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Research Article

Combining Anerobic Degradation and Chemical Precipitation for the Treatment of High Strength, Strong Nitrogenous Landfill Leachate

Treatment of organics and ammonium from sanitary landfill leachate was investigated using a combination of anaerobic treatment and magnesium ammonium phosphate precipitation (MAP) processes. The effect of organic loading rate (OLR) was evaluated for the removal of COD and BOD_5 in an anaerobic treatment by anaerobic sludge blanket (UASB) reactor. The UASB reactor removed organics successfully. OLR has a significant effect on the reactor performance and the maximum COD removal was 90% at an OLR of 9 kg COD/(m^3d). On average, 86.3% of COD and 95.3% of BOD_5 were removed at a steady state of anaerobic treatment. MAP precipitation was performed in order to decrease the ammonium concentration of anaerobically-treated leachate. Furthermore, the effect of pH and the molar ratio of MAP constituents on the removal of ammonium were evaluated. The maximum ammonium removal was observed as 98% at a pH of 8.5 and a stoichiometric ratio of $NH_4/PO_4/Mg = 1/1/1$. The results obtained indicate that a combination of UASB and MAP can be successfully used for the removal of organics and ammonium from sanitary landfill leachate.

Keywords: Ammonium; Anaerobic sludge blanket reactor (UASB); Degradation; Leachate; Magnesium ammonium phosphate precipitation (MAP); Precipitation; Sanitary landfill

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1 Introduction

Sanitary landfilling is the preferred method for the disposal of municipal solid waste due to its economic advantages. Microbial degradation and physico-chemical degradation of the organic contents of wastes in combination with percolating rainwater contributes to leachate generation in landfill sites. Municipal landfill leachate is one of the most important environmental contaminants and the discharge of high strength-leachate without treatment into surface and groundwater bodies may cause severe environmental hazards [1]. The composition of leachate contaminants is mainly effected by the quality of waste deposited, hydrogeological factors, age of the landfill and climate conditions effects [2 – 4].

Landfill leachate is a high-strength wastewater with a deep color, and is characterized by extremes of biochemical oxygen demand (BOD), chemical oxygen demand (COD), and ammonium [5]. COD levels as high as 30,000 mg/L are very common from active landfills [6]. The degradation of portions of organic nitrogen in waste, e. g., proteins, amino acids or urea, leads to the generation of ammonia. At pH 7 – 8 and 25°C, more than 95% of ammonia is in the ionic ammonium form (NH_4^+) and the concentration increases up to 3000 to 5000 mg/L [7, 8]. Ammonium is one of the most significant components of leachate that has a toxic affect on the biological organisms in treatment plants and water receiving sources.

Since the main concerns in landfill leachate are organic and ammonia contents, the development of efficient pollution reduction methods entails the removal of theses pollutants. Biological methods, such as aerobic and anaerobic treatment are effective for the removal of organics from wastewater. In the literature, it is reported that aerobic processes are successful for the treatment of easily biodegradable and low concentration leachates, whereas anaerobic processes are suitable for the treatment of high strength leachate [9, 10]. Moreover, anaerobic treatment has the advantages of lower operation costs, as well as the production of useable biogas and lower amounts of sludge [11]. Many researchers have found that anaerobic treatment can successfully remove the COD content of wastewater but that it is insufficient for decreasing the high ammonium concentration to desirable levels [12 – 14]. The necessity for ammonium elimination from leachate entails the application of additional treatment methods.

In the literature, several combination treatment systems have been investigated for increasing the removal performances of contaminants from leachate since individual processes fail to reach effluent limits [15 – 17]. In this study, the feasibility of combining anaerobic treatment and magnesium ammonium phosphate (MAP) precipitation for the treatment of Odayeri landfill leachate was evaluated. An anaerobic sludge blanket (UASB) reactor was used for the removal of organics and MAP precipitation was preferred as a subsequent step to increase ammonia removal efficiency.

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Abbreviations: BOD, Biochemical oxygen demand; **COD**, Chemical oxygen demand; **HRT**, Hydraulic retention time; **MAP**, Magnesium ammonium phosphate precipitation; **OLR**, Organic loading rate; **UASB**, Anaerobic sludge blanket; **VFA**, Volatile fatty acid

2 Materials and Methods

2.1 Leachate Sample

Landfill leachate was taken from the Odayeri sanitary landfill site located on the European side of Istanbul. The leachate was collected in a tank at the lowest side of the landfill and the samples were taken from the landfill once every week. The leachate samples were kept in refrigerator at 4°C before use.

2.2 UASB Reactor

Anaerobic treatment of the leachate was performed in a laboratory scale mesophilic upflow anaerobic sludge blanket reactor (UASB). The schematic diagram of the UASB reactor is given in Fig. 1. The reactor was made of plexiglass and had a diameter of 10 cm and a height of 105 cm, with an active and total volume of 8.1 and 10.6 L, respectively. The leachate was introduced through the bottom of the reactor using a Watson Marlow peristaltic pump and the gas-liquid separator was positioned at the top of the reactor. The mesophilic conditions (35°C) of the reactor were maintained by pumping hot water from an external thermostat through a water jacket surrounding the reactor. The UASB reactor was seeded up to 7 L of its active volume, with anaerobic granular sludge obtained from an anaerobic reactor that was used to treat alcoholic wastewater. The granular seed has a diameter of 0.5 to 2 mm and was fed with the same wastewater from the alcohol treatment plant.

2.3 MAP Precipitation

Ammonium removal from anaerobically-treated leachate was performed in the MAP precipitation experiments. Analytical grade magnesium chloride ($MgCl_2 \cdot 6\,H_2O$) and di-sodium hydrogen phosphate ($Na_2HPO_4 \cdot 12\,H_2O$) chemicals were used as magnesium and phosphate sources. Weighed chemicals and 200 mL of leachate were added into a 250 mL beaker. After mixing at 90 rpm for 5 min, the pH was adjusted with 10 M NaOH to obtain the minimum MAP solubility. The solution was mixed at 90 rpm for 15 min and allowed to reach equilibrium and stable pH. The sample was then allowed to settle for 30 min in order to separate the MAP crystals from the bulk liquid. After precipitation, the supernatant was used for ammonium, phosphate, magnesium, color and turbidity measurements.

2.4 Analytical Methods

The performance of the UASB reactor was monitored using the results of COD, BOD_5, and biogas measurements. The data obtained from the ammonium, phosphate and magnesium measurements were used to evaluate the performance of the MAP process. COD analysis was performed according to the closed-reflux method [18]. The pH was monitored with the help of a Jenway ion electrode and the amount of magnesium was performed using atomic absorption spectrophotometry (Unicam 929A). Color was measured using a Merc SQ 118 spectrophotometer. The volume of biogas produced and its composition were estimated using an Orsat apparatus (APHA, 1989). BOD_5, COD, volatile fatty acid(VFAs), ammonium, Kjeldhal nitrogen and phosphorous analyses were performed according to Standard Methods [18]. The difference between Kjeldhal nitrogen and ammonium represents the organic nitrogen level present.

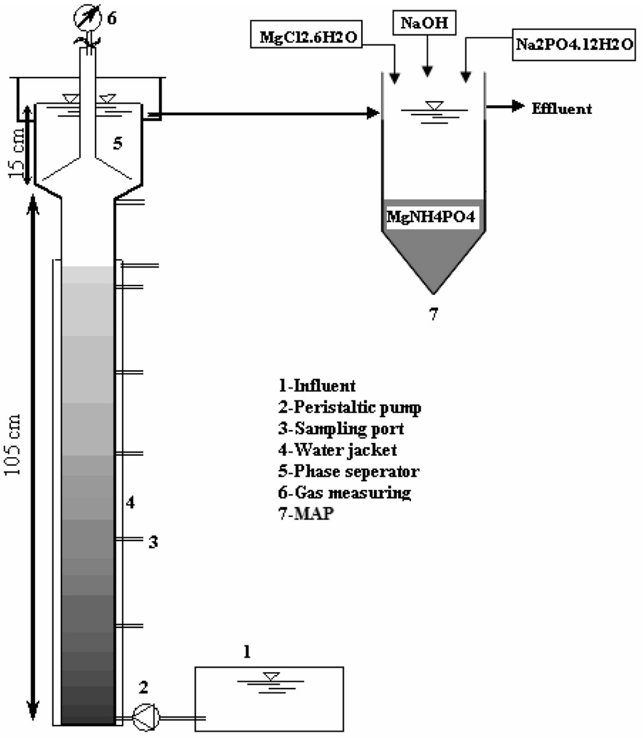

Figure 1. Schematic diagram of the combined treatment system.

Table 1. Composition of raw leachate used in anaerobic treatment.

Parameter	Value	Parameter	Value
pH	7.2–8	Ca^{2+} (mg/L)	250–900
COD (mg/L)	14000–33000	Mg^{2+} (mg/L)	420–600
BOD_5 (mg/L)	6600–21500	Na^{2+} (mg/L)	2500–3200
VFAs (mg/L)	3160–5880	K^{2+} (mg/L)	1600–2300
TSS (mg/L)	480–1270	Fe^{2+} (mg/L)	30–100
NH_4N (mg/L)	2070–2730	Mn^{2+} (mg/L)	5–25
Org-N (mg/L)	30–400	Zn^{2+} (mg/L)	0.5–1.3
Total Phosphorus (mg/L)	17–37	C^{2+} (mg/L)	0.7–1.0
Alkalinity (mg/L $CaCO_3$)	10000–19000	Ni^{2+} (mg/L)	1.2–1.7

3 Results and Discussion

3.1 Leachate Characteristics

The composition of leachate used in the experiments is shown in Tab. 1. The leachate has high values of organic pollutants, and COD and BOD_5 levels increases up to 33000 and 21500 mg/L, respectively. The concentration of VFAs varies between 3160 and 5880 mg/L and these values indicate that 1/3 to 1/4 of total BOD_5 is contributed to by VFAs. Butric and acetic acids were the main composition of VFAs (40–65%), which indicates that the degradation of waste in the landfill is in the early stages.

The total nitrogen concentration of leachate varies between 2100 to 3170 mg/L. The nitrogen composition of leachate is mainly composed of ammonium (90%) and the low organic nitrogen indicates that almost all the organic composition of leachate has been hydrolyzed. The total phosphorus varies between 17 to 37 mg/L and the pH was at almost neutral values.

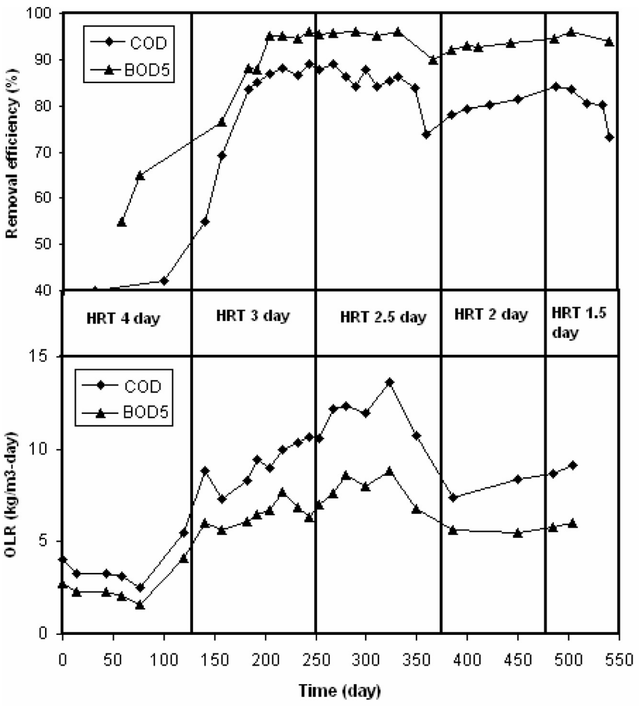

Figure 2. The effect of organic loading rate and hydraulic retention time on the performance of the UASB reactor.

The concentration values in Tab. 1 indicate that Odayeri landfill leachate has the characteristics of high strength and strong nitrogenous waste wastewater. The ratio of BOD_5/COD varied in the range of 0.45 to 0.67. Although, the leachate sample contained high amounts of alkali metals, the heavy metals were measured at very low concentrations. In the literature, it is reported that strong wastewater with high biodegradability can be treated successfully by anaerobic degradation [11, 19, 20]

3.2 UASB Performance

The UASB reactor was operated at mesophilic conditions (35°C) for the treatment of leachate for 550 days. The effect of organic loading rate (OLR) on the treatment performance of the UASB reactor was evaluated by monitoring COD, BOD_5 and biogas. Figure 2 shows the COD and BOD_5 removal performance of the UASB reactor with respect to OLR. During the operation of the reactor, the OLR was increased stepwise from 4 to 13 kg COD/(m^3d) with a reduction of the hydraulic retention time (HRT) from 4 to 1.5 days. Between 310 to 360 days, the OLR decreases with decreasing HRT values due to the fluctuations in the organic content of the leachate.

During the start-up period, i.e., days 1 to 120, diluted leachate was fed to the reactor in order allow the inoculums to adapt and the HRT was constant after four days. For the first 80 days, the OLR was decreased from 4 kg COD/(m^3d) to 2.75 kg COD/(m^3d) and the removal efficiency remained below 50% for 10,000 to 15,000 mg/L COD leachate feed. After the start-up period, the COD content of the leachate feed varied between 20,000 and 30,000 mg/L.

From day 80 to day 325, the OLR was increased from 2.75 to 13 kg COD/(m^3d) by decreasing the HRT gradually from 4 to 2.5 days. During days 120 to 250, there was an increase in the removal of the organic content of leachate with increases of the OLR. The maximum removal efficiency was obtained at 90% for COD and 95% for BOD_5 at an OLR of 9 kg COD/(m^3d) on day 200. The COD removal was in the range of 54.5 and 90.0%, and the average value was obtained as 81.2%. Moreover, BOD_5 efficiencies were in the range of 76.5 to 95.0% and the average value was 91.4%. These results are comparable with the results obtained for COD removal efficiencies in leachate treatment in the literature [21, 22].

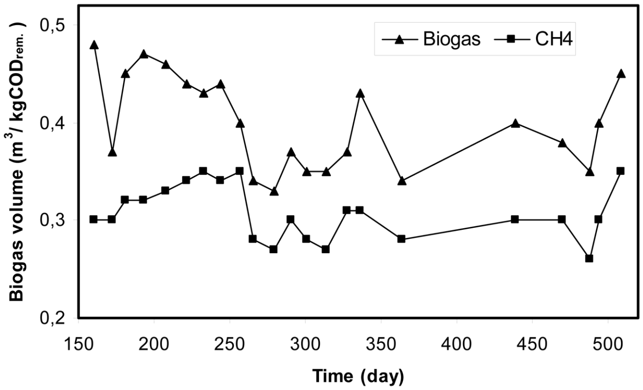

Figure 3. Biogas production at the UASB reactor.

The OLR was increased from 10 to 12 kg COD/(m^3d) at a HRT of 2.5 day and the COD removal efficiency was constant for 250 to 325 days, and the average removal efficiencies were higher than 85 and 95% for COD and BOD_5, respectively. After day 300, the performance of the reactor deteriorated with decreasing OLR levels. The removal rates of COD and BOD_5 decreased with increasing OLR levels above 12 kg COD/(m^3d), which indicates that the OLR should be lower than this level for treatment of leachate in a UASB reactor.

With the increase of OLR from 8 to 10 kg COD/(m^3d) at a HRT value of two days, the removal of COD increased slightly but there was a decrease in the removal efficiency at a HRT of 1.5 days. Consequently, the OLR has a major effect on the organics removal in the UASB and the average values at steady state were obtained as 86.3 and 95.3% for COD and BOD_5, respectively. The HRT should be higher than two days for the efficient removal of organics by anaerobic treatment. In addition, the ammonium level of the leachate decreased slightly during the operational period.

The carbonaceous content of the leachate was converted to methane and carbon dioxide during the anaerobic treatment. Figure 3 shows the methane composition of biogas produced at steady state. The range of biogas production per COD removal was between 0.33 and 0.47 m^3 and the CO_2 content changed between 20 to 23% of biogas. The methane production rate was observed as 0.27 to 0.35 m^3/kg COD removed during operation time. Therefore, the UASB reactor can be monitored by the evaluation of biogas and methane production.

3.3 MAP Precipitation

The anaerobic treatment was successful for the removal of organics but the ammonium content of leachate did not decrease and 2600 mg/L ammonium remained in the effluent of the UASB reactor. Magnesium ammonium phosphate (MAP) precipitation was carried out in order to decrease the ammonium concentration of the effluent. The characteristics of anaerobically-treated leachate used in MAP experiments are shown in Tab. 2. The effect of the pH and molar

Table 2. Characteristics of anaerobically-treated leachate used for MAP precipitation.

Parameter	Value
pH	8.3
NH_4^+-N (mg/L)	2600
Org-N (mg/L)	160
Total P (mg/L)	27
Mg^{2+} (mg/L)	370
Ca^{2+} (mg/L)	50
Color (Hazen)	2080
Turbidity (NTU)	3190
Fe^{2+} (mg/L)	17
Mn^{2+} (mg/L)	0.9

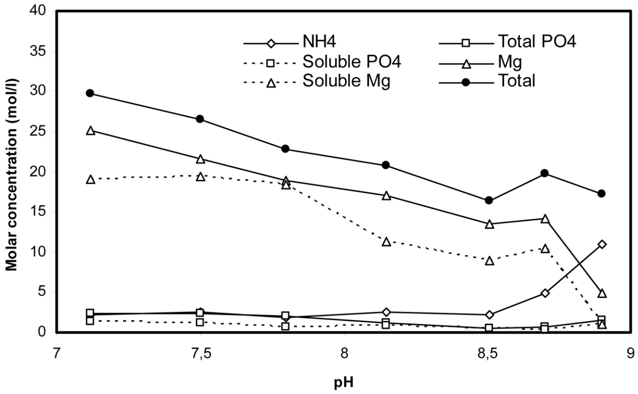

Figure 4. Variation of residual concentrations of MAP components with pH.

ratio of MAP constituents on the ammonium removal was investigated in batch mode. At optimum conditions, MAP precipitation was evaluated for the removal of ammonium, color and turbidity from anaerobically-treated leachate.

3.3.1 Effect of pH

The MAP precipitation is primarily controlled by pH because the concentration of the ions forming MAP crystals is entirely pH dependent [23, 24]. After adding the required amount of chemicals to obtain a molar ratio of $NH_4/Mg/PO_4$ of 1/1.1/1.1, MAP crystals were formed according to Eq. (1) and the solution pH decreased from 8.3 to 5.3 due to the release of hydrogen ions. Without pH adjustment, the ammonium concentration decreased from 2600 to ca. 1000 mg/L and only a minute amount of very small MAP crystals were formed in the solution. Battistoni et al. [25] reported that the Ca/Mg molar ratio strictly influences the formation of MAP and almost all crystals formed are MAP when the molar ratio of Ca/Mg is lower than 1.8. According to the concentration values in Tab. 2, the molar ratio of Ca/Mg in the leachate was 0.08. Therefore, almost all of the precipitate obtained during the experiment was expected to be MAP crystals.

$$Mg^{2+} + NH_4^+ + H_2PO_4^- \leftrightarrows MgNH_4PO_4 \cdot 6\,H_2O \downarrow + 2\,H^+ \qquad (1)$$

In order to increase the ammonium removal efficiency, the pH of the solution was increased by adding 10 M NaOH. The effect of equilibrium pH (at 25 min) on the MAP precipitation was monitored using the residual molar concentrations of ammonium, magnesium and phosphorus, and the results are demonstrated in Fig. 4. The lowest residual ammonium was obtained for a pH of 7.8. On the other hand, the remaining PO_4–P and Mg concentrations decreased gradually with an increase of the pH up to 8.5 and the lowest values were 0.57 and 13.54 M, respectively. Above pH 8.5, the ammonium and PO_4–P concentrations increased due to the decreasing solubility of the MAP crystals. As shown in Fig. 4, most of the residual magnesium in the leachate was composed of soluble Mg. Although the Mg concentration increased at pH 8.7, it decreased sharply at pH 8.9. The amount of soluble Mg was measured as 1 mol/L at pH 8.9 and it is believed that the sharp reduction in the magnesium level was caused mainly by $Mg(OH)_2$ precipitation.

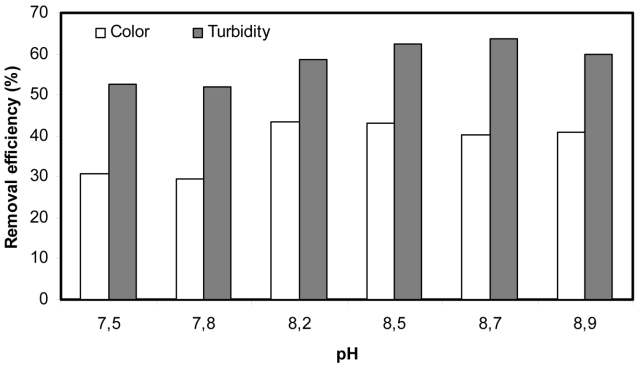

Figure 5. Removal of color and turbidity from anaeorobically-treated leachate as a function of pH.

Similarly, the total concentrations of residual ions decreased with increasing pH and the minimum value was obtained at pH 8.5. Consequently, these figures indicate that the minimum MAP solubility is achieved at a pH of 8.5 and this is consistent with the results in the literature [26, 27].

As shown in Fig. 5, MAP precipitation also enhanced the removal of color and turbidity from the leachate. A comparison of the influent and effluent results of MAP precipitation shows that the highest color and turbidity removal levels were achieved at a pH of 8.50 as 43 and 62%, respectively.

3.3.2 Effect of Molar Ratio

The effect of molar ratio on the MAP precipitation was evaluated using the residual concentrations of Mg, NH_4–N, and PO_4–P. As can be seen from Fig. 6, the residual ammonium concentration decreased gradually with the increasing molar ratio of $Mg/NH_4/PO_4$ but it remained constant above a value of 1/1/1.1. The residual concentrations of Mg and PO_4 obtained were different for all cases but the minimum total concentration was obtained at the stoichiometric ratio. The comparison of the settling characteristics of MAP sludge in Fig. 7 indicates that the ratio of settled sludge volume (SSV) increased sharply with the increasing molar ratio and has its highest values above the stoichiometric ratio. Although lower SSV values were obtained below stoichiometric ratio, the total residual Mg and PO_4 concentrations were higher. Since higher ammonium removal efficiency and lower residual ion concentrations in the supernatant and volume of sludge are difficult to handle, the optimum conditions were determined as pH 8.5 and a molar ratio of 1/1/1. At the optimum conditions, the ammonium concentration of anaerobically-treated leachate can be decreased by ca. 97% and this value is compatible with the results in the literature [13, 28]. On the

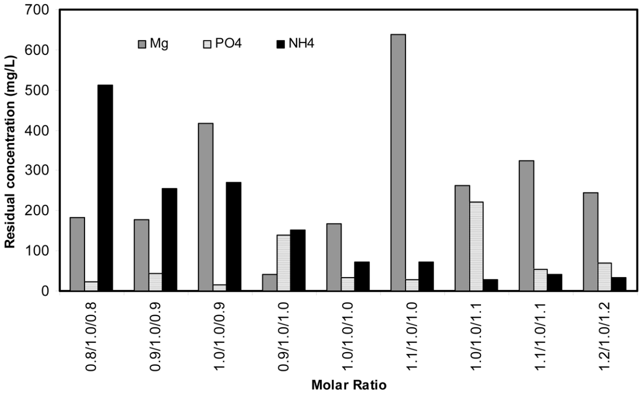

Figure 6. Concentration of residual MAP constituents vs. molar ratio (pH = 8.5).

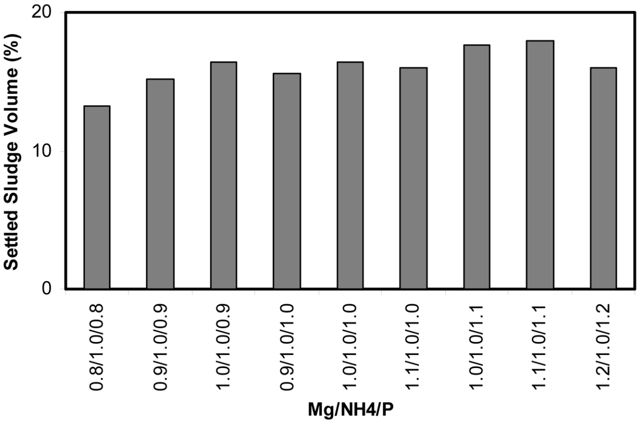

Figure 7. Settled volume of MAP sludge as a function of molar ratios.

other hand, COD and organic nitrogen could not be removed by MAP precipitation.

4 Conclusions

In the present study, the UASB reactor was shown to be feasible for the removal of organics from sanitary landfill leachate at mesophilic conditions. The average values for COD and BOD_5 were achieved as 86.3 and 95.3%, respectively at the optimum conditions of 8 to 12 kg COD/(m^3d). Although UASB resulted in a high rate for the removal of organics, ammonium removal was not observed during anaerobic treatment. MAP precipitation was applied to anaerobically-treated leachate in order to enhance the removal of ammonium effluent. At the optimum conditions, 98% of ammonium was treated successfully in MAP and it was seen that MAP precipitation has no effect on the removal of COD and organic nitrogen.

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