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INTEGRATED PROCESSES OF CHEMICAL AND BIOLOGICAL OXIDATION OF WASTEWATERS

Biological treatment of wastewater has been known for almost hundred years; however, in the case of industrial wastewaters this method is not sufficient because they carry high content of xenobiotics along. The efficiency of biodegradation may significantly be increased by chemical oxidation. Recently, the so-called advanced oxidation processes (AOPs) combined with biological treatment have appeared to be successful in large-scale systems, therefore it is reasonable to present the fundamentals of the application of combined methods in wastewater treatment. The general statements about the integration of biodegradation and chemical oxidation are exemplified by case studies of textile wastewater biodegradation combined with AOPs, which have been the subject of the author's research for last years.

1. INTRODUCTION

Although as early as in ancient (in Athens and Jerusalem) people knew the methods for utilizing wastewaters as fertilizers, in the Middle Ages these methods were neglected and forgotten. As late as at the end of the 19th century, after discoveries of Pasteur, a fresh impetus is given to separating wastewater from water sources. It was then that the first wastewater treatment facilities were built. Wastewater was treated there by mechanical separation of suspensions and biological conversion of the pollutants dissolved. However, an increasing population density and rapid development of industry caused that natural autopurification of water appeared to be insufficient and the methods for mechanical and biological treatment of wastewater did not satisfy the requirements either. An example may be the replacement of soap with synthetic detergents. Industrial wastewater contains now more xenobiotics – substances which do not occur in nature, which are resistant to biodegradation and even toxic to living organisms. Xenobiotics are of anthropogenic origin, and their chemical structure is unrecognizable by existing degradative enzymes. They penetrate into environment in

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unnaturally high ceoncentrations and are accumulated. As examples of xenobiotics we can mention pesticides, insecticides, polychlorinated biphenyls (PCBs), polycyclic aromatic hydrocarbons (PAHs), some synthetic dyes and detergents as well as other constituents of textile wastewater.

This broad spectrum of pollutants, which are resistant to biodegradation or toxic to living organisms, induced the necessity of applying the so-called third stage of wastewater treatment in which mainly physicochemical methods are used. In the last two decades, a new approach to the applicability of biological and chemical oxidation methods has been used, however they should not be applied separately but as integrated processes. This refers also to the sequence of application.

Before giving some examples of the integrated wastewater treatment, the basis of biological and chemical oxidation of xenobiotics will be discussed.

2. BIODEGRADATION OF XENOBIOTICS

Biodegradation of xenobiotics may occur becasue natural enzymes are not specific enough, particularly if bounding and transport of substrates are concerned. These properties of enzymes cause that the biodegradation of xenobiotics is called a spontaneous, random degradation. If a microorganism breaks down xenobiotics in such a random way it can use the energy released and utilize carbon skeleton to grow; then biodegradation takes place as in the case of natural substrates. If bacteria are not able to degrade xenobiotics, these compounds can be biodegraded only in the presence of an additional, well assimilated carbon (like aliphatic hydrocarbons) and energy as is presented in figure 1. This biotransformation of xenobiotics in the presence of substrates, which are the source of carbon and energy, is called a cometabolism. Cometabolism occurs when one organism growing on a particular substrate also oxidises the second substrate, but it is unable to assimilate or use the latter as a source of carbon and energy. The products of oxidation of the second substrate are then utilized by other bacteria in the community. The experiments with biodegradation of some pesticides, whose degradation was greatly enhanced by the addition of ethanol or glucose [1], may serve as an example of cometabolism. The products of oxidation of the second substrate are then used by other bacteria in the mixed cultures as growth substances. For example, polychlorinated biphenyls are cometabolized to chlorobenzoic acids by biphenyl-utilizing bacteria [2]. Similarly, chlorinated alkenes are cometabolized by Xantobacter [3]. There are numerous examples of xenobiotic cometabolism, e.g., polyaromatic hydrocarbons (PAHs) are cometabolized by both aerobic mixed cultures and by enriched bacterial cultures [4].

It is worth mentioning that very often, due to oxidative biodegradation of xenobiotics, the intermediate products are more toxic than substrates, hence it seems that the term "biotransformation" is more correct than "detoxication".

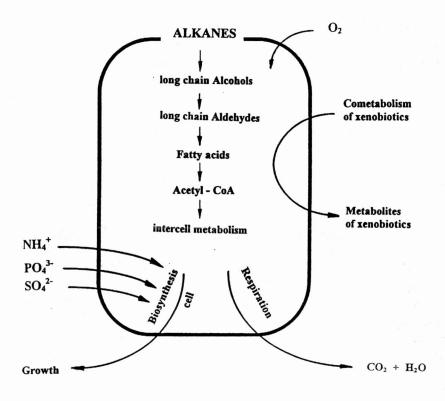


Fig. 1. Biodegradation of alkanes with cometabolism of xenobiotics

Biodegradation of xenobiotics may be controlled by modifying genetically microorganisms in such a way that they break down efficiently a broad range of pollutants without producing toxic intermediate products which is often a case. Many genes responsible for xenobiotic metabolism are present on plasmids, or less frequently on chromosomes. The role of plasmids in the spread of an antibiotic and metal resistance in microbial populations is well documented. It seems likely that plasmids play a similar role in the transfer of novel catabolic pathways. This can be illustrated by a genetically modified strain of *Pseudomonas putida* KT 2442::mer-73 with a minitransposon containing mer TPAB operon which is capable of lysing phenyl-mercury, reducing mercury ion and degrading the resultant benzene [5]. Possibilities of genetic engineering are enormous; however, applicability of recombinants in wastewater treatment has been limited so far to closed systems due to the lack of data on the behaviour of these superbacteria in the natural environment.

The action of most xenobiotics is completed when they are removed or deactivated due to the biotransformations being made by microorganisms with specialized enzymatic systems. Xenobiotic biodegradation is usually a two-step process which includes

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oxidation and coupling. Water-soluble hydrophilic compounds need not to be degraded because they may be removed in their original form, while hydrophobic substances are subjected to oxidation and become more polar and water soluble, thus gaining bioavail-ability. The first-step reactions take place in the presence of enzymes called monooxige-nases, or the enzymes cooperating with the cytochrome P-450. The cytochrome is a mixture of at least 10 isosymes of different reactivity. We often deal with an enzymatic induction when the xenobiotic enhances enzyme biosynthesis as is the case of an aromatic hydrocarbon hydroxylase (AHH) taking part in the conversion of inactive procarcinogenes, i.e. PAHs, inhaled by smokers. The AHH concentration in smokers is higher than in non-smoking people. Typical hydroxylation reactions

 $RH + O_2 + NADPH + H^+ \rightarrow ROH + H_2O + NADP$

catalyzed by monooxigenases consist in introducing one atom from oxygen molecule to RH substrate which gives ROH hydroxyl derivative, and the other atom of oxygen – into water molecule. For example, phenol compounds are hydroxylated by monooxygenases giving a molecule of pyrocatechol, an hydrogen donors are nucleotides (NADPH – nicotinamideadenine dinucleotide phosphate). The fission of an aromatic ring is often preceded by removal of substituents. Halogen, nitro or sulfo groups are substituted for hydroxyl groups. Dioxygenases cleave aromatic rings due to building-in molecular oxygen. The ring is broken either between two neighbouring hydroxyl groups (ortho-cleavage) or between hydroxylated and neighbouring nonhydroxylated carbon atom (meta-cleavage). Figure 2 illustrates the possible ways of cleavage of aromatic ring. Products of the two reaction types are included in the main metabolic pathways through pyruvate, acetaldehyde or oxalacetate.

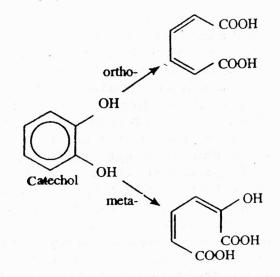


Fig. 2. Ortho- and meta-cleavage of aromatic ring

Abiotic factors, including pH, pO_2 , temperature, etc., have been shown to affect xenobiotic degradation. Because of a limited scope of this paper I wish to emphasize water-solubility which determines the so-called bioavailability of pollutants and thus their biodegradability. In order to improve this property of xenobiotics, detergents, and biosurfactants in particular, are used. They enhance significantly PAHs solubility [6].

Wastewater is also subjected to chemical oxidation which greatly improves biodegradability.

3. CHEMICAL OXIDATION

Advanced oxidation processes (AOP) take place with simultaneous action of several oxidizing agents such as ozone, hydrogen peroxide and UV irradation. As a result, very reactive agents are obtained in which a predominant role is played by hydroxyl radicals (OH[•]). Redox potentials of the main oxidizing agents decrease in the following sequence: OH[•] (2.80 V), O₃ (2.08 V), H₂O₂ (1.78 V), Cl₂ (1.36 V), O₂ (1.23 V).

Extensive investigations have been carried out worldwide on advanced oxidation because of a large potential of these methods. High efficiency and low selectivity are their basic advantages. As we deal mainly with radical reactions, these methods can be applied to the degradation of many organic compounds contained in aqueous solutions.

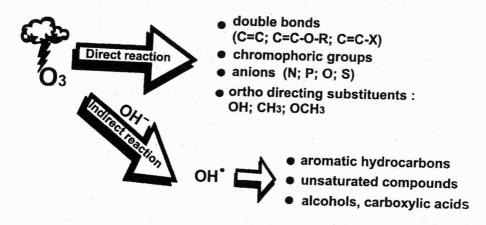


Fig. 3. Mechanism of ozonation

Ozone in aqueous solutions may react in two ways (figure 3), depending on compounds being oxidized:

directly; ozone molecule is linked to the oxidized substance and ozonides are formed – this is the so-called ozonolysis [7];

through indirect chain reactions; as a result of ozone molecule disintegration, atomic oxygen and OH[•] radical are formed, both having strong oxidizing properties [8].

A direct attack of ozone predominates in the acidic environment or when substances dissolved in the solution react very quickly with ozone molecules. These are highly selective reactions. An indirect attack of ozone takes place through free radicals formed when reacting with water and its components. Hydroxyl radicals, which are very reactive, play a major role in the reaction. The radical processes predominate in alkaline environment and are not selective. Rate constants of the radicals reacting with organic compounds are very high [8].

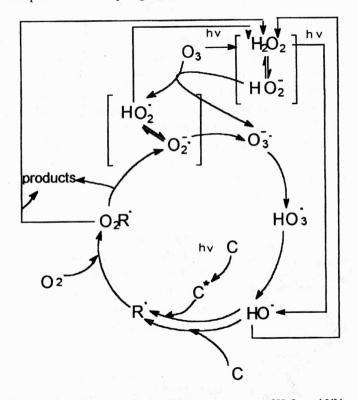


Fig. 4. Mechanism of ozonation in the presence of H₂O₂ and UV

The efficiency of indirect ozone action may be enhanced by introducing such radical-generating agents as H_2O_2 and UV irradiation into the system [9], [10]. Figure 4 illustrates the mechanism of ozonation in the presence of H_2O_2 and UV.

Many studies have confirmed that when H_2O_2 is added to a solution, ozone is rapidly degraded and OH[•] radicals are formed, thus the process of degradation of pollutants takes place much faster [10]–[12]. Theoretically the molar ratio of hydrogen peroxide to ozone is 1:2. However, under real conditions this ratio can change at high concentration of pollutants [10]. This parameter is one of the main factors affecting reaction yield and rate. Hydrogen peroxide is the so-called "radical sweeper", hence too much of it may have a negative effect on the reaction [10], [12]. The rate constant of the reaction of H₂O₂ with OH[•] radicals is $2.2 \cdot 10^7 \text{ (mol/dm}^3)^{-1}\text{s}^{-1}$, while the rate constants of the reaction of these radicals with organic compounds present in water are most frequently of the order of $10^3 - 10^{10} \text{ (mol/dm}^3)^{-1}\text{s}^{-1}$ [9].

When analyzing oxidation in H_2O_2/UV system, it was observed that disintegration of hydrogen peroxide due to UV irradiation was the most direct reaction of hydroxyl radicals formation [10], [11], [14]. One quantum of absorbed UV light of wavelength of 254 nm was sufficient to form 2 OH[•] radicals.

Recently, a growing interest in using Fenton's reagent to advanced oxidation processes of wastewater resistant to traditional methods of treatment is observed. In parallel, as a result of the reaction of Fe^{2+} with H_2O_2 , trivalent iron ions, known to be good coagulants, are formed, and in such a way a part of pollutants can be removed by precipitation. HABER and WEISS have found that the actual oxidizing species are hydroxyl radicals [16], [17]. Many theories have been developed on the mechanism of oxidation of organic compounds by Fenton's reagent (H_2O_2 and $FeSO_4$). The mechanism of this reaction according to Walling is presented in figure 5.

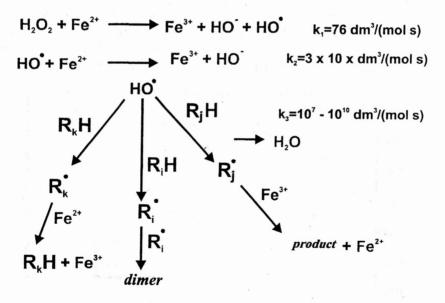


Fig. 5. Mechanism of Fenton's reaction according to Walling

Summing up, this short review of chemical oxidation methods and the results obtained should be considered. As a result of chemical oxidation large particles of organic pollutants are degraded into smaller parts to which oxygen is introduced, thus giving alcohols, aldehydes or carboxyl acids. These oxidized forms obtained due to the attack of OH[•] radicals are in most cases much easier biodegradable than the output forms.

Review articles on industrial applications of advanced oxidation of wastewater [18], [19] have noted a growing interest of various industrial branches in these methods. However, all these articles emphasize that further studies, particularly on oxidation mechanism and kinetics, are necessary.

4. INTEGRATION OF BIOLOGICAL AND CHEMICAL OXIDATION METHODS

When discussing biodegradation and chemical oxidation of wastewater, we should clearly define the notions of biodegradability and susceptibility to oxidation as well as efficiency of these processes. The most generally used global parameters characteristic of wastewater are as follows: biological oxygen demand (BOD₅) and chemical oxygen demand (COD), and, frequently, total organic carbon (TOC) or dissolved organic carbon (DOC) which denote the presence of organic compounds that due to chemical oxidation are entirely mineralized giving CO₂. As a measure of biodegradability the BOD₅/COD ratio is assumed. For example, for biodegradable municipal wastewater this ratio is 0.4-0.5 and this is why in view of the advanced oxidation of industrial wastewater there is a tendency to increase the BOD₅/COD ratio to this value and then to subject the wastewater to biological treatment. The effect of ozone and UV light on the enhancement of biodegradability of textile wastewater is exemplified in figure 6 [20].

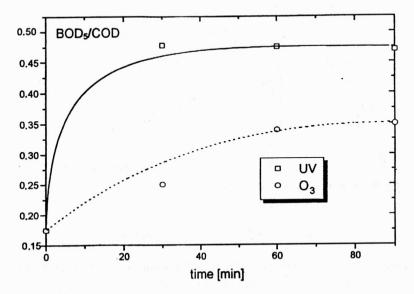


Fig. 6. BOD₅/COD ratio versus time for various AOPs of textile wastewater

The BOD₅/TOC ratio is also suggested to be used as a measure of biodegradability [21]. The COD/TOC ratio informs of susceptibility of a substance to oxidation. Low values of this ratio mean a high extent of oxidation, for example for oxalic acid COD/TOC = 0.6 contrary to alkanes for which these values range from 4 to 5. Not always these parameters represent the actual extent of biodegradability, because due to chemical oxidation also BOD₅ decreases and the BOD₅/COD ratio can remain unchanged or even may decrease as has been observed when treating textile wastewater by means of the activated sludge method [20]. This ratio provides no information on wastewater toxicity or biodegradation rate and degradation kinetics which are so important from the point of view of a designer of wastewater treatment plants [22]. Few publications are concerned with kinetics studies. The most popular form of describing the biological oxidation kinetics is Monod's equation:

$$\frac{dS}{dt} = -\frac{1}{Y_{X/S}} \frac{r_{\max}S}{K+S} X$$

where the substrate S concentration is represented by COD, with kinetic parameters r_{max} and K, depending on the chemical oxidation method applied. It can be expected that the Monod constant K, denoting a capability of a given compound to serve as a carbon source in metabolic processes, should decrease due to chemical oxidation. The yield coefficient $Y_{X/S}$ can be determined from direct measurements of the substrate S concentrations at the inlet (in) and outlet from the reactor and biomass concentration X using the formula:

$$\frac{1}{Y_{X/S}} = \frac{S_{\rm in} - S}{X}.$$

It is very important to determine the global efficiency of the process consisting of chemical and biological oxidation steps. There is an interaction between the two steps, and the extent of mineralization in particular steps is not always a relevant value. For example, wastewater can almost entirely be mineralized (95%) in the second biological process, while in the chemical process mineralization did not occur, whereas without this pretreatment, biomineralization would be impossible. YAMAZAKI et al. [23] suggested a simple method for comparing efficiencies of these subsequent processes. First, the goal has to be determined: whether mineralization should be completed or some global indices (COD, TOC) should be decreased to some admissible values, or wastewater toxicity should be eliminated, etc. If the chemical process efficiency is denoted as $E_c = (\%$ of treatment goal attained in chemical step)/(100% attainment of treatment goal) and, by analogy, the biological process as $E_b = (\%$ of treatment goal attained in biological step)/(100% attainment of treatment goal), the total efficiency of the combined process would be equal to $E_{tot} = E_c + E_b$, and relative efficiencies of respective steps are illustrated by ratios of E_c/E_{tot} and E_b/E_{tot} .

To estimate the efficiency of wastewater treatment, SCOTT and OLLIS [24] propose to use the ratio of efficiences attained in particular methods to the efficiency that would be accomplished if either of the individual processes were used solely, assuming equal reaction time, oxidant dose, economic cost, etc. This ratio provides a measure of the synergism obtained when combining processes and allows for comparison of this measure as a function of system variables such that optimal reaction conditions may be identified. Naturally, there are clear limitations both economic and physical (reactor volumes, flow rates, treatment time). ESPULGAS and OLLIS [25] modelled such a combined system of chemical and biological oxidation of a substance resistant to biodegradation using the limitations listed above. The authors found a maximum efficiency of the process as a function of reaction time. For example, the treatment efficiency reaching 85% can be attained in 7.2 hours in a (photo)chemical reactor alone, or in 3 hours in the (photo)chemical reactor and 7 hours in a biological reactor. The second variant is much more economical.

In general, it can be stated that if both chemical and biological oxidation methods are compared, the capital and operating costs of the biological processes are much lower (5 to 20 and 3 to 10 times, respectively) than those of the chemical process. These effects often decide that biological methods are used both prior to and after chemical treatment, as in the case of textile wastewater biological steps in order to improve the biologicadability of the so-called hard detergents and synthetic dyes resistant to biologicaline [26].

A survey of studies on the combined chemical and biological degradation of organic pollutants in wastewater published up to 1995 is presented by SCOTT and OLLIS [24]. However, much advancement is observed in this field and it is impossible to discuss all studies which have been carried out during the last two years. To realize how fast the advances are, one should mention four international conferences on oxidation technologies [27]-[30] which were organized during last years. At each of them several papers were devoted to the subject of combined chemical and biological oxidation. An exemplary application of this technology is the wastewater treatment plant opened in 1994 at BASF Schwarzheide GmbH [31]. The raw wastewaters containing polyethers (PE) that are difficult to biodegrade and nitrocresols from the dinitrotoluene (DNT) production, which are resistant to biological degradation, are subjected to adsorption onto excess activated sludge, which removes insoluble and hydrophobic fractions. The sludge is dewatered by thickening and sent combustion. The following biological pretreatment mineralises the easily biodegradable substances in the wastewater, which is then ozonated and subjected to the second biotreatment. In the central wastewater plant, the final purification occurs together with the treatment of the collected sewage of the site. Further, the off-gas from the ozonation step, which contains almost pure oxygen, is used for the aeration of the activated sludge. Figure 7 presents the schematic view of wastewater treatment plant at BASF Schwarzheide,

Germany. Annually 68000 m^3 of wastewater are treated at the cost of treating 1 m^3 equal to 18 DM.

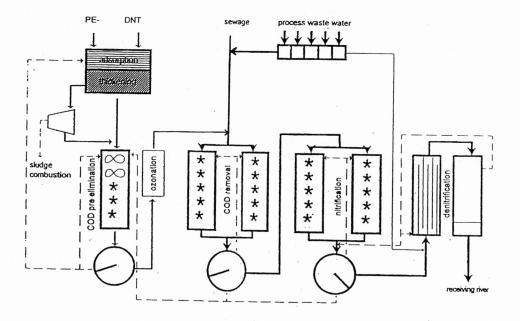


Fig. 7. Scheme of wastewater treatment plant at BASF Schwarzheide, Germany

Another example of the integration of physicochemical and biological methods of noxious wastewater treatment is a system based on Biomembrat-plus technology which operates in the waste disposal site in Berg, Landkreis Germersheim [32]. The system has worked since January 1996. Effluents from the landfill at the volumetric flow rate ~50 m³/day and COD equal to 4500 mg/dm³ are directed to a biological treatment plant where, contrary to the traditional treatment plants with activated sludge, instead of settlement tanks the ultrafiltration method is applied due to which biomass concentration is kept at the level of 40 g/dm³. After the biological process, the wastewater is 5-10 times concentrated during nanofiltration; the permeate, which satisfies the requirements established for COD, AOX and nitrogen content, is removed from the system and the concentrated substance is subjected to catalytic ozonation. Due to the application of the membrane technique in wastewater concentration, ozone consumption (6 kg O₃/h, at concentration of 120 g/m³) decreased by half as compared to the use of combined biological and chemical methods only. Reduction of pollution during the chemical oxidation attained 60-70% with the rest being degraded biologically. By returning the ozonated wastewater to the biological

treatment plant the cycle is completed. This combination of physical, chemical and biological methods seems to be the most recommendable for the treatment of other noxious industrial wastewaters.

The first industrial applications of the integrated methods of chemical and biological treatment of wastewater are successful and very promising for the further development to our prosperity and better living conditions in a clean and healthy environment.

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ZINTEGROWANE PROCESY CHEMICZNEGO I BIOLOGICZNEGO UTLENIANIA ŚCIEKÓW

Biologiczne oczyszczanie ścieków stosuje się od stu lat, jednakże nie zawsze jest ono skuteczne. zwłaszcza w przypadku ścieków przemysłowych, ze względu na obecność ksenobiotyków. Efektywność biodegradacji można znacznie zwiększyć, stosując metody utleniania chemicznego. Ostatnio obserwuje się wzrastające zainteresowanie zastosowaniem tzw. procesów pogłębionego utleniania (AOP), zwłaszcza w skali przemysłowej, dlatego postanowiono zaprezentować podstawy zastosowań kombinowanych metod utleniania ścieków przemysłowych. Uogólnienia dotyczące integracji metod utleniania ścieków na drodze chemicznej i biologicznej zilustrowano przykładami biodegradacji ścieków włókienniczych, będących od lat obiektem badań autora.

