Ozone treatment of organic micro-pollutants in sewage sludge

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Abstract Organic micro-pollutants accumulate to a certain extent in sewage sludge and are therefore ecologically relevant if the sludge is to be reused in both agriculture and landscaping applications. This paper describes the degradation of endocrine-disrupting compounds and other micro-contaminants by means of ozone and ultrasonic treatment. One of the findings is that, as expected, aromatic compounds such as polycyclic aromatic hydrocarbons can be degraded easily by ozone. Thus it could be shown that, apart from enhanced VSS reduction and increased biogas production, sludge disintegration processes using ozone provide an additional benefit, i.e. the degradation of micro-pollutants, which is practically unattainable with ultrasonic treatment.
Keywords: endocrine disrupting compounds; micro-pollutants; ozone; sewage sludge; sludge disintegration; ultrasonic.

1. Introduction

1.1. The relevance of organic micro-pollutants in sewage sludge

Micro-pollutants have been detected in surface waters in several European countries (Kreuzinger, 2002; Andersen et al., 2003) and they are of increasing importance in water pollution control. In particular, the role of chemicals that are suspected of having an impact on the hormonal systems of humans and wildlife, i.e. endocrine-disrupting compounds (EDC) and other micro-pollutants such as pharmaceutical compounds, has to be emphasised. It is thought that endocrine-disrupting compounds and pharmaceutical residues enter rivers, streams and surface waters through the effluents and residual sludge from wastewater treatment plants (WWTPs). Therefore, the elimination of these xenobiotic substances in WWTPs is of elementary interest. At WWTPs operated with a solids retention time (SRT) of longer than 10 days, relatively low effluent concentrations can be achieved, which leads to a significant reduction in emissions (Clara, 2005). As compared to design guidelines, the recommended minimum sludge age corresponds to the requirements for nitrogen removal (nitrification, denitrification) and according to the urban wastewater directive of the European Community (91/271/EEC), this recommended treatment efficiency tallies with the standards for WWTPs located in sensitive areas. It can be concluded that WWTPs with nitrogen removal also efficiently remove degradable micro-pollutants. However, a large fraction of micro-pollutants is adsorbed into the sludge and thus has to be considered during sludge processing. In addition to heavy metals, which are already regulated in most countries, it has become clear that organic contaminants such as plasticizers and surfactants and their primary degradation products can accumulate, especially during anaerobic processing (Rogers, 1996).

The following organic pollutants are usually detectable in wastewater sludges: adsorbable organic halogens (AOX), linear alkylbenzene sulfonates (LAS), hydrocarbons (HC), nonylphenols/ nonylphenol ethoxylates (NPE) and di-ethyl-hexyl-phthalate (DEHP). Other chemicals that are toxic to human beings and threaten potentially chronic long-term effects are polycyclic aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB) and polychlorinated dibenzodioxins, and dibenzofurans (PCDD/F).

Furthermore, it has been observed that the level of these xenobiotics (LAS, PAH, NPE, DEHP) is much higher in anaerobically digested sludge than in aerobically stabilized sludge (Fountoulakis et al., 2005). This indicates that they can be partly or fully degraded under aerobic conditions, but not under anaerobic conditions (Knudsen et al, 2000). However, recent findings indicate that degradation of LAS is possibly initiated by desulfonation under anaerobic conditions (Haggensen et al., 2002).

The aerobic post-treatment of anaerobically digested sludge represents an established solution for the elimination of organic sludge pollutants. However, for certain organic, xenobiotic substances its efficiency is limited. Blika et al. (2007) have reported on the limited removal of 4-NP techn., phenanthrene, fluoranthrene and pyrene even after extended aerobic treatment.

In recent years, the ultrasonic pre-treatment of sludge upstream to anaerobic digestion has been investigated by a number of research groups. One of the main advantages of this technique is that the high energy intensity enhances the disintegration of particulated matter (Wang et al., 2005), as evidenced by a reduction in particle size and an increase in the soluble matter fraction. Research has also focused on the effects of ultrasonic pretreatment on environmental micropollutants (Benabdallah El-Hadj et al., 2007). The intensification of organic matter solubilization induced by ultrasonic action can lead to an increase in the bioavailability of some micropollutants (Tiehm, 1997).

Within the context of sludge disintegration, a comparative study sought to investigate whether disintegration would reduce the content of organic micro-pollutants in the residual digested sludge. In the course of these experiments, ozone and ultrasonic sludge treatment were subjected to parallel testing and a special focus was placed on the removal of NPE (nonylphenol and nonylphenol ethoxylates with one or two ethoxy groups) and polycyclic aromatic hydrocarbons (PAH). Other compounds investigated included linear alkylbenzene sulfonates (LAS) and di-ethyl-hexyl-phthalate (DEHP).

2. Material and Methods

2.1. Sludge Samples

The tested sludge was taken from a municipal WWTP nearby Vienna that is designed for 300,000 p.e. and treats the wastewater from 12 surrounding municipalities and three industrial dischargers.

2.2. Sludge disintegration units

The ozonization plant unit consisted primarily of a tube-type, laboratory ozone generator (Kaufmann OZ-LC) with a maximum capacity of 8 g ozone per hour. At the beginning of the experiments, technical oxygen gas (in gas bottles) was utilized as an oxygen source, but in the course of the experiments it was substituted by a pressure swing adsorption unit (Air Sep AS-12). A Venturi-type injector system was employed for the injection of the ozone gas into the sludge suspension. With this type of injector, high gas transfer rates with respect to specific power input are achieved due to the high turbulence in the bio-suspension (flow rate 1.5 m/s) caused by a progressing cavity pump. This generates micro-bubbles, which inhibit coalescence. The injection system is located in an external loop, which is connected to the reaction tank. For the comparative test with ultrasonic treatment, VTA GmbH provided a lab-scale system (Type GSD), consisting mainly of a reaction tank, stirrer, sonotrode and control device. This system was operated with the expert assistance and supervision of a specialist from the supplier company.

2.3. Analytical methods to determine micro-pollutants

The standard CEN/TC 308/WG 1/TG 4 N 30 method for the determination of the PAH, DEHP, NPE and LAS environmental contaminants in sludge was prepared for the Danish Environmental Protection Agency by the Water Quality Institute of the National Environmental Research Institute Miljøkemi Inc. The analyses were conducted by the Austrian Federal Environment Agency according to the aforementioned standard method.

3. Result and Discussion

In the first series of experiments only ozonization was considered for the investigation of the effects on micro-pollutants in the sludge. In this context, digested sludge from a full-scale digester was treated batchwise in the lab-scale ozone plant. The specific ozone doses applied to the sludge were 0.021 g O_3/g DS (test I); 0.032 g O_3/g DS (test II); and 0.046 g O_3/g DS (test III). These settings were chosen in order to identify the lower limit of effective ozone application within the context of micro-pollutant reduction. Based on the analysis results of the parameters PAH, NPE, LAS and DEHP, the effects on organic xenobiotic sludge compounds were monitored.

In the second series of experiments, digested sludge of the same origin was subjected to disintegration with ozone and parallel ultrasonic treatment. For ozonization, the following specific ozone doses were applied: 0.046 g O_3/g DS (ozone test IV); 0.063 g O_3/g DS (ozone test V); 0.081 g O_3/g DS (ozone test VI). These settings were selected in view of the results gained from the first series of experiments, which revealed that a specific ozone dose of 0.046 g O_3/g DS appeared to be the lowest effective dosage capable of reducing the PAH concentration to below the proposed EU standard value.

As far as ultrasonic treatment is concerned, the reaction time is determined by the specific energy input. Typically, depending on the treatment objective, energy input ranges from 0.1 - 0.2 kWh/kg DS, which is why the following energy doses were selected for the investigation of the effect of ultrasound: 0.15 kWh/kg DS (ultrasound test I); 0.20 kWh/kg DS (ultrasound test II); 0.30 kWh/kg DS (ultrasound test III). The experiments were conducted in order to be able to assess the disintegration effects of both ozonization and ultrasonic treatment in view of their potential to reduce organic micro-pollutants in digested sludge.

3.1. Nonylphenols, NPE

The NPE content in the untreated sludge samples investigated, varied in a range of 25-51 mg NPE /kg DS. It was noted that the concentration of 4-NP-techn. remained more or less stable, whereas the portions of NP1EO and NP2EO were about 6-times higher in the second series of experiments as compared to the preceding experiment

As opposed to most of the other types of surfactants, nonylphenol polyethoxylates are biotransformed starting at the hydrophilic part of their molecules. The degradation proceeds by shortening the polyethoxylate chain, while leaving the hydrophobic part of the molecule untouched (Van Ginkel, 1996). Such a biotransformation pathway results in the formation of a number of stable metabolic intermediates including 4-NP-techn., short chain nonylphenol polyethoxylates (NP1EO, NP2EO) and nonylphenoxy carboxylic acids (Ahel et al.,1994). Nonyphenol (4-NP-techn.) is considered to be a pseudo-estrogen and moreover is the most recalcitrant intermediate of the nonylphenol polyethoxylates. That explains why the NPE content in sludge is only biodegradable to a very limited extent (Blika .S. et al., 2007 and Scheffknecht, 2005).

After ozonization, the elimination rates of total NPE in the sludge were considerably higher, amounting to 37%-70.5% at specific ozone doses of 0.021-0.046 O_3/g DS (see Table 1) and 33.5-63% at specific ozone doses of 0.046-0.081 O_3/g DS (see Table 1). In particular, the reduction of nonylphenol (4-NP-techn.) was remarkably efficient. Figure 1 shows the reduction of the NPE compounds in the second series of experiments. The ozonization elimination rates for NPE amounted to 33.5% at 0.046 g O_3/g DS, 46% at 0.063 g O_3/g DS and 63% at 0.081 g O_3/g DS. As indicated in Figure 1, the reduction of NPE due to ultrasonic treatment was about 8-10%, the ratios remaining practically unaffected with regard to the individual NPE compounds.

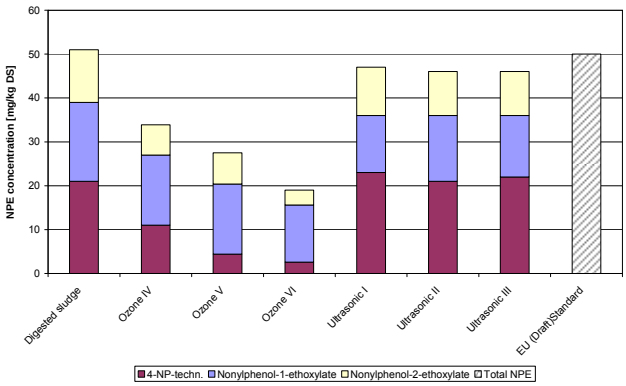

Figure 1 Concentration of NPE in digested sludge and sludge disintegrated by ozone and ultrasonic treatment

Degradation rates of up to 88% were achieved for 4-NP-techn., while the nonylphenol polyethoxylates (NP1EO, NP2EO) were only partly affected by ozone treatment. In the first series of experiments, the concentrations of NP1EO and NP2EO were already rather low in untreated sludge and represented the relative NPE content with 11% and 8% respectively. This was why the reduction of NP1EO and NP2EO by ozonization was at the sensitivity limit of the applied analysis method. However, in the second series of experiments the distribution of NPE compounds was 41% (4-NP-techn.), 35% (NP1EO) and 24% (NP2EO). Under these circumstances the respective degradation rates were as follows: 48-88% (4-NP-techn.); 11-28% (NP1EO) and 43-72% (NP2EO). With these results it was proven that ozonization is an efficient method of reducing NPE in wastewater sludge, which cannot be achieved by biodegradation.

As far as the NPE content in sludge is concerned, the experiments conducted revealed that ultrasonic treatment did not cause a systematic reduction of these compounds. Even at the highest energy input through the sonotrodes, reduction rates of less than 10% were observed. As opposed to ozonization, the effects were generally poor and no individual NPE compound was more affected than the others. Moreover, it was clearly demonstrated that the elimination rate was improved at increasing ozone doses, which was not the case with ultrasound.

3.2. Polycyclic Aromatic Hydrocarbons, PAH

According to Benabdallah El-Hadj et al. (2007) an important family of refractory compounds that can be affected by sonochemical reactions is that formed by the polycyclic aromatic hydrocarbons (PAH). Therefore much attention was allotted to the comparison of both disintegration methods for that compound group.

PAH are transported to wastewater treatment plants by effluent discharge and runoff waters. As they possess low water solubility and are highly lipophilic, they adsorb and accumulate in sludge throughout wastewater treatment (Wild et al., 1990). The degradation of PAH by anaerobic digestion has been already investigated in aqueous media, soils or sediments (Bamforth and Singleton, 2005), but not much work has been done on the PAH adsorbed in sludge.

Bernal-Martinez (2007) has reported that the ozonation pre-treatment of sludge at a transferred ozone dose of 0.1 g O_3/g DS allows an increase in PAH biodegradability or bioaccessibility, which leads to the enhancement of PAH removal up to a level of 68% during anaerobic digestion. PAH removal during the anaerobic digestion of ozonated sludge was classified according to the solubility of PAH in water: the higher the solubility, the higher the removal.

As far as PAH is concerned, after ozonization a reduction rate of 18-46% was observed in the first series of experiments (see Table 1) and one of 40-60% in the second series of experiments (see Table 1). This result is also in line with the aforementioned literature data.

The elimination rate was constantly enhanced by increasing ozone transfer levels. In fact it was observed that the PAH content (10.8 mg/kg DS) in untreated sludge was markedly lower than that in the second series of experiments, which is why it was possible to comply with the recommended value defined in the Draft European Guideline “Sludges” (2000) in this case (see Figure 2). However, in the second series of experiments, the PAH concentration was 16.1 mg/kg DS, which was so high that even the highest applied ozone dose was insufficient to reduce the PAH content in the sludge to below the draft EU standard value (see Figure 3).

In the second series of experiments, individual PAH compound reduction ranged from 24-81%. The substance groups in which systematic degradation due to ozonization was observed were acenaphtene, fluoranthene, and benzo(ghi)perylene, whereas pyrene (4 aromatic rings) and benzo(a)pyrene (5 aromatic rings) were substantially reduced by 73% and 84% respectively.

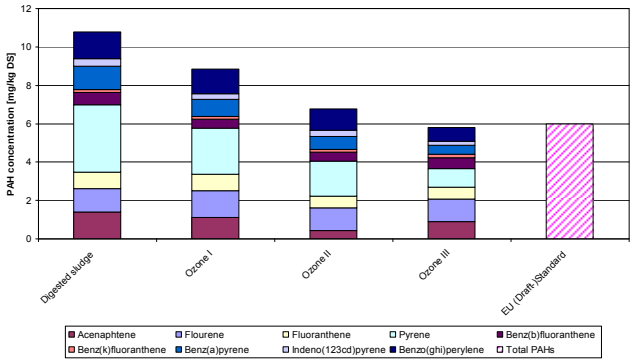

Figure 2 Concentration of PAH in digested sludge and sludge disintegrated by ozone - first series of experiments

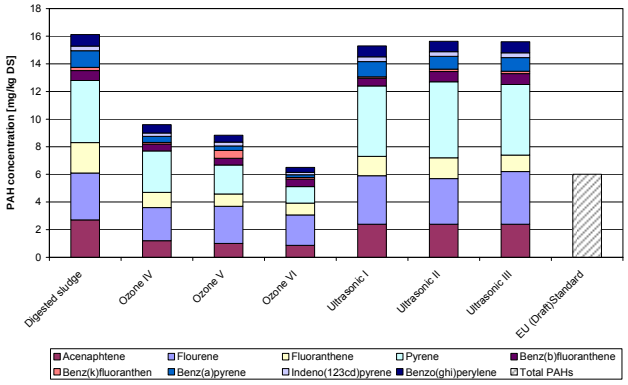

Figure 3 Concentration of PAH in digested sludge and sludge disintegrated by ozone and ultrasonic treatment – second series of experiments

It was noted that at 25°C, the water solubility of benzo(a)pyrene is lower than that of fluoranthene (Young and Cerniglia, 1995), but that degradation occurred to a markedly greater extent. Accordingly, in this case, the observation that higher water solubility is linked to higher removal rates (Bernal-Martinez, 2007) was not confirmed.

Benabdallah El-Hadj et al. (2007) have reported that ultrasonic pretreatment has caused a diffusion of polycyclic aromatic hydrocarbons (PAH) compounds to the aqueous phase and thus a reduction in the content of micropollutants in pretreated sludge. However, naphthalene was removed more efficiently with sonication than without it, particularly during mesophilic digestion. Conversely, pyrene removal remained at same efficiency level both with and without ultrasonic pretreatment.

For the nine PAH compounds analyzed in sludge samples as part of this work, the ultrasonic sludge treatment had no evidently substantial effect (see Table 1 and Figure 3). According to the analysis data, an elimination rate of 3-5% was observed.

Table 1 Reduction of NPE and PAH due to disintegration with ozone and ultrasound

Experiments	Specific Ozone Dose	Specific Energy Input	NPE Reduction	PAH Reduction
	[g O_3/g DS]	[kWh/kg DS]	[%]	[%]
1st Series of Experiments				
Test-1 (OZ I)	0.021	0.32	37.2	17.9
Test -2 (OZ II)	0.032	0.48	63.6	37.2
Test -3 (OZ III)	0.046	0.69	70.5	46.3
2nd Series of Experiments				
Test -4 (OZ IV)	0.046	0.70	33.5	40.4
Test -5 (OZ V)	0.063	0.94	46.1	45.2
Test -6 (OZ VI)	0.081	1.21	62.7	59.6
Test -7 (US I)		0.07	8.5	5.1
Test -8 (US II)		0.15	10.9	3.0
Test -9 (US III)		0.30	9.8	3.2

3.3 Linear Alkylbenzene Sulfonates (LAS) and Di-ethylexyl phthalate (DEHP)

The LAS in wastewater sludge is the xenobiotic compound most affected by the applied sludge stabilization technology. As compared to anaerobically digested or non-stabilized sludge, the LAS concentration in aerobically stabilized sludge is substantially reduced (Pinnekamp and Friedrich, 2006).

As far as the LAS content is concerned, it was noted that the concentration level in the investigated sludge was in the range of the EU draft standard. However, in the course of the experiments conducted, no substantial reduction was observed either after ozonization (max. 22% removal), or after ultrasonic sludge disintegration (max. 8% removal).

Conversely, households discharge di-ethylhexyl phthalate (DEHP) into wastewater diffusely, as it is used as a plastisizer in PVC. Hence the number of connected households contributes directly to the increase of the DEHP content in the wastewater. In addition, it is accumulated in anaerobically digested sludge (Pinnekamp and Friedrich, 2006).

In the course of the disintegration experiments conducted, it was found that the ozonization and ultrasonic treatment of digested sludge had no effect on the reduction of the DEHP content in sludge. However, it was noted that the DEHP concentration in the investigated sludge samples was far below the draft standard and thus not critical to the current discussion concerning the EU standards. Moreover, it was observed that in general, the DEHP accumulation in sludge is slowly decreasing, since the replacement of DEHP in PVC production by phthalates with higher substitution levels (UMK-AG 2000) and other plasticizers.

Similar results for the limited removal of LAS and DEHP were also observed in the course of ozonization experiments at Schermbeck WWTP in Germany (Braunisch et al. 2005).

4. Conclusions

As expected, aromatic compounds can be degraded by ozone treatment. The degradation rate increases with the amount of ozone employed. This is not the case with ultrasonic treatment. The ozonization elimination rates for NPE ranged from 33.5 - 70.5% depending upon the applied ozone dose. The reduction of NPE due to ultrasonic treatment was only 8 - 10% and was not positively affected by increasing the energy inputs. The reduction rate of PAH was 18 - 81% due to ozonization. Increases in the ozone dosing levels led to an almost linear enhancement of the elimination rate. Conversely, ultrasonic sludge treatment had no obviously significant effects on the PAH content in the sludge. According to the analytical data, an elimination rate of 3 - 5% was observed at 0.3 kWh/kg DS. At a comparable specific energy input, the reduction rate for PAH after ozone treatment was calculated to be at least 300% higher. Therefore, it was concluded that the ozonization of digested sludge provides a positive side-effect (in addition to enhanced VSS reduction and increased biogas production) with regard to sludge disintegration, as it reduces micro-pollutants such as NPE and PAH. This is of ecological relevance, especially if the sludge is to be reused in agriculture and landscaping applications.

Sewage sludge application in agriculture is only one source of organic contamination in soils, water or plants. Consequently, environmentally sound decisions need to be based on an integrative evaluation of contaminant sources and transfer pathways. The idea of nutrient recycling gains fresh importance when development is seen in the light of sustainability. There is general agreement however, that the recycling of nutrients by means of sludge application in agriculture must not lead to adverse effects on the quality of products or the environment and therefore sludge contamination has to be prevented (UMEG, 2001).

If adopted, the standards proposed in the Draft Document (EU, 2000) for PAH, DEHP, NPE and LAS would have very serious implications for the use of sewage sludge in agriculture in Europe. Jones and Northcott (2000) expect that most sewage sludges in Europe, including those originating from rural and domestic wastewater treatment plants, are likely to exceed the proposed limits for PAH and LAS. They also point out that trace organic contaminant analyses require sophisticated analytical instrumentation and specialized analysts, which can be very expensive.

Chaney et al. (1998) conclude that biosolids can be beneficially used in sustainable agriculture at such low agricultural and environmental risk, that utilization on farmland should be the preferred method of "ultimate disposal". The pretreatment of industrial and nonindustrial sources of some contaminants may be required in order to achieve the required biosolid quality, but technology is already available for the pretreatment needed. In addition, new sludge treatment technologies may have to be applied should sludge production increase as predicted (Anderson, 2001).

In this context it can be stated that the ozonization of digested sludge would represent an efficient technical solution for the reduction of the NPE and PAH content in biosolids prior to their application to farmland. Conversely, disintegration with ultrasound cannot fulfil this requirement.

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