Technology for Water
ENVIROCHEMIE

White Paper

Removal of trace substances from industrial wastewater by means of advanced oxidation processes (AOPs)

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2. Introduction

Trace substances such as those used as active ingredients in pharmaceutical products, which are difficult to break down using biological means, are increasingly being found in wastewater. The application of advanced oxidation processes (AOPs) is a potential method of pre-treating wastewater concentrates, with the objective of converting toxic materials into non-toxic ones and substances that are difficult to break down into biologically recyclable ones. The oxidation reaction with hydroxyl (OH) radicals used to achieve this is normally effective very quickly. This paper explores the formation of OH radicals and the removal of trace substances by means of ozone, UV/H_2O_2 and the Fenton and photo-Fenton reaction in more detail and outlines some examples of application in practice.

3. Removal of trace substances by means of advanced oxidation processes (AOPs)

Highly active organic substances, such as the active ingredients in drugs, biocides, pesticides and their metabolites, are increasingly being found in wastewater and are the focus of considerable public debate. Production processes in the pharmaceutical and chemical industries generate rinsing water and wastewater, which can contain these and other key substances at various concentrations. Because these substances are difficult to biodegrade and thus cannot be eliminated in a biological treatment process, this generally makes treating wastewater considerably more difficult. The wastewater often has to be disposed of in an external thermal process such as incineration, which entails heavy costs for businesses. Physical treatment options such as membrane technology or activated carbon, which are used to separate out or adsorb residues, are time-consuming and expensive. These methods leave behind substances that have to be removed, which entails further costs.

A more cost-effective alternative to these solutions is pre-treatment of wastewater concentrates by means of advanced oxidation processes (AOPs). These include oxidation methods in which highly reactive compounds such as OH radicals are formed. Oxidation of these compounds, which are difficult to biodegrade, causes a breakdown of the highly active substances, leaving behind intermediate products that are generally easy to biodegrade. The pre-treated wastewater can then be indirectly introduced to the sewer system, or even fed into a facility’s own biological treatment stage. Some typical industries and applications of AOPs in industrial wastewater treatment are listed in Table 1:

Table 1: Typical industries and applications of AOPs in industrial wastewater treatment

Typical industries	Typical applications
Chemical industry Pharmaceutical industry Biotechnology Steel, metal and surface treatment Life sciences Textiles industry Paper industry	Eliminating: • toxic substances (e.g. pesticides) • persistent substances • active pharmaceutical ingredients (APIs) • complexing agents • inorganic components (e.g. nitrite, cyanide, thiosulphate or hydrazine) Improved biodegradability (BOD_5/COD ratio) Reduction of COD or TOC concentration Decolouration Sterilisation Odour neutralisation

3.1. Basics

The objective of these various oxidation methods is to convert toxic materials into non-toxic ones and substances that are difficult to break down into biologically recyclable ones. After this chemical treatment, the wastewater can be fed into a subsequent biological treatment stage. Complete oxidation of the substances, generating carbon dioxide, water and sometimes salts, is in many cases not economical owing to the high consumption of oxidising agents and energy it would require. For this reason, the oxidative pre-treatment is intended to carry out oxidation of the wastewater only to the extent that further biological treatment methods are viable, presenting a clear advantage from an economic viewpoint. Table 2 shows the typical changes to the wastewater parameters DOC (dissolved organic carbon), COD (chemical oxygen demand) and BOD_5 (5 day biochemical oxygen demand) for complete and partial oxidation. There are two types of partial oxidation processes: one involving oxidation of organic substances (molecular scission) and another in which inorganic compounds such as Fe^{2+} and Mn^{2+} are subject to oxidation at higher oxidation levels. Biodegradability can be improved in particular by reducing the COD/BOD_5 ratio or increasing the BOD_5 concentration, as is the case with partial oxidation by molecular scission.

Table 2: Change in wastewater parameters after oxidation (⇔ unchanged, ⇑ increasing, ⇓ decreasing) (source: Kreuzinger, N., Schaar, H. (2011), adapted)

Reaction		DOC	COD	BOD_5	COD/BOD_5
Partial oxidation	Increase of oxidation number	⇔	⇓	⇔	⇓
	Molecular scission	⇔	⇔	⇑	
Complete oxidation	Separation of CO_2	⇓	⇓	⇓⇔	⇔

Table 3 lists the standard electrode potential for some oxidising agents with respect to the normal hydrogen electrode in an aqueous medium. The higher the potential of a substance, the greater its oxidising effect. The agent in Table 3 with the highest oxidation power is the OH radical. When compared to ozone or hydrogen peroxide, OH radicals generally react faster by a factor of 10^6 to 10^9 (between a million and a billion times faster).

Table 3: Standard electrode potential of selected oxidising agents

Oxidising agent	Standard electrode potential E^0 [V] (pH value = 0)
Hydroxyl (OH) radical	+2.80
Oxygen (atomic)	+2.42
Ozone	+2.07
Hydrogen peroxide	+1.77
Hypochlorite	+1.49

Enhancing the formation of OH radicals by applying specific parameters such as ultraviolet (UV) radiation is known as an advanced oxidation process (AOP). The following section will explore the formation of OH radicals by means of ozone, UV/H_2O_2 and the Fenton and photo-Fenton reaction in more detail.

4. Ozone

4.1. General

Ozone (O_3) is a very powerful oxidising agent, which is a gas under normal conditions. It has already long been in use as a disinfectant in water installations such as drinking water facilities and for the treatment of water in swimming pools. In physical applications, the water-solubility and stability of ozone is determined by a range of factors, including pressure, water temperature, ozone concentration and pH value (see Table 4).

Table 4: Factors affecting the water-solubility and stability of ozone

Pressure	Higher pressure = greater water-solubility of ozone
Water temperature	Lower temperature = greater water-solubility of ozone
Ozone concentration	Higher ozone concentration = greater water-solubility of ozone
pH value	Lower pH value = acidic environment = higher stability of ozone (mostly direct ozone reaction) Higher pH value = alkaline environment = reduced stability of ozone (mostly formation of hydroxyl radicals)

4.2. Direct/indirect ozone reaction

Ozone can react with substances in two different ways:

- Directly
- Indirectly, through the formation of hydroxyl radicals (OH•).

Ozone’s reaction pathways and effects are therefore highly complex.

A direct ozone reaction prevails in the acidic to neutral pH range and is selective, i.e. has different decomposition rates depending on the other reactants. This reaction tends to attack saturated double or triple bonds, cyclical aromatic compounds or amino groups. Ozone therefore causes partial oxidation, which reduces the size of the molecule and thus forms intermediate products. Using this method, the biodegradability of the substances is in many cases improved. However, care must be taken with regard to any toxicological effects of the transfer products formed.

On the other hand, in alkaline environments, the indirect ozone reaction via OH radicals prevails. These short-lived OH radicals react with almost all types of bonds and are thus not selective in their effects. The advantage is that this also oxidises compounds that are difficult for ozone to break down. The drawback is that the OH radicals also react with scavenger substances. This “uses up” the radicals and removes them from the reaction. Scavengers can include humic substances, water hardness (HCO_3^-/CO_3^{2-}) and even orthophosphates.

4.3. Reaction rates

Table 5 shows the reaction rates with ozone and OH radicals for specific trace substances. As a basic principle, the reaction rate of OH radicals with substance-dependent reaction constants is significantly greater than that of ozone, at between 10^8 and 10^{10} $M^{-1}s^{-1}$. The most important factor here is the chemical structure of the substance being treated. Generally speaking, ozone reacts with alkanes, alcohols and ketones at a very slow rate. Ozone also reacts with aromatic compounds like benzene significantly more slowly than OH radicals do.

Table 5: Reaction rates of specific substances with ozone and hydroxyl radicals (source: Gunten (2003); Abegglen et al. (2009), adapted)

Ligands, disinfection by-products	Solvents, fuel additives	Inorganic substances	Pharmaceuticals, pesticides, medical applications	Reaction with ozone k_{O3} ($M^{-1}s^{-1}$)	Reaction with OH radicals K_{OH} ($M^{-1}s^{-1}$)
NTA^{3-} CaEDTA^{2-} HEDTA^{3-}	Chloroethene	Nitrite (NO_2^-) Cyanide (CN^-) H_2S S^{2-}	Bisphenol A Carbamazepine Clarithromycin Diclofenac Dinoseb Estrone Propranolol Sulfamethoxazole Trimethoprim	Fast ($k_{O3} \geq 10^4$)	Very fast ($k_{OH} \geq 10^9$)
EDTA^{4-}		Iron (Fe$^{2+)}$		Fast ($k_{O3} \geq 10^4$)	Fast ($10^8 \leq k_{OH} < 10^9$)
Zn (HDTPA^{2+} / H_2DTPA)	Cis-1,2-dichloroethene	Bromide (Br-)	Atenolol Benzotriazole Bezafibrate Carbofuran Methoxychlor Mecoprop Sotalol	Moderately fast ($10^2 \leq k_{O3} < 10^4$)	Very fast ($k_{OH} \geq 10^9$)
Fe(III)EDTA				Moderately fast ($10^2 \leq k_{O3} < 10^4$)	Fast ($10^8 \leq k_{OH} < 10^9$)
		Manganese (Mn$^{2+)}$		Moderately fast ($10^2 \leq k_{O3} < 10^4$)	Moderately fast ($10^7 \leq k_{OH} < 10^8$)
Fe(III)(DTPA^{2+}/ HDTPA) Iodoform	Benzene Chlorobenzene *p*-Dichlorobenzene Ethanol MTBE Toluene Trichloroethylene Tetrachloroethylene o-Xylene	Dihydrogen arsenite (H_2AsO3^-)	Atrazine Alachlor Endrin Diuron Ibuprofen Iopromide	Slow ($k_{O3} < 10^2$)	Very fast ($k_{OH} \geq 10^9$)
Bromoform	t-BuOH			Slow ($k_{O3} < 10^2$)	Fast ($10^8 \leq k_{OH} < 10^9$)
Chloroform Trichloracetate		Ammonium (NH3/NH4+)		Slow ($k_{O3} < 10^2$)	Moderately fast ($10^7 \leq k_{OH} < 10^8$)

4.4. Technical application

Because of its high level of reactivity, ozone is highly unstable, which is why it is synthesised directly at its location of use. Unlike other oxidation methods, this eliminates the need for transportation and alleviates concerns over the on-site availability of chemicals. In industrial settings, ozone is produced using electrical energy and air or liquid oxygen. The amount of ozone in the reaction mixture is between 20 and 50 g/m³ when using air, and between 100 and 200 g/m³ when using oxygen. Because of the hazardous nature of ozone, appropriate safety measures must be in place. For example, exhaust fumes from the reaction tanks must be treated before being let out into the environment. This is achieved using residual ozone destructors, which operate using thermal/catalytic or purely thermal methods. Moreover, both the exhaust fumes and indoor air must be continuously monitored, and operating personnel must receive appropriate training.

The investment costs associated with an ozone oxidation system are comparatively high, and ozone production itself requires a relatively high amount of energy. For example, if liquid oxygen is used to generate the ozone, around 8 to 10 kWh of energy is required for each kilogram of O_3 created. And because there are losses in the form of heat, the ozone generator must be cooled.

5. UV / H_2O_2

5.1. General

Hydrogen peroxide is a relatively cheap oxidising agent, which is available commercially as an aqueous solution at various concentrations. The oxidation potential of H_2O_2 alone is rarely enough to break down trace organic substances, so it requires activation. This photolytic activation is achieved using a special UV lamp. Application of energy in this way causes the dissociation (photolysis) of the hydrogen peroxide molecule, which leads to the formation of OH radicals:

$$H_2O_2 \overset{UV}{\rightarrow} 2\,HO\cdot \qquad \textbf{(1)}$$

The oxidation potential of the highly reactive OH radicals is significantly higher than that of hydrogen peroxide and ozone (cf. Table 3).

H_2O_2 concentration, pH value, temperature, scavenger concentration, self-absorption of wastewater and the reaction time are all factors that significantly influence the effectiveness of UV/H_2O_2 oxidation (see Table 6).

Table 6: Factors influencing UV/H_2O_2 oxidation

H_2O_2 concentration	Excessively high H_2O_2 concentration is not practical (see Section “H_2O_2 concentration”) The optimum H_2O_2 concentration should be adapted to every oxidation process
pH value	Oxidation most effective when in an acidic environment
Temperature	Higher temperatures are more favourable for the chemical decomposition process
Scavenger concentration	E.g. the concentration of humic substances, orthophosphate, water hardness (HCO_3^-/CO_3^{2-})
Self-absorption of wastewater	High absorption or low transmission of wastewater makes the process more difficult
Reaction time	Optimum reaction time to be determined experimentally

5.2. H_2O_2 concentration

In technical applications, the high solubility of hydrogen peroxide in water, as well as its ease of dispensing, is a significant advantage. An excessive concentration of H_2O_2, however, is highly impractical. Even though it improves the formation of radicals, it can diminish the oxidation effect of the process because of scavenger reactions. These reactions include the recombination of OH radicals and the formation of hydroperoxyl radicals ($HO_2\bullet$), which are weaker oxidising agents than OH radicals:

$$2\,HO\cdot \;\rightarrow\; H_2O_2 \qquad (2)$$

$$H_2O_2 + HO\cdot \;\rightarrow\; H_2O + HO_2\cdot \qquad (3)$$

The optimum H_2O_2 concentration should be adapted to every oxidation process in order to reduce the associated scavenger reactions of the H_2O_2.

5.3. UV lamp, self-absorption in wastewater

In order to permit photolysis of H_2O_2, a UV spectrum lower than 280 nm is required. Wastewater oxidation normally involves the use of medium-pressure UV lamps that emit radiation at a wavelength (λ) of between 200 and 400 nm.

One factor that needs to be considered is whether the wastewater has high self-absorption properties in the λ = 200–280 nm range (known as the “inner filter” effect), since this can significantly reduce the effectiveness of the UV/H_2O_2 method. This effect can be caused by nitrate and nitrite ions, which can also be formed during the oxidation of organic molecules containing nitrogen. Low transmission properties of the wastewater is inherently a hindrance to treatment with UV radiation because the UV rays are not able to penetrate deep enough into the solution. In such cases, a high amount of turbulence above the UV lamp can boost the chemical decomposition rate.

5.4. Technical application

Hydrogen peroxide is normally used in the form of an aqueous solution with no more than 35% concentration of H_2O_2 by weight. The chemical can be handled easily and safely when the usual precautions are taken for corrosive substances. It can generally also be stored in clean delivery containers for months without any significant loss of effective substance. Because of the non-selective oxidative effect of the OH radicals, the technology has a wide scope of application (cf. Table 5). This is particularly advantageous for applications involving varying wastewater compositions. It is also relatively easy to upgrade a system (e.g. when the volume of wastewater exceeds the system’s design capacity) by taking the following actions:

- Increasing the amount of hydrogen peroxide being dispensed
- Installing additional UV lamps
- Introducing a reaction accelerant such as titanium dioxide or iron → the advantages of the photo-Fenton process can be used (see Section 7)

This allows a facility to remain in operation even under varying conditions with only minor technical modifications. This flexibility is an advantage when compared to oxidation using ozone, where the ozone generator is generally only designed for a specific capacity.

At low wastewater volumes (up to 50 m³ per day) a batch treatment system can be implemented, while at higher volumes the treatment is continuous. The operation of UV lamps introduces heat energy, which means that a cooling system is generally necessary for batch-wise treatment of wastewater. In order to extend the service life of UV lamps, frequent switching of the lamps on and off should be avoided.

UV irradiation is associated with a relatively high energy demand, which is heavily dependent on the wastewater composition and the desired level of purification.

6. Fenton reaction (H_2O_2/Fe^{2+})

In aqueous solution, the Fenton reaction causes the formation of OH radicals. It is an oxidation method involving an iron salt catalyst and hydrogen peroxide and can be represented in this simplified equation:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO \cdot + OH^- \qquad \textbf{(4)}$$

The iron salt used is generally iron(II) sulfate ($FeSO_4$). The method is widespread in the field of wastewater treatment, has been validated by technical evaluations, and is easy to control (simple equipment and facility operation). In addition, coloured or cloudy wastewater is not a problem because the reaction is not induced by photolysis. Thus the Fenton reaction does not require any energy input, making it less energy-intensive as a whole.

In the Fenton process, oxidation and flocculation occur simultaneously, removing organic substances and materials without creating toxic decomposition products. Non-degradable organic components can be removed and toxicity can be reduced by reaction via radicals.

The optimum pH value for the Fenton process is between 2 and 4, so in many cases the pH value of the wastewater needs to be reduced.

There are, however, some drawbacks to be considered, including the production of high volumes of sludge (→ high removal costs), a high consumption of acids in order to lower the pH value, and high concentrations of chlorides and sulfate ions (depending on the iron salt used).

7. Photo-Fenton reaction ($H_2O_2/Fe^{2+}/UV$)

The photo-Fenton reaction involves the formation of OH radicals according to the Fenton process, as described in equation (4). The application of UV radiation causes a regeneration of Fe^{2+} (see equation (5)), which can reduce the used quantity of Fe^{2+}.

$$Fe^{3+} + H_2O \overset{UV}{\rightarrow} Fe^{2+} + HO \cdot + H^+ \qquad \textbf{(5)}$$

The use of UV radiation accelerates the Fenton process, leading to higher reaction rates.

The primary advantages of the photo-Fenton method when compared to the conventional Fenton process are as follows:

- Considerably lower consumption of chemicals

- Higher reaction rates
    - Enhanced chemical decomposition effect
    - Improved space-time yield

  → Good chemical yield with high decomposition effect
- Reduced sludge production

8. Examples in practice

The following sections outline some practical examples from the pharmaceutical and chemical industries in which wastewater is treated with various oxidation methods.

8.1. Pharmaceutical industry (UV/H_2O_2)

At a German pharmaceutical company, a modular wastewater treatment facility was installed in order to treat the water left over from the tablet production process. This wastewater contains various active pharmaceutical ingredients (APIs). To ensure adequate treatment, it was important to choose a method that (a) could handle the wide range of active ingredients found in the wastewater, (b) could remove the ingredients from the water without leaving residues, (c) was easily scalable, and (d) did not require elaborate safety measures. Based on these criteria, the company decided to adopt a method involving oxidation via UV/H_2O_2. If it was not treated, the costs of disposing of the wastewater would have been very high. But with a wastewater treatment system in place involving oxidation via UV/H_2O_2, it became possible to return the wastewater indirectly to the sewer system.

The volume of wastewater processed each day is up to 25 m^3, with the API concentration fluctuating between 2000 and 10,000 µg/l. The objective of the treatment process was to produce wastewater whose concentration was below 10 µg of API per litre. For this purpose, the daily wastewater volume is collected in a buffer tank with the addition of H_2O_2 and then exposed to UV-light radiation in a batch treatment process in a complete cycle lasting 24 hours. The treated wastewater is then mixed into the remaining wastewater and fed into the sewer system.

The exact amount of hydrogen peroxide and UV radiation required was calculated and tested in a laboratory beforehand. Even unusually high concentrations of APIs, constituting the worst-case scenario, was taken into account in testing.

Determining the concentration of pharmaceutical residues in wastewater with precision is a non-trivial undertaking because each active ingredient calls for different measurement methods. For day-to-day operation of the treatment facility, substance-specific determination processes would be too complex and expensive. As a result, the sum parameters TOC (total organic carbon) and COD (chemical oxygen demand) would have to suffice. As part of a correlation study, the decomposition of active pharmaceutical ingredients, with a reduction in COD and TOC, was explored in order to apply a quick and reliable analysis method in the day-to-day operation of the facility. This method can be used to determine with high precision that the active ingredients in the water, at a specific COD, have been removed by up to 99.9% (see Figure 1).

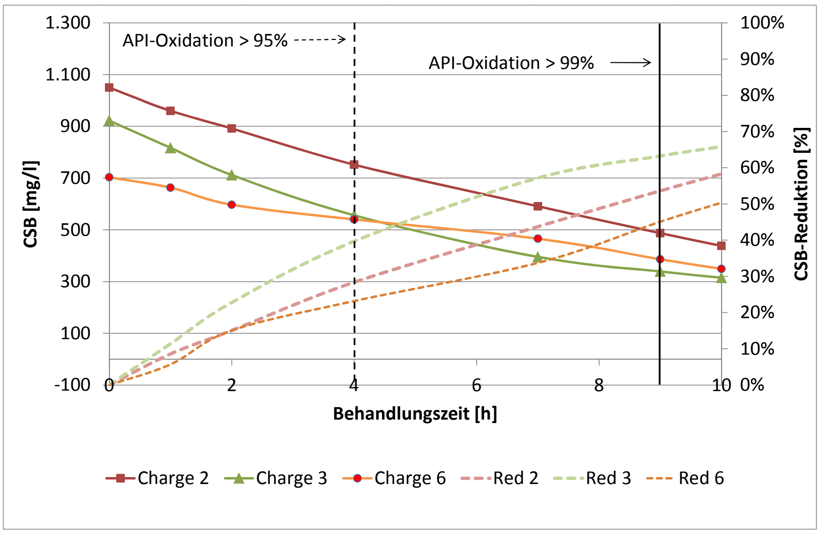

Figure 1: API oxidation correlated with a reduction in COD

Thanks to its modular construction, the treatment facility can be installed separately in front of the production building and is also easy to upgrade. Plans are currently in place to extend the system so it can handle a throughput of 50 m³ per day. Its modular construction also meant that the pharmaceutical company did not have to build a new structure, saving it the trouble of obtaining planning permission and fire safety certification, for example.

Figure 2: Modular facility construction with space for upgrades

8.2. Pharmaceutical industry (ozone)

At a large pharmaceutical company, a production facility was built at their plant in Mexico in order to formulate highly effective drugs for the treatment of cancer. Analyses showed that the two highly active pharmaceutical ingredients (APIs) used could have an adverse ecotoxicological impact and should therefore, as a precaution, not be introduced into wastewater. During production, a daily wastewater volume of 10 m³ is generated, which has to be treated. A series of preliminary laboratory tests were conducted on the UV/H_2O_2 method, ozone oxidation and adsorption using activated carbon, taking into account ecological and economic considerations.

The results of the laboratory testing showed that the two active ingredients can basically be removed from the wastewater with all three methods. Alongside reliable removal of active ingredients, the pharmaceutical company also specified a number of criteria, including the following:

- High degree of automation
- Compact overall construction
- As little handling of chemicals as possible
- Generation of as little waste as possible

In this specific case, ozone treatment was found to be the best method at meeting these criteria. Figure 3 shows an example of oxidation of one of the two APIs using ozone, for which a sample with an active ingredient concentration of 200 mg/l was produced. The oxidation process was completed after 20 minutes and required a total of 18.33 g ozone.

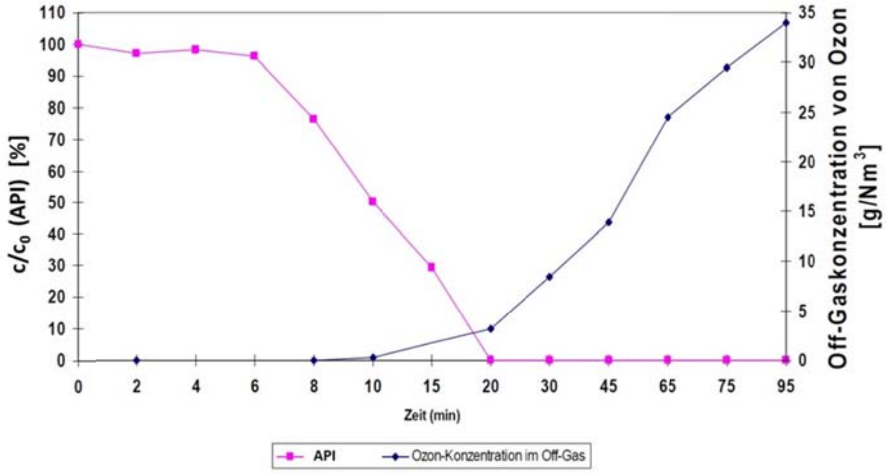

Figure 3: Ozone treatment of an active pharmaceutical ingredient (API) in a laboratory test

The obtained data was used as a basis for the development of a customised wastewater treatment facility that meets all requirements. The system is housed completely in a six metre long container and is designed to run fully automatically 24 hours a day so that the wastewater can be treated in a continuous process. The aim was to achieve a 99.9% reduction in the API concentration.

The ozone is synthesised by means of an ozone generator that uses the surrounding air. The design concept ensures ozone is mixed with the wastewater under slight overpressure by means of an injection system. Because the ozone requirement depends on the chemical properties and the concentration of drugs in the wastewater, the system is fitted with sensors for measuring the dissolved ozone concentration after treatment. As a result, the facility can automatically regulate the output of the ozone generator so that a defined ozone concentration (e.g. 2 mg/l) can always be maintained.

In order to eliminate the residual ozone, the treated water passes through an activated carbon filter. In order to ensure adequate safety, the container is fitted with an ozone detector, which triggers an alarm when ozone is detected in the indoor air.

8.3. Fine chemicals production (UV/H_2O_2)

At a German fine chemicals company, the production of special organic and inorganic chemicals generates wastewater that is highly polluted, highly acidic and contains a high amount of phenolic compounds (pH < 1, COD > 25,000 mg/l). This toxic wastewater, produced at volumes of 5 m³ per day, was previously disposed of externally. A laboratory study on the UV/H_2O_2 method showed that, in addition to oxidation of the COD, the bioavailability could be increased to such an extent that the wastewater can be fed into the facility's own wastewater treatment system ready for further biological treatment (see Figure 4).

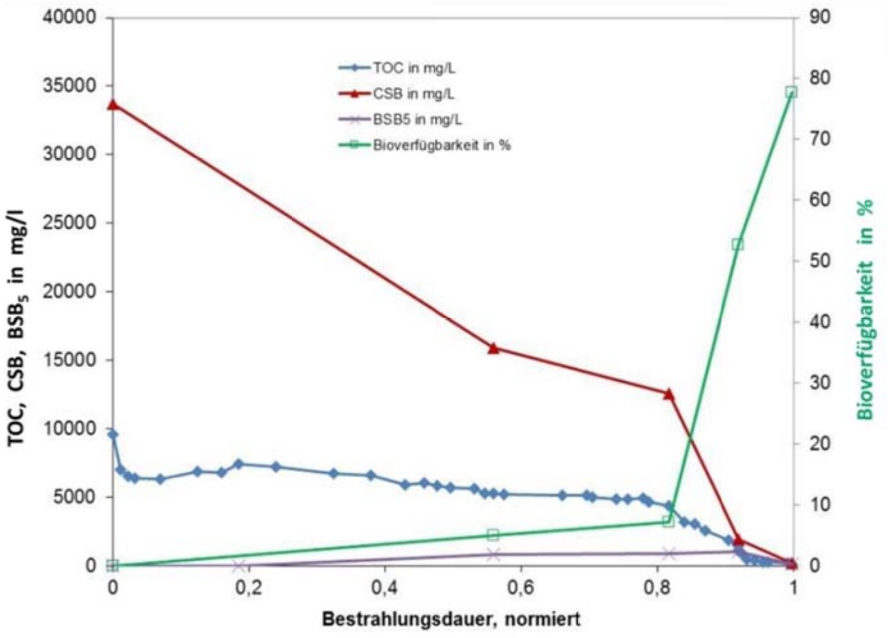

Figure 4: Increase in bioavailability with UV/H_2O_2 oxidation in wastewater from a fine chemicals production facility

Figure 4 shows the decomposition of organic active ingredients using the parameters TOC (total organic carbon) and COD. The chart also shows the change in concentration of BOD_5 and the resulting bioavailability. The first data curves for decomposition over time were typical, reflecting the oxidation of organically bound carbon. But as the exposure time increased, so too did the amount of intermediate product that is oxidised into carbon dioxide and then mineralised. Once decomposition had all but stagnated, the oxidation conditions were adjusted until the decomposition process was accelerated significantly. Subsequent BOD_5 measurements confirmed that bioavailability had been increased to approximately 80%.

8.4. Fine chemicals production (photo-Fenton)

At a German fine chemicals company, the production of special chemicals results in highly polluted and acidic wastewater with a pH value below 1, which is jet black in appearance and has a COD greater than 100,000 mg/l (Figure 5, left sample). The wastewater contains carcinogens and is generated at a volume of 3 m³ every day, which previously had to be disposed of externally. In a laboratory study, a Zahn-Wellens test was conducted in order to demonstrate that the photo-Fenton oxidation process could increase the bioavailability of active ingredients in wastewater to such an extent that the wastewater can then be sent for further biological treatment following neutralisation in the facility's own wastewater treatment system. It was possible to demonstrate the decomposition of organic active ingredients with analytical methods and optically by decolouration of the treated wastewater. On the basis of these results, plans are currently being drawn up for a large-scale technical solution for treating the wastewater.

Figure 5: Decolouration of wastewater during treatment

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