Simultaneous removal of organics and ammonium-nitrogen from reverse osmosis concentrate of mature landfill leachate

Tavseef Mairaj Shah*, Sreenivasan Ramaswami, Joachim Behrendt, Ralf Otterpohl

Hamburg University of Technology, Institute of Wastewater Management and Water Protection, Hamburg, 21073 Germany

ABSTRACT

Leachate treatment forms one of the main issues in landfill management. The recirculation of reverse osmosis (RO) concentrate of mature leachate, which is characterized by low biodegradability, into the landfill has not proven itself to be a sustainable treatment method. Hence alternate methods for the treatment of the concentrated leachate products have to be devised. In this work, the treatment of a mature leachate and its RO concentrate using different combinations of physico-chemical and biological treatment methods for the removal of organic carbon and ammonium-nitrogen ($N\text{-}NH_4^+$) were investigated. The coupling of electrocoagulation and ozonation with biological treatment was examined in this study. The combination of biological treatment with pretreatment processes like ozonation, electrocoagulation and adsorption proved to be a successful strategy for the removal of refractory organics as well as ammonium-nitrogen ($N\text{-}NH_4^+$). Ozonation led to a total organic carbon (TOC) removal of up to 12.5 % whereas with electrocoagulation, up to 40 % removal of TOC was observed. In combination with biological treatment, up to 90 % TOC and 65 % ammonium-nitrogen removals were observed during the experiments, all of which were performed in batch mode.

Keywords: aerobic treatment; TOC; ammonium-nitrogen; landfill leachate; reverse osmosis concentrate; ozonation; electrocoagulation

INTRODUCTION

Landfilling is the most widely used procedure to dispose of municipal solid waste [1–3]. A colossal 1.3 billion tons of landfill waste is produced worldwide annually, and is projected to increase to 2.2 billion tons by 2025 [4]. Although landfilling has proved to be a competitive waste management alternative over the decades, the production of a huge quantity of leachate is a matter of great concern. Hence, landfill management and leachate treatment form vital areas of environmental research.

Landfill leachate is the wastewater generated in a landfill as a result of percolation of precipitation, water produced from biochemical reactions within the waste mass, and the inherent water content of the waste material [5]. Landfill leachate is an example of a highly loaded wastewater containing a wide variety of pollutants, including an array of organic compounds, ammonium-nitrogen, heavy metals, and inorganic salts [5–7]. The concentrations of most components in landfill leachates exceed the concentrations of the same in sewage sludge [8].

* Corresponding author. Email: tavseef.mairaj.shah@tuhh.de, Fax: +49 40 42878 2684

Initially, large amounts of biodegradable organic compounds are present in leachates from young landfills. When anaerobic conditions develop inside the landfill, these compounds undergo anaerobic fermentation leading to the formation of volatile fatty acids (VFAs). As much as 95 % of the total organic carbon in the leachate can be made up of VFAs during this phase [9]. As the landfill gets older, the VFAs get converted into acetate and finally into landfill gas. Once the VFA content in the leachate gets exhausted, the remaining organic matter is predominantly refractory. Leachates from mature landfills contain a high quantity of refractory organic compounds, which are not biodegradable under normal conditions, and a high percentage of the total nitrogen exists as ammonium ions [10,11]. Table 1 shows the typical change in leachate quality with the ageing of a landfill.

Table 1. Characteristics of different types of landfill leachates [12,13]

Parameter	**Type of leachate**		
	Young	Intermediate	Old/Mature
Landfill age (years)	< 5	5-10	> 10
pH	< 6.5	6.5-7.5	> 7.5
Ammonia nitrogen (mg/L)	< 400	n.a.	>400
COD (g/L)	> 10	4-10	< 4
TOC/COD	< 0.3	0.3-0.5	> 0.5
BOD/COD	0.5-1.0	0.1-0.5	< 0.1
Biodegradability	high	medium	low

n.a. – data not available

Table 2. Values of different parameters of the RO concentrate from the company on-site measurement data

Parameter	**Value (mg/l)**
TOC	4060
Total nitrogen (TN)	3120
Ammonium-nitrogen	3000
pH	7.4

Environmental regulations enforce strict discharge limits for various pollutants contained in a wastewater that is to be released into natural waters like lakes, rivers, or seas. These include limits on the chemical oxygen demand (COD), total organic carbon (TOC) and ammonium-nitrogen (N-NH_4^+) concentrations among many others, which are detrimental to the hydrosphere if present in high concentrations. Selection of suitable processes for the treatment of leachate depends on its composition, which is in turn dependent on the age of the landfill.

In order to achieve high treatment efficiency and to minimize the risk of contamination, usually a combination of several physical-chemical and biological methods is used for treating landfill leachates [10,12,14–16]. High pressure membrane processes have to be utilized to complement biological and physical-chemical methods to achieve completeness of treatment. Reverse osmosis is widely utilized in combination with other treatment processes, and is in some cases employed standalone [17–19]. Although the quality of the produced permeate is very good that it can be

discharged in to nature, reverse osmosis generates a highly polluted retentate that has to be further treated [5].

Biological treatment is known to be effective in removing organics and nitrogen from young leachates with a high BOD/COD ratio. Fluidized bed reactor systems, as an example, have been reported to remove up to 82% COD and 60% DOC in young leachates [20,21]. For methanogenic leachate, the effectiveness of the process is lesser as refractory (non-biodegradable) compounds form the bulk of the organic content [5]. Welander et al. (1998) reported nearly 90% total nitrogen removal with a COD removal of only 20% from a mature landfill leachate using a suspended carrier biofilm reactor. In such cases, biological processes can be used in combination with other pretreatment or polishing processes.

The use of ozonation and electrocoagulation has been widely reported in literature [11,23–29] for the treatment of inert organics in several wastewaters. Ozonation promotes the formation of microflocs of organic matter, in addition to oxidizing recalcitrant and colour-causing humic substances to biodegradable compounds [30]. For a methanogenic leachate with low biodegradability, the pretreatment with ozonation before biological treatment hence has practical importance. Wang and Gamal El-Din [31] reported up to 100-fold increase in the BOD to COD ratio after ozonation of methanogenic leachate.

Electrocoagulation is usually used as a pre-treatment or a post-treatment polishing process, and not as a main treatment method in case of highly polluted wastewaters. Ilhan et al. [25] reported 59% decrease in COD and 14% ammonium-nitrogen removal from a mature leachate during electrocoagulation using aluminum electrodes. Djelal et al. [11] have reported removal of organics with up to 56% COD reduction using a current density of 95 A/m^2 and a contact time of 150 minutes. Most studies have reported an acidic operating pH as optimum for electrocoagulation [32].

The investigation of a possible synergy of electrocoagulation and ozonation processes in combination with biological treatment has not yet been investigated for the treatment of mature leachates. This work was aimed to be a preliminary evaluation in that direction. The goal of this study was to determine the effect of (i) pretreatment strategies of ozonation, electrocoagulation and a combination of the two and, (ii) the type of reactor system (attached growth vs. suspended growth); on the removal of TOC and ammonium-nitrogen content during biological treatment of a mature landfill leachate. The study investigated aerobic biological treatment of pretreated landfill leachate and its RO concentrate using an up-flow aerobic packed bed reactor (attached growth) and continuously stirred tank reactor (suspended growth). Ozonation and electrocoagulation were used as pre-treatment processes in the study.

MATERIALS & METHODS

Materials

The leachate used in this work was from the Ihlenberg landfill, located near the north German city of Luebeck. This landfill has been in operation since 1983 and has 113 hectares available for landfilling. With a maximum yearly intake capacity of 1 million tonnes, this landfill has been described as 'the biggest of its type in Europe' and is expected to run for many years to come [33].

The leachate generated at this landfill site, methanogenic in nature, exhibits a TOC value of 800 mg/l and an ammonium-nitrogen value of 600 mg/l. The raw leachate is subjected to an on-site multi-step purification. A two-stage reverse osmosis plant, operated at 60 and 120 bar with a total processing capacity of 40 m^3/h, recovers about 80% permeate (clean water) generating about 100 m^3 of highly polluted concentrate per day. This RO concentrate exhibits an electrical conductivity of about 92 mS/cm and TOC and ammonium-nitrogen concentrations of about 4000 mg/L and 3000 mg/L, respectively. Concentrations of other major solutes in the retentate can be found elsewhere [7]. RO retentate was provided from the landfill site for this study. The activated sludge was obtained from the county wastewater treatment plant at Seevetal (Lower Saxony, Germany) for inoculating the aerobic biological degradation trials.

Methods

The feed samples used in the experiments were different dilutions of the RO concentrate. A 5-times dilution (RO5x) of the concentrate was used as an approximation of the raw leachate (80% water is recovered from raw leachate), while as experiments were also performed with the concentrate itself (RO) and a 3-times dilution of the same (RO3x). The dilutions were done with deionized water. Dilution was done mainly to investigate possible inhibitory effects of contaminants like chloride as the given leachate had a very high chloride content (30,000 mg/l in the RO concentrate).

Ozonation

Figure 1 shows a schematic of the ozonation setup used in the study. Oxygen (99.99%, Westfalen AG, Germany) was supplied to an ozone generator (Labor-Ozonisator 301.7, Erwin Sander Elektroapparatebau GmbH, Germany) at 1.2 bar (abs) at a flowrate of 120 L/h, producing 2.125 g O_3 per hour. The generated ozone was fed into a coiled tubular reactor (8 mm ID, 18.6 m long, made of Teflon) in which it came into contact (cocurrent) with the leachate. The leachate was pumped using a Masterflex variable speed peristaltic pump (Cole-Parmer Instrument Company, Illinois, USA) at a flowrate of 350-400 mL per minute. Ozone dosages ranging between 0.2 to 1.3 g/$L_{leachate}$ were used during the experiments, corresponding to ozonation times of 5 to 30 minutes. The ozonated leachate was recirculated into the feed flask. The residual ozone in the leachate was removed by bubbling gaseous nitrogen before it was fed for biological treatment.

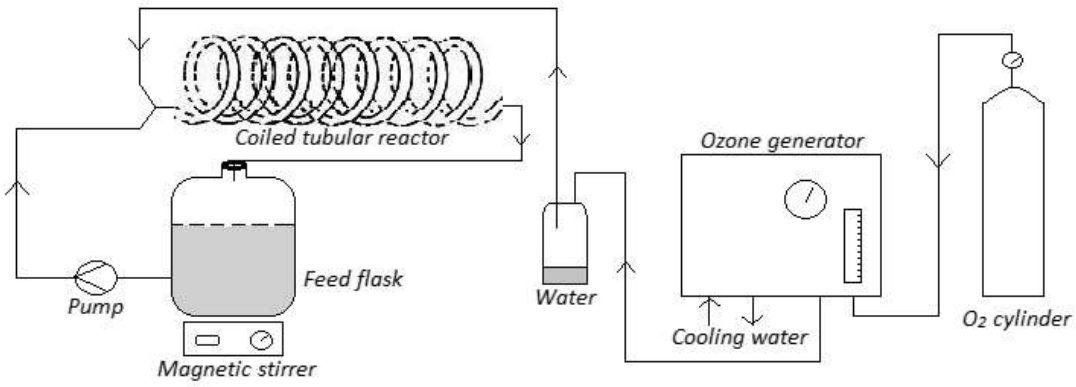

Figure 1. Schematic representation of the ozonation setup used in the study

Electrocoagulation

Figure 2 shows a schematic of the electrocoagulation setup. For all experiments, 700 mL of raw

leachate or its RO concentrate was taken in a HDPE cuboidal vessel (10.5 cm long, 9 cm wide and 15.5 cm high). Aluminum electrodes (EN AW 5754 $AlMg_3$), 10x10 cm (thickness 1 mm) were fully immersed into the leachate separated by 8 cm, and were connected to an AC-to-DC converter (6225 SMPS DC, PeakTech Prüf- und Messtechnik GmbH, Germany). Experiments were performed with current densities ranging from 8-20 mA/cm^2 corresponding to dosages of 1-3 g Al^{3+}/L. The reported dosages are theoretical values based on Faraday's law. The electrocoagulation mixture was continuously stirred at 200 rpm using a RZR 2020 overhead stirrer (Heidolph Instruments, Germany). After the experiments, the mixture was subjected to centrifugation at 3000 rpm for 30 minutes using a Heraeus Multifuge X3 centrifuge (Thermo Fisher Scientific, Massachusetts, USA).

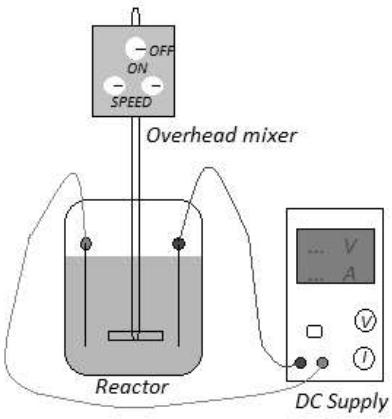

Figure 2. Schematic representation of the electrocoagulation setup

Biological treatment

Two types of reactors were used for the aerobic post-biological treatment of the leachate or its concentrate. Suspended growth process was realised using two continuously stirred tank reactors (CSTR) and attached growth system was realised by a two packed bed reactors (PBR). Crushed coke with an equivalent diameter of 1-3 mm was used as the packing material in plexiglas tubes of 2.2 cm diameter and 51.2 cm height to form the packed bed. The resulting void fraction of the packed bed was 27–30%. A 20 times diluted solution of activated sludge in deionized water was pumped through the packed bed overnight before the experiments were started, to inoculate the columns. Figure 3 shows a schematic of the PBR system used in this work. The leachate was pumped using a Pumpdrive 5001/5101 (*Heidolph*, Germany) operating at 40 rpm, corresponding to a flowrate of 10-11 L/h from bottom to top in the packed bed reactors, before being recycled back into the feed tank. Compressed air at 2 bar pressure was supplied cocurrently at 40 L/h. The air flow rate was regulated using Vögtlin 100-300.01 rotameter (*Vögtlin*, Switzerland). The CSTR system consisted of a 1000 ml Schott bottle placed on a magnetic stirrer. The leachate samples were pretreated with ozonation or electrocoagulation before being fed to the respective biological treatment systems. All experiments were carried out in duplicate using two reactor setups each for PBR and CSTR. Samples were taken on a daily basis and analysed to determine the TOC and ammonium-nitrogen concentrations.

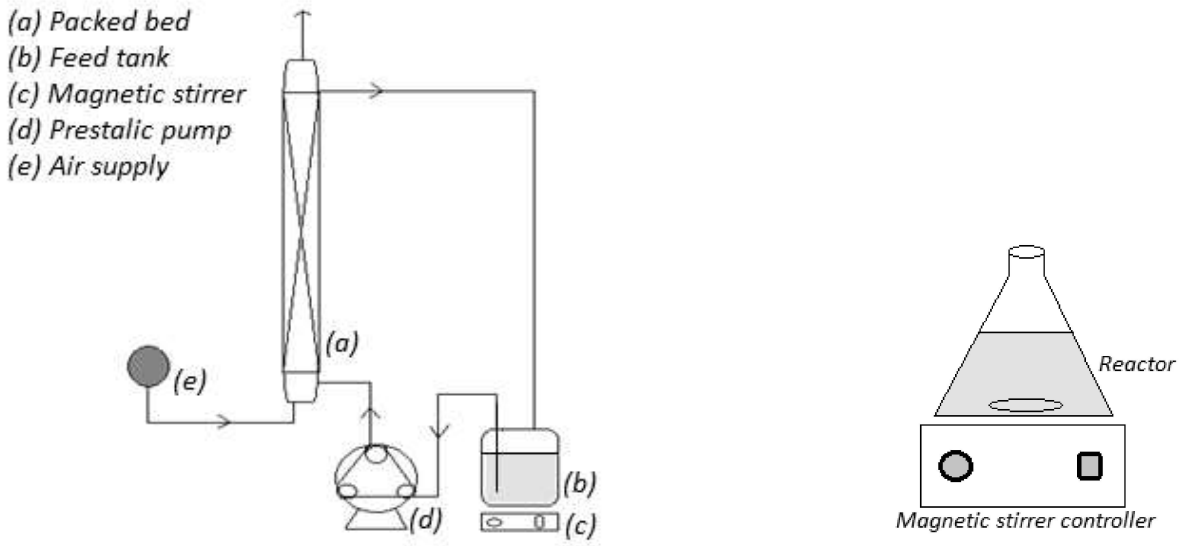

Figure 3. (Left) Schematic representation of the experimental PBR setup for aerobic biological treatment. (Right) A schematic of the CSTR setup.

Analyses

TOC and total nitrogen (TN) concentrations in the samples were determined using a *Multi N/C 3000* analyser (*Analytik*Jena, Germany) following the German standard methods DIN EN 1484-H3 and DIN EN 12260, respectively. Ammonium-nitrogen concentration was measured using a V-550 UV/visible spectrophotometer (*Jasco Analytical Instruments*, Germany) following the German standard method DIN 38406-E5-1.

RESULTS & DISCUSSION

Removal of organics

The following table gives a summarized overview of the measured TOC values of different streams after the pretreatment processes in some experiments of this study. In pretreatment, O-*xy* represents ozonation for *xy* minutes and E-*xyz* represents electrocoagulation for *xyz* minutes. COD measurements were found to be inaccurate, possibly due to interference by high chloride concentration in the samples [34,35]. The samples had to be diluted before accurate COD measurements could be made and it was a time-intensive process, hence the focus was kept on TOC measurements. The COD of the RO concentrate as reported in the on-site measurement data was 11700 mg/l O_2 and was reduced to 6860 mg/l O_2 during one of the ozonation (O-20) pretreatment experiments.

Table 3. TOC removal in combined pretreatment experiments

Feed	Initial TOC	Pretreatment	O_3 dosage (g/L)	TOC removed (%)	Al^{3+} dosage (g/L)	Total TOC removed (%)
RO5x	692	O-5	0.18	**2.5**	**---**	**2.5**
RO5x	655	O-7	0.25	**3.8**	**---**	**3.8**
RO5x	638	O-9	0.32	**4.7**	**---**	**4.7**
RO3x	1100	O-20,E-180	0.9	**6.4**	1.15	**17.7**
RO	3630	O-30,E-240	1.3	**12.5**	1.15	**34.7**
RO5x	660	O-20,E-360	0.9	**6.4**	2.91	**34.5**

Figures 5a and b show the observed TOC removal during electrocoagulation (at different pH) and ozonation respectively, for increasing dosages of Al^{3+} and O_3. A maximum of 12.5 % TOC removal was observed for an ozone dosage of 1.3 g/L (349 mg TOC/g O_3) during the pretreatment of RO concentrate. On the other hand, a maximum of 27 % and 40 % TOC removal was observed during electrocoagulation of RO concentrate and raw leachate for Al^{3+} dosages of 1.57 g/L and 1.86 g/L, respectively. This corresponds to TOC removals of 665 mg TOC/g Al^{3+} and 219 mg TOC/g Al^{3+} respectively, for RO concentrate and raw leachate.

An acidic pH value of 4.5 was found to be optimum for the removal of organics during electrocoagulation. The higher removal rates can be attributed to the increased concentrations of oxidizing species (e.g. highly reactive hydroxyl radicals weakly adsorbed at anode surface and active chlorine species electro-generated in the bulk solution) under acidic pH conditions [36].

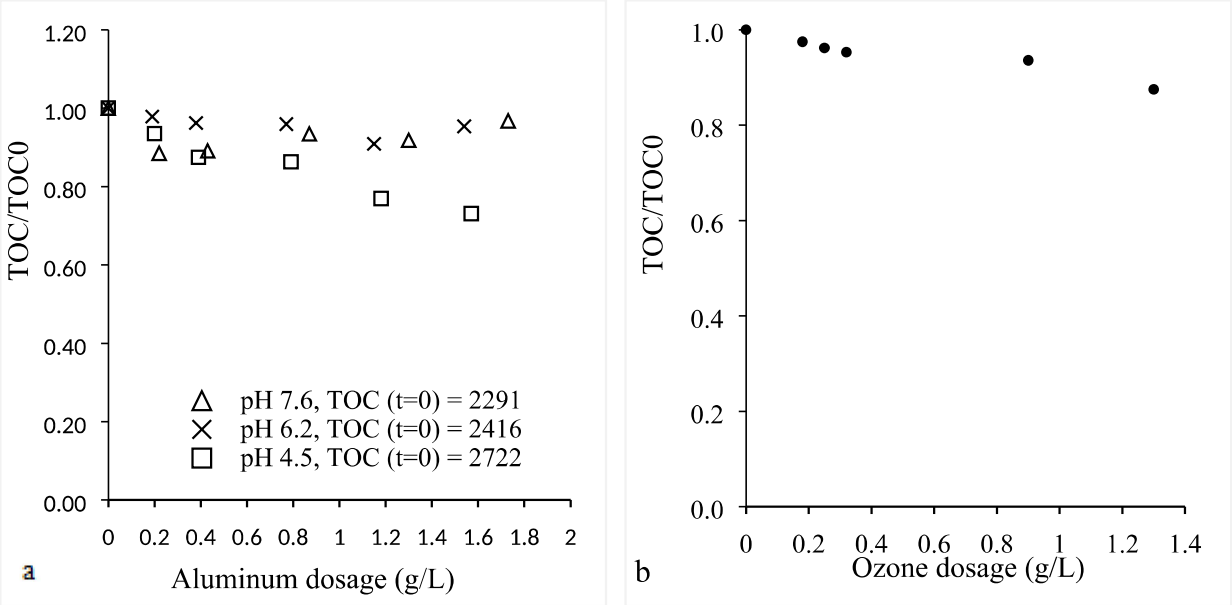

Figure 5. (a) Decrease in TOC concentration of RO concentrate with Al^{3+} dosage at different pH. (b) Decrease in TOC of RO concentrate of leachate with increase in ozone dosage (TOC_o = 3630 mg/L).

Figure 6a shows the measured TOC concentration averaged from the two fixed bed reactor systems. Removal of TOC during aerobic treatment was highest at 90% in the first run (Trial 1). TOC removal decreased progressively during subsequent trials, indicating the influence of adsorption of the organics on the packing material. A steep decrease in the TOC concentration was observed in the first 24 hours followed by a gradual decrease over the following days. This trend was observed in the later experiments as well in which a lesser percentage of TOC was removed during the biological treatment. In the later experiments, the effect of adsorption was less marked. This can be attributed to the decreased absorption capacity of the packing over the course of experiments (Figure 6b).

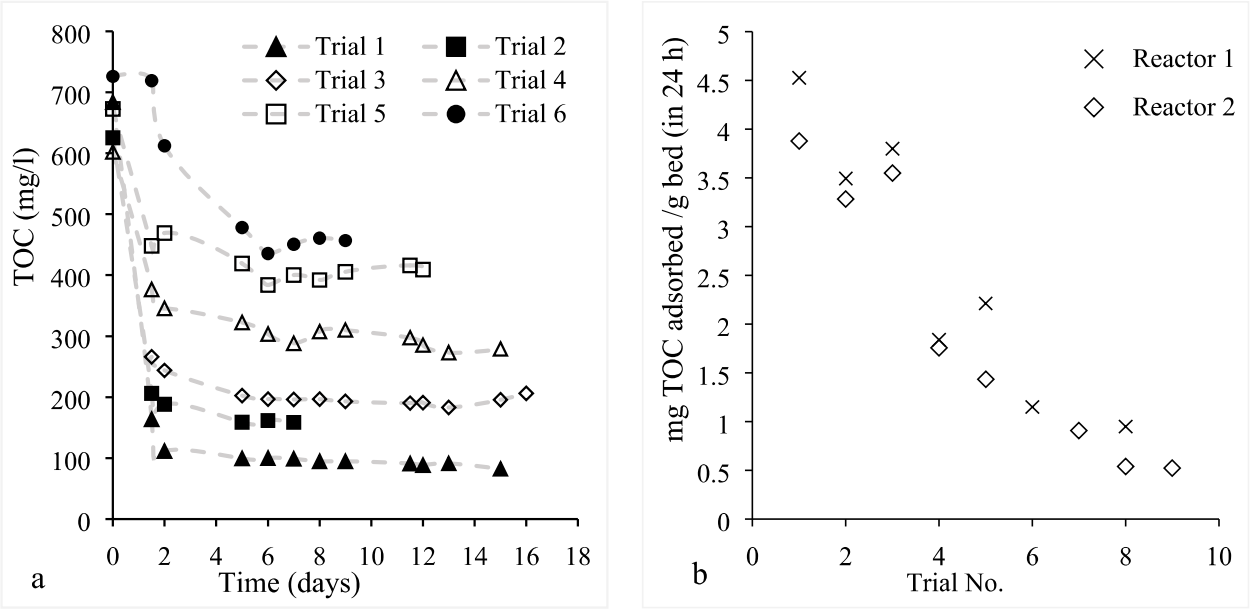

Figure 6. (a) Comparison of the TOC decrease in experiments from first to sixth trials, highlighting the effect of adsorption on TOC removal. (b) Progressive decrease of TOC removed during the initial 24 hours in different experiments.

Xing et al [37] have reported a maximum adsorption capacity of 200 mg TOC per gram of coal-based, micro-porous powdered activated carbon (PAC), whereas Fettig et al. [38] have reported a lower adsorption capacity of 60-80 mg TOC per gram of PAC. The packing material used in our experimental study was just crushed coke. So a lower value adsorption capacity was observed during the experiments. The efficiency of using adsorption as a single process for the treatment of mature leachates has been described as weak and been classified as an expensive method [39].

The removal of TOC during biological treatment was larger when the leachate samples were pretreated with ozone as compared to the samples that were pretreated with electrocoagulation and the samples without any pretreatment. Although electrocoagulation removed a larger percentage of TOC as compared to ozonation in the pretreatment, the TOC removal in the whole process together with biological treatment was higher when ozonation was used for pretreatment (Figure 7a). Nevertheless, a feed pretreated with electrocoagulation exhibited better response during biological treatment than a feed with no pretreatment at all. This can be attributed to more availability of organics to biological activity due to the removal of suspended and inorganic solids that might have taken place during electrocoagulation [11].

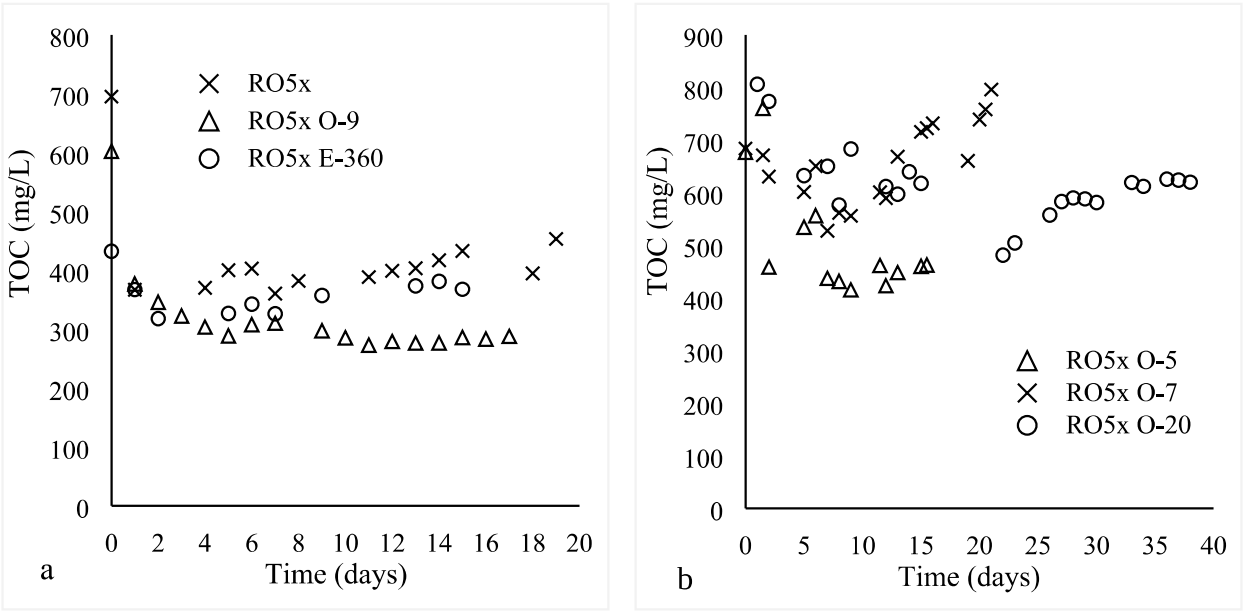

Figure 7. Comparison of TOC decrease during biological treatment of leachate. (a) PBR: Leachate without pretreatment (RO5x), pretreatment with ozonation for 9 minutes (O-9), pretreatment with electrocoagulation for 6 hours (E-360). (b) CSTR: Pretreatment with different ozonation times; 5, 7 and 20 minutes.

In experiments where no decrease in the organic content due to biological activity was expected, due to low biodegradability of the feed, the TOC value followed a progressive increase over the course of the experiment after showing a steep decrease in the beginning (Figure 7a). This increase in the observed TOC is attributed to desorption of the organics that might have initially been adsorbed on the packing. This supports the notion that organic content was not stripped away by air but was adsorbed in the packing and subsequently degraded biologically, if it is biodegradable.

Figure 7b shows the results from the experiments carried out parallelly in suspended growth system. Removal rates were slower in the CSTR than in the attached growth system. It was observed that in packed bed reactors, the same extent of TOC removal was achieved in less than half the time as was required by the CSTR. Hence attached growth system was more effective for TOC removal than suspended growth system. Faster removal in the case of packed beds is due to the phenomenon of adsorption. Adsorption facilitates the biological degradation of organics, wherein organics are first adsorbed and then subsequently biodegraded in the packed beds.

The hike in the concentration of TOC towards the end of the experimental runs, typically seen in the case of CSTR, as seen in Figure 7b can be attributed to endogenous respiration and cell lysis occurring in the treatment volume. Endogenous processes in biological wastewater treatment systems have been widely reported to affect the effectiveness of the treatment systems [40].

Removal of ammonium-nitrogen

The removal efficiencies of the different process schemes were also evaluated in terms of ammonium-nitrogen concentration. PBR was observed to be effective for the removal of ammonium-nitrogen present in the raw leachate and RO concentrate. A decrease in the ammonium-nitrogen content during biological treatment was observed even in absence of an ozonation pretreatment. Ozone pretreatment of the feed, however, led to a greater removal of ammonium-nitrogen during the succeeding biological treatment. Ozonation serves to promote biological nitrification by reducing organic matter

that is the energy source of heterotrophic bacteria, which are the competitors of the nitrifying bacteria [30].

However, an overdose of ozone was found to hinder a further decrease in ammonium-nitrogen. For an ozone dosage of 0.9 g/L, the decrease in the ammonium-nitrogen content during the biological treatment was 67 % and for a dosage of 1.3 g/L it was 48 % only. This can be attributed to the fact that ozonation destroys some of the alkalinity in aged leachates, the lack of which (lower buffer capacity) leads to pH decline during nitrification, which ultimately arrests the process [12,41].

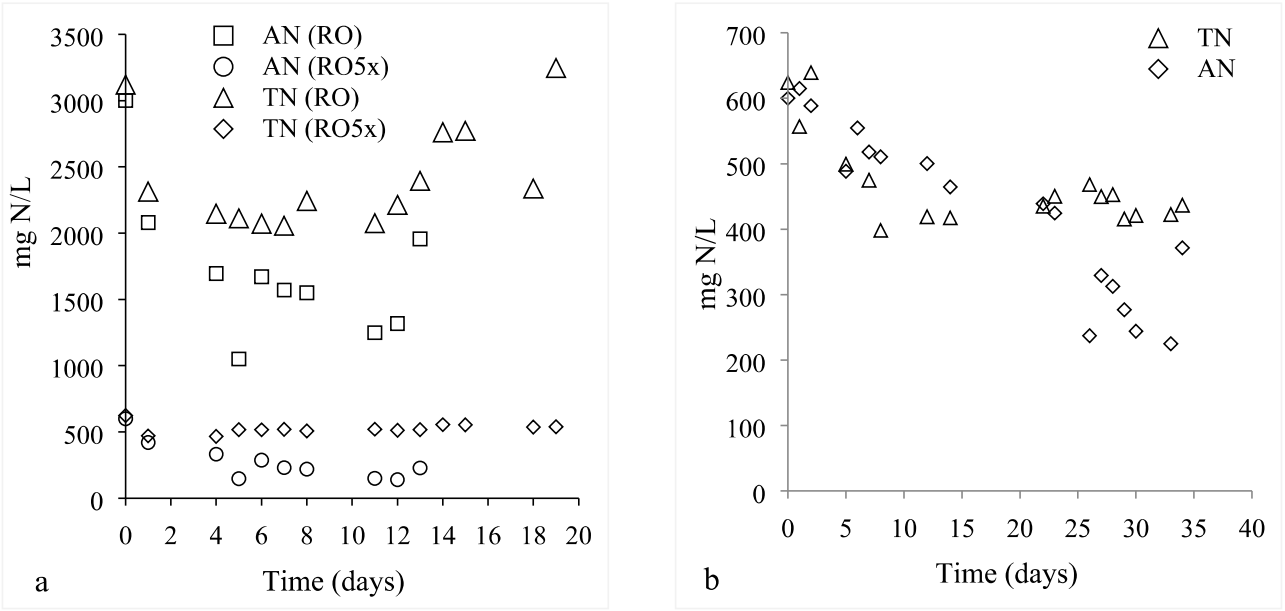

Figure 8. Removal of ammonium-nitrogen (AN) and total nitrogen (TN) during biological treatment: (a) Raw leachate (RO5x) and RO concentrate (RO) without pretreatment in PBR. (b) Pre-ozonated raw leachate in CSTR.

As can be seen from the Figures 8, the decrease in the ammonium-nitrogen is rapid in the first days of the biological treatment batch followed by a gradual decrease over the next days in case of attached growth system. This supports the suggestion that the components first get adsorbed on the surface of the packing material and are then successively biologically removed, as was observed in TOC removal as well. Ghauri et al (2012) have also reported, that the adsorption of ammonia on activated carbon takes place in two phases – a relatively fast phase followed by a slower one [42]. In case of the suspended growth system, however, the decrease is gradual throughout. For ammonium-nitrogen removal as well, attached growth systems proved to be more efficient than suspended growth systems, as was seen for TOC removal.

It was observed that pretreatment with electrocoagulation hinders the nitrification process (see Figures 9a-b). After a pretreatment with ozonation alone, the biological treatment resulted in a 67% decrease in ammonium-nitrogen; whereas as for the same feed subjected to electrocoagulation after ozonation, the ammonium-nitrogen decrease during biological treatment was 38% only.

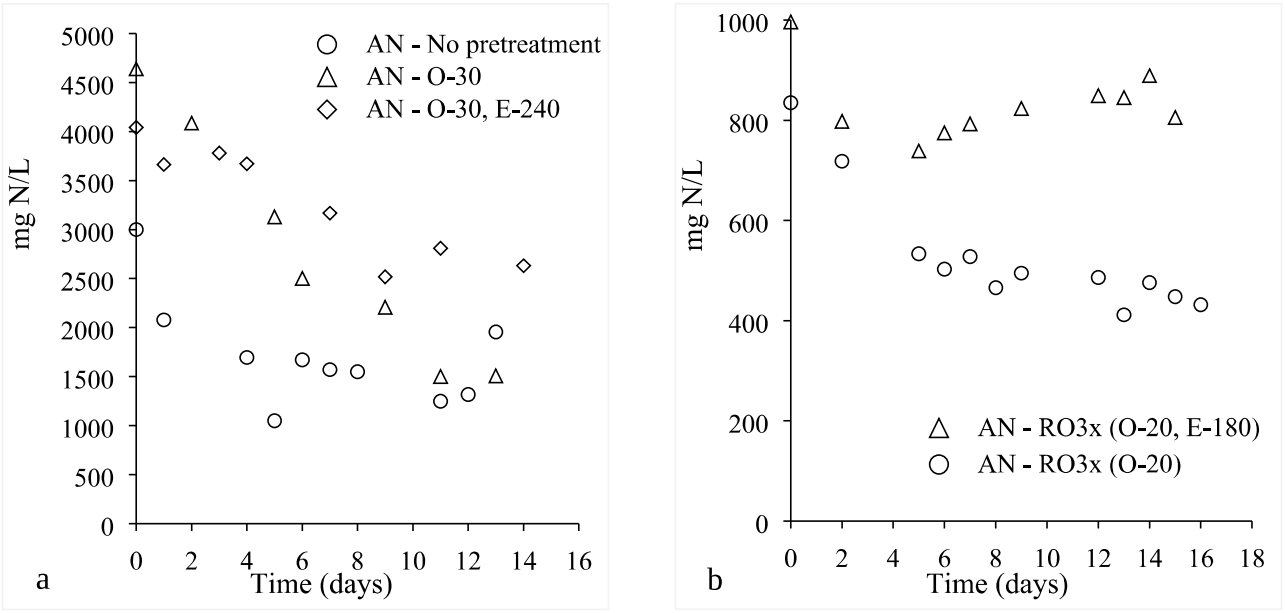

Figure 9. Comparison of ammonium-nitrogen (AN) removal during biological treatment in PBR: (a) RO concentrate without pretreatment and with different pretreatment strategies. (b) 3-times dilution of the RO concentrate with different pretreatment strategies.

If the current density applied during electrocoagulation exceeds a certain value, the ability of the bacteria to oxidise ammonia is affected. The extent of this negative effect on nitrification depends also on the duration for which the current is applied. This adverse effect can be attributed to the inhibition of the metabolism of nitrifying bacteria due to the application of electric current [43,44].

The total nitrogen measurements during the experiments showed that TN did not follow a decreasing trend proportional to the decrease in ammonium-nitrogen. This supports that the ammonium-nitrogen was oxidized to nitrate.

CONCLUSION

Ozonation led to a significant improvement in the TOC-removal effectiveness of the biological treatment process. Up to 12.5 % removal of the initial TOC of the RO concentrate was achieved during ozonation with subsequent removal during biological treatment. Packed bed reactor system was observed to be a good way to realise an efficient attached growth system with TOC removal up to 90 % from an ozone-pretreated RO concentrate in a freshly packed PBR. Adsorption played a major role in the removal of pollutants during the biological treatment in packed bed reactors. The possibility of harnessing the adsorptive capacity of the packing material in synergy with biological treatment might be given due consideration in the design of an attached growth system.

Electrocoagulation had an adverse effect on ammonium-nitrogen removal as was the case with an overdose of ozone. Ozone over-dosage led to a decrease in ammonium-nitrogen removal by up to 20 %. Electrocoagulation on the other hand led to an even greater decrease in the removal rates of ammonium-nitrogen – overall up to 30 % lower removal was observed. If the aim is the simultaneous removal of inert organics and ammonium-nitrogen, this should be duly considered while designing an RO concentrate treatment scheme. The use of electrocoagulation in combination with biological treatment might be more effective as a polishing process than a pretreatment process when the simultaneous removal of TOC and nitrogen content is intended.

For a packed bed reactor, regular cleaning of the bed would help maintain a high adsorptive capacity leading to more effective removal of TOC. A recirculation of the treated effluent with a 2nd ozone treatment in between two biological treatment steps could also be a productive prospect. This could potentially lead to a complete removal of TOC from the RO concentrate.

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