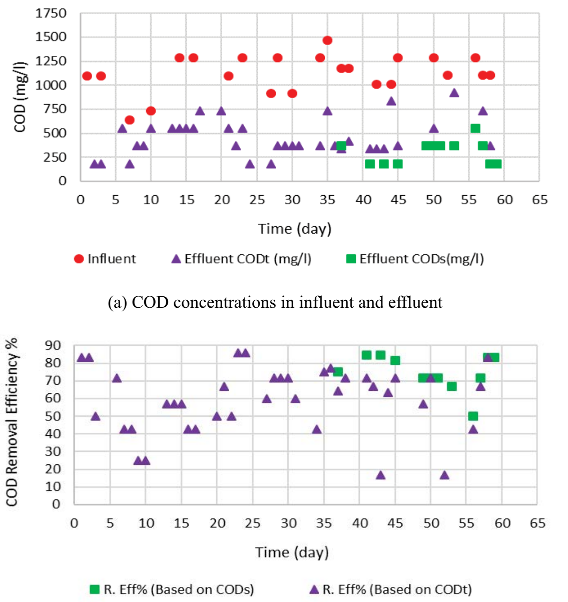
3. Results

Table 2 shows characteristics of the 25% fresh urine solution used to feed the EGSB reactor, together with the original fresh urine that it was diluted from. Characteristics of the confectionery wastewater solution are added once again to show the compatibility between the original wastewater with which the granular sludge was produced, and the feed solution specifically used in this work. A comparison of the urine feed with confectionery wastewater shows that the pH was compatible with the wastewater as well as the pH levels recommended for anaerobic treatment as 6–8 [16]. The COD was low, however, at a reasonable level for anaerobic treatment when compared with the low end values that were previously reported [17], as 1,500 mg COD/L, as the value preferred for a sufficient CH_4 production.

Free ammonia and ammonium are factors that may lead to inhibition of anaerobic processes. A concentration between 50 and 200 mg/L of total ammonia nitrogen (TAN) is advantageous, while a concentration in the range of 1,500–7,000 mg/L of TAN is inhibitory and toxic [18–20]. Table 2 shows that concentrations of nitrogen were by far higher in urine as compared with the confectionery wastewater, however, when diluted to 25%, ammonium concentration was at an acceptable level regarding ammonium inhibition.

Regarding salinity, 100% fresh urine has a salinity (as measured in units of conductivity) level which is about 28 fold higher of that for confectionery wastewater. Salinity inhibition in anaerobic processes was investigated [21], to indicate that an electrical conductivity of 35,000 μS/cm could decrease methane production, while an electrical conductivity of 80,000 μS/cm inhibits methane and CO_2 generation as well inhibiting the degradation of organic matter. With the 25% urine feed, although there was more than 10-fold difference, conductivities were below these limits.

Under experimental conditions employed in this study, the results revealed that with 25% fresh urine, COD removal efficiency fluctuated in the first month of operation in the startup period, but stabilized thereafter as shown in Fig. 2. The total and soluble CODs of the influent were comparable, 1,100 mg COD/L for soluble and 1,200 mg COD/L for total COD as averages. In the effluent, the total COD was around 550 mg COD/L at the end of the first month of adaptation, which after one month started to stabilize at a concentration of 440 mg COD/L and a soluble COD of 300 mg COD/L. Tiny particles were observed in the effluent from time to time that was most probably due to biomass escaping from the column which led to higher total COD concentrations as compared with soluble COD in the effluent. Based on this observation, soluble COD analysis was also made and the results have revealed that the system was reasonably successful in terms of reducing total COD concentrations from about 1,200 to 440 mg COD/L with a reduction of 60%, and for soluble COD from 1,100 mg COD/L to 300 mg COD/L with a reduction of 75%, as shown in Fig. 2. The results obtained in this work were in line with studies which had been conducted to investigate the performance of EGSB with low strength wastewaters. In a previous study [12], in which the EGSB reactor performance operated with a synthetic wastewater simulating the municipal wastewater in the laboratory, the COD removal ranged from 45% to 75% with OLR values of 1.35–2.70 kg COD/m^3 d, respectively, under mesophilic conditions. Another study using an EGSB reactor with a synthetic wastewater simulating a low strength wastewater of a 600–800 mg/L of COD, showed that removal efficiencies around 80% and 66% could be achieved with an OLR of 6.5 and 12 kg COD/m^3 d, respectively [22]. In both of these studies, salinity is supposed to be much lower than the salinity of urine employed in this investigation as the liquid phase involved simulated domestic wastewater.



(a) COD concentrations in influent and effluent

) COD removal efficiencies based on total COD (CODt) and soluble COD (CODs

Fig. 2. COD removal from urine in the EGSB reactor (a) COD concentrations in influent and effluent and (b) COD removal efficiencies based on total COD (CODt) and soluble COD (CODs).

Fig. 3 shows the pH values of the EGSB reactor. pH of the influent was always kept at values around 7.5 to maintain a suitable pH for the anaerobic sludge used, and to be able to prevent conversion of ammonium to ammonia at higher pHs, as this study also aims to investigate the release of ammonium at the effluent of the EGSB reactor. A slight increase in pH was observed in the effluent.

The ammonium concentration in the influent was within a range of 720–1,020 mg NH_4^+–N/L and the effluent was with the range of 820–1,030 mg NH_4^+–N/L. The fluctuation in the start-up period was observed to be less with ammonium concentrations as compared with that of COD. A small ammonium release was observed in the effluent at an average of 6%, which was rather low as shown is Fig. 4, and that was consistent with TKN results in which the increase between the influent and the effluent was 3% at an average, as shown in Fig. 5. The increase in the nitrogen content in terms of ammonium and TKN can be attributed to the ammonification of the organic nitrogen that was converted to ammonium. In the current study, the conversion was observable but low which is in contrast with a low ammonia wastewater with an average value of 8.4 mg NH_3–N/L in the influent and 17.5–20.5 mg NH_3–N/L at the effluent, where it was reported that organic nitrogen was completely converted to ammonia form [12]. Investigation of the release of ammonium is an important observation in case of treating source separated urine using anaerobic processing, because it will dictate if it would be meaningful to use a second stage ion exchange column to remove the additional ammonium released for nutrient recovery purposes or to meet the discharge standards.

Fig. 6 presents salinity measured as conductivity in the influent and the effluent of the EGSB reactor. The conductivity in 25% fresh urine influent solution was 8,100 µS/cm, and

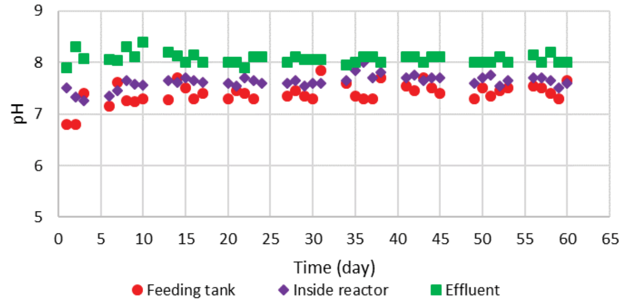


Fig. 3. pH values of the anaerobic system.

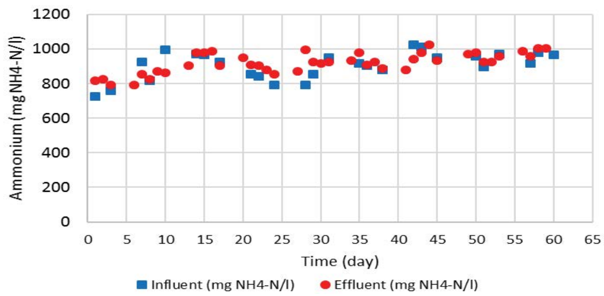


Fig. 4. Ammonium concentrations in the influent and effluent.

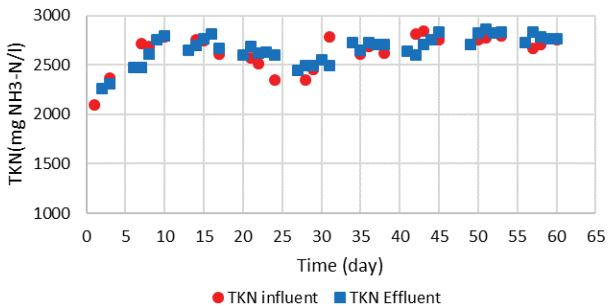


Fig. 5. TKN concentrations in the influent and effluent.

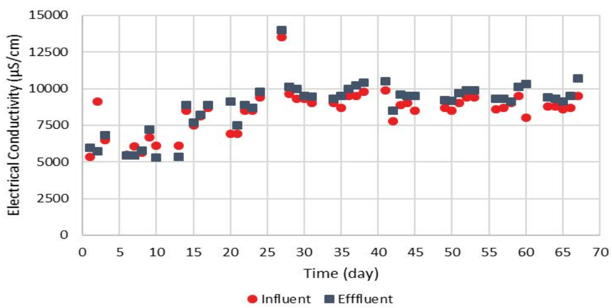


Fig. 6. Conductivity of influent and effluent of 25% fresh urine in µS/cm.

there was a slight change at the effluent which was 8,800 µS/cm as an average. Conductivity levels fluctuated also till it stabilized after almost a month from the start of the EGSB reactor similar to the COD concentration. Increasing electrical conductivity at the effluent may be attributed to the increase in alkalinity ions that is produced in EGSB reactor as

Table 3
Summary of major experimental results as averages

Parameter	COD (mg/L)		NH_4^+ (mg NH_4^+/L)	TKN (mg NH_3–N/L)	pH	Conductivity (μS/cm)
	Total	Soluble				
Influent	1,200	1,100	906	2,660	7.5	8,100
Effluent	440	300	930	2,690	8.1	8,800

a result of biochemical reactions. Summary of major parameters are presented in Table 3 as averages.

The amount of methane produced was calculated based on the COD removed per day using the assumption 0.35 L CH_4/g COD [16] and the calculation showed that an average of 1.3 L CH_4/d or 0.24 L CH_4/L urine will be produced which is equivalent to 5 W.

The results obtained in this work indicate that treatment of urine using anaerobic processes is possible and that best operational conditions are to be determined taking into consideration optimal results for combined sorption and anaerobic processes to lead to a concurrent nutrient recovery accompanied by treatment of residual urine with the possibility of producing biogas, to support sustainability concepts.

4. Conclusion

Following an adaptation period of 2 months, the EGSB reactor performance with 25% fresh urine was considerably significant in terms of removing organic matter with about 75% efficiency based on soluble COD, and 60% based on total COD. Ammonium release in this work was found to be low with 6% only. This work aimed to investigate the adaptation of an anaerobic granular sludge from an industry (confectionery industry in this case) to process urine (25% fresh urine feed in this case) to make it available for further use for anaerobic treatment of the liquid residue from sorption processes in which nutrients had been removed from urine, and the results provided evidence that this could be accomplished.

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