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Reactions of Non-Ionic Surfactants, Triton X-n Type, with OH Radicals. A Review

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Abstract

The mechanism of decomposition of non-ionic surfactants, Triton X-n type, under the influence of ionising radiation is described. The survey of the available data strongly suggests that OH radicals are the main intermediates responsible for the radiation-induced destruction of wastewater containing non-ionic surfactants. The OH radicals' contribution to advanced oxidation processes is discussed, including the case of textile wastewaters. The importance of pulse radiolysis technique is underlined with regard to the kinetic data determination.

Key words: non-ionic surfactants, decomposition, ionising radiation oxidation process, wastewater, pulse radiolysis technique.

Introduction

Synthetic surfactants have become a significant fraction of dissolved organic pollutants in water eco-systems. The contribution of non-ionic surfactants is still growing, and at present alkylphenyl polyethoxylates are most widely used at industrial level [1]. A significant amount of pollution by non-ionic surfactants has its origin in textile wastewater. In the near future a decreasing tendency to apply them may be expected, due to their resistance to biodegradation and the generation during the degradation of some persistent metabolites which are much more toxic than the primary substrate [1,2].

One of the most extensive method of wastewater treatment is oxidation. Chemical oxidation processes have been used to decompose surfactants to terminal end-products (CO_2 and H_2O), or to intermediate ones that are more sensitive to biodegradation, or less toxic.

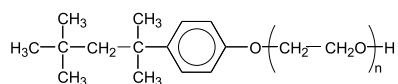
Oxidation can be carried out in the simplest way by the direct reaction of oxygen with the wastewater. In order to increase the yield of the process, oxidation at high temperatures and high pressure can be applied. Oxidation of pollutants can also be done by using not only oxygen but also other agents. Ozone, hydrogen peroxides, chlorine, chlorine dioxide, persulphate and potassium permanganate are oxidants which have been success-

fully applied in water and wastewater treatment.

Ozone is a very strong oxidant, more powerful than others commonly used in water purification [3,4]. When ozone is introduced into wastewater containing organic impurities, it can react with them directly under acidic conditions, or through oxidation of the additives via reaction with OH radicals. These very reactive species are also generated by activation of hydrogen peroxide using different stimulation agents such as UV light, ionising radiation and Fenton's reagent, or the photo-catalytic action of TiO_2 .

It is commonly accepted to refer to the simultaneous (synergetic) action of the oxidising agents under consideration as 'advanced oxidation processes' (AOP). The efficient reaction of OH radicals with impurities can explain the good yield and versatility of AOPs.

The aim of this paper is to discuss the reactivity of OH radicals against hard, non-ionic surfactants of the alkylphenyl polyethoxylate type (Triton X-n). The structure of Triton X-n is shown below:



where 'n' can be within the range of 3-40. These non-ionic detergents are common constituents of household wastewater, but especially from industrial processes including textile factories.

The pulse radiolysis technique seems to be the best direct experimental tool for investigating fast processes in the timescale from picoseconds to seconds [5]. This method helps to establish the rate constants of many reactions involving reactive species i.e. e_{aq}^- , $^{\bullet}\text{OH}$, H^{\bullet} , HO_2^{\bullet}

radicals, and compounds under consideration including surfactants.

Many of the issues examined in this paper relate to topics already addressed in our own work. We apologise for this, but as a matter of fact a research project devoted to this subject was carried out in our laboratory in the first half of the nineties.

Reactive Species in Water Radiolysis

The radiolysis of de-aerated water produces hydrated electrons e_{aq}^- , H atoms and OH radicals. These primary intermediates are generated together; hence, oxidation (OH radicals) and reduction (e_{aq}^- and H atoms) may occur at the same time, making the system rather complex.

The reaction of any compound with e_{aq}^- has to be investigated in an aqueous solution containing tert-butanol ($0,1 \text{ mol dm}^{-3}$) as a OH radical scavenger ($k(\text{OH} + \text{tert-butanol}) = 5,9 \cdot 10^8 \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ [6]).

An H atom reaction with the solute can be investigated in an aqueous solution containing tert-butanol ($0,01 \text{ mol dm}^{-3}$) as an OH radical scavenger and HClO_4 ($0,1 \text{ mol dm}^{-3}$) in order to convert e_{aq}^- into H atoms ($k(\text{H}^{\bullet} + e_{aq}^-) = 2,3 \cdot 10^{10} \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ [7]).

The reaction of the surfactant with OH radicals can be investigated in an aqueous solution saturated with N_2O in order to convert e_{aq}^- into OH radicals ($k(e_{aq}^- + \text{N}_2\text{O}) = 9,1 \cdot 10^9 \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ [8]). More details concerning the mechanisms and kinetics involved in the above-mentioned reactions can be found in [9].

In the presence of oxygen e_{aq}^- and H, atoms can be converted into $\text{O}_2^{\bullet -}$ and

HO₂[·]. The rate constants of these reactions are very high: $k(e_{aq}^- + O_2) = 1.9 \cdot 10^{10} \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ [10]; $k(H + O_2) = 2.1 \cdot 10^{10} \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ [11]).

The Reducing Species in Radiolysis of Water-Non-Ionic Surfactant System

The reactivity of transient intermediates generated during the radiolysis of water can be tested at a very low concentration of pollutants, i.e. $\mu\text{mol} - \text{mmol}$ per litre, depending on the rate constants of the reactions under consideration.

In more concentrated aqueous solutions of non-ionic surfactants, a change in the microstructure of the system can be expected. When solubilised in water, surfactants generally tend to self-associate spontaneously above a particular con-

centration called the critical micelle concentration (CMC). Such an effect should influence the kinetic properties of the detergent molecules. The CMC values for Triton X-n - water system are rather low, less than 1 mmol dm^{-3} [12]. In addition, for very concentrated systems the energy deposited would be proportional to the electron fraction of each of the surfactant and water components.

A pulse radiolysis investigation demonstrated the formation of two solvation sites for radiolytically generated excess electrons in 20-70% Triton X-100 - water mixtures [13]. The electrons solvated in the aqueous 'phase' showed the well-known absorption band with $\lambda_{\text{max}} \sim 720 \text{ nm}$, whereas the electrons solubilised in the surfactant's palisade layer were characterised by absorption with $\lambda_{\text{max}} \sim 630 \text{ nm}$. The formation of Triton X-100 radical cations was

observed; after recombination with the electrons, a strong emission with $\lambda_{\text{max}} \sim 305 \text{ nm}$ was generated. It was proved that the structure of the surfactant-water system had influenced the reactivity of both types of solvated electrons against impurities [13].

The reaction of Triton X-100 with e_{aq}^- was investigated in an aqueous, homogeneous solution containing Triton X-100 $(1.6 \div 22) \times 10^{-4} \text{ mol dm}^{-3}$ and 0.1 mol dm^{-3} tert-butanol as a OH radical scavenger [14]. The transient spectrum caused by the reaction of e_{aq}^- with Triton X-100 represented well-known e_{aq}^- absorption with $\lambda_{\text{max}} \sim 730 \text{ nm}$ and a very weak band in near UV produced as a result of an OH reaction with tert-butanol [14]. The initial, end-of-pulse absorption of e_{aq}^- did not depend on the Triton X-100 concentration in this system. The reactivity of Triton X-100 against e_{aq}^- seems to be very low, close to the value found for 'pure' water [14]. The results are in good agreement with previously described experiments [13].

The reaction of Triton X-100 with H atoms was found to be quite fast. The corresponding rate constant was calculated to be $1.25 \cdot 10^9 \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ [14].

The Reactions of OH Radicals

As mentioned earlier, the reaction of OH radicals with surfactants can be observed in an N₂O-saturated solution. The transient absorption spectra generated by the reaction of OH radicals with Triton X-n (where n=45, 100 and 405) can be detected in near-UV range ($\leq 380 \text{ nm}$) [12,14], with maxima at ca. 300-320 nm (Figure 1). These spectral bands can be attributed to cyclohexadienyl-type radicals formed as a result of OH addition to the aromatic ring of detergents under consideration. The cyclohexadienyl-type radical bands decayed according to second-order kinetics for [Triton X-n] < CMC, probably leading to dimer formation [15]. The pseudo-first order rate constants of the reaction of Triton X-n with OH radicals were calculated from the rates of build-up of the absorption at 300 nm (see Figure 1 inserts). The dependence of the pseudo-first order rate constants of the product band growth vs. the Triton X-n concentration was characterised by two linear regions which intersect in the range of the CMC values for each surfactant (Figure 2, Table 1). This type of relationship indicates a sharp decrease in OH reactivity toward the surfactant in aggregated form.

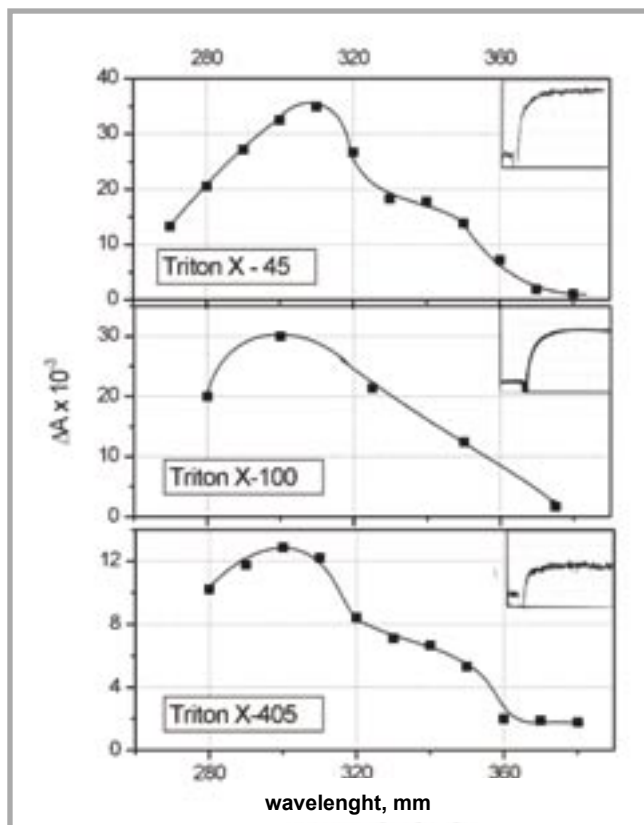


Figure 1. The absorption spectra of transients, following pulse radiolysis of N₂O-saturated Triton X-n neutral aqueous solutions. [Triton X-45] = $2.35 \cdot 10^{-4} \text{ mol dm}^{-3}$, [Triton X-100] = $1.6 \cdot 10^{-4} \text{ mol dm}^{-3}$, [Triton X-405] = $0.51 \cdot 10^{-4} \text{ mol dm}^{-3}$. The dose in the 17 ns pulse equals 36-40 Gy. Spectra taken at 1 μs after the pulse. Insert: The formation of product absorption at 300 nm, 1 $\mu\text{s}/\text{div}$. Adapted from [12,14].

Table 1. The rate constants of OH radicals with monomeric and aggregate forms of the Triton X-n, along with CMC values. Data taken from [12,14].

| Triton X-n | n | Rate constant $k_1 \cdot 10^{-9}, \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ | Rate constant $k_2 \cdot 10^{-9}, \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$ | CMC, mmol dm^{-3} |
|------------|------|---|---|----------------------------|
| X-45 | 5.0 | 5.53 | 0.56 | 0.30 |
| X-114 | 8.5 | 6.60 | 0.35 | 0.27 |
| X-100 | 9.5 | (8.8 - 9.60) | 0.50 | 0.23; 0.27 |
| X-305 | 30.0 | 18.0 | 0.50 | 0.11 |
| X-405 | 40.0 | 32.7 | 0.28 | 0.076 |

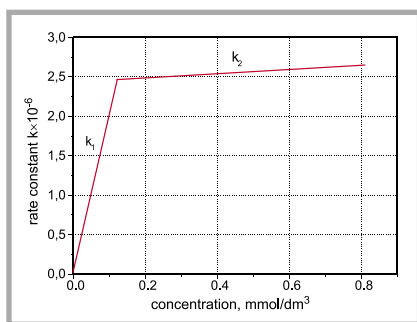


Figure 2. The influence of Triton X-n concentration on the pseudo-first order rate constants of the reaction between OH radicals (measured at 300 nm) and Triton X-n; 17 ns pulse, dose equals 40 Gy. Typical dependence for data from [12,14].

The corresponding second-order rate constants for OH reactions with Triton X-100 in monomer (below CMC) and aggregate forms (above CMC) are presented in Table 1. The CMC value found for Triton X-100, i.e. $2.3 \cdot 10^{-4} \text{ mol dm}^{-3}$, is in good agreement with the average value ($2.26 \cdot 10^{-4} \text{ mol dm}^{-3}$) calculated on the basis of the literature data listed in [12].

The rate constants k_1 of the OH reaction with the monomeric form of Triton X-n strongly suggest that the linear size of the molecule influences the k_1 value. Assuming that the length of the oxyethylene chain is proportional to the number n of oxyethylene groups $-\text{CH}_2-\text{CH}_2-\text{O}-$, the linear dependence of k_1 against n can be found (Figure 3). No such dependence is observed in the case of k_2 rate constants of OH reaction with aggregated forms of Triton X-n.

The rate constant of the reaction of OH radicals with the Triton X-100 micelles may be calculated by multiplying the k_2 by the agglomeration number, which is only known for Triton X-100. Using the values $100\text{--}140$ [16], one can obtain the respective numbers $(5\text{--}7) \cdot 10^{11} \text{ mol}^{-1} \text{ dm}^3 \text{ s}^{-1}$, indicating the diffusion-controlled reaction of OH with these surfactant micelles [12]. For Triton X-100 concentration $>$ CMC, the micelles and monomer molecules are in dynamic equilibrium and compete against OH radicals. However, in both reactions the cyclohexadienyl type radicals were formed as a product [14]. In addition, the OH radicals can also attack the surfactant molecule via H atom abstraction at either the aliphatic or poly(ethylene oxide) chains, but such radicals are absorbed at wavelengths substantially shorter than 280 nm. Such a band was successfully detected in the case of Triton X-405 [12].

OH Radicals Induced Decomposition of Triton X-100

The total decomposition of the Triton X-100 surfactant was observed in the gamma-irradiated, de-aerated aqueous system, but the highest yield was found in an N_2O -saturated, neutral solution due to the reaction with OH radicals [14,17].

The absorption spectrum of Triton X-100 in a neutral aqueous solution containing N_2O can be characterised by two bands in the UV spectral range: $\lambda=225 \text{ nm}$ ($\epsilon_{225}=7490 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$) and $\lambda \approx 275 \text{ nm}$ ($\epsilon_{275}=1435 \text{ dm}^3 \text{ mol}^{-1} \text{ cm}^{-1}$) due to aromatic ring absorption. As a result of gamma-irradiation of the ^{60}Co source, the 225 nm band evidently decreased with the dose decreasing to the negligible value in the dose range down to 30 kGy (Figure 4). This effect was Triton X-100 concentration-dependent.

The decomposition yield $G(\text{Triton X-100})$, found using the polarographic method [17] for Triton X-100 concentrations in solution from $8.3 \cdot 10^{-5} \text{ mol dm}^{-3}$ to $1.8 \cdot 10^{-3} \text{ mol dm}^{-3}$, increased from $G(\text{Triton X-100}) = 1.06$ up to $G(\text{Triton X-100}) = 4.22$ respectively [17]. The radiation yield G can be defined as the number of molecules generated (decomposed) as a result of the 100 eV dose. This original, old unit for these yields is still in use, although the use of mol J^{-1} is recommended. The relationship between these units is $1 \text{ mol J}^{-1} = 9.65 \cdot 10^6 \text{ molecules (100 eV)}^{-1}$.

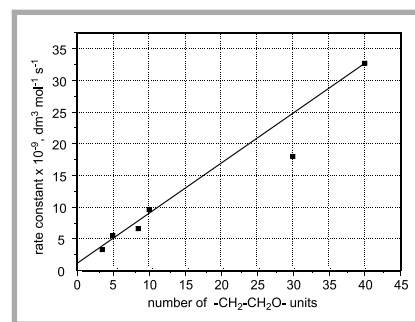


Figure 3. The dependence of rate constants of OH radicals' reaction with the monomeric form of the surfactant vs. the number of $-\text{CH}_2-\text{CH}_2\text{O}-$ units.

Up to a dose of ca. 30 kGy, no turbidity effect was observed for the solution containing $1.6 \cdot 10^{-4} \text{ mol dm}^{-3}$ Triton X-100. For more concentrated Triton X-100 solution ($\geq 8 \cdot 10^{-4} \text{ mol dm}^{-3}$), turbidity was found after a 30 kGy dose. The turbidity effects observed in our experiments can be explained in terms of the crosslinking of the surfactant molecules [18]. The formation of a large network requires bridges between all the participating surfactant molecules. The turbidity effect was found to be dependent on the Triton X-100 concentration and the dose rate (time of irradiation). The competition between the reactions of OH radicals added to the aromatic ring and H atoms abstracted from the side chains of Triton X-100 can explain the turbidity concentration effects found in the case of OH radicals with tenside molecules.

Bearing our pulse radiolysis data [14] in mind, the cyclohexadienyl type adduct

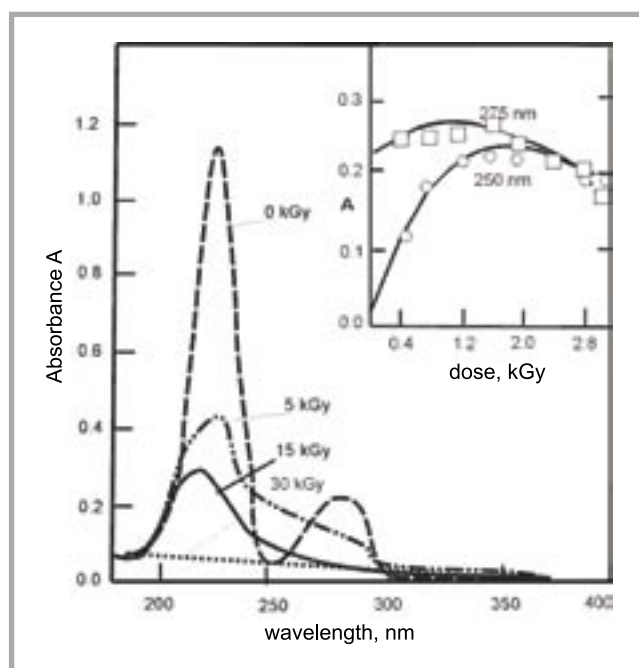


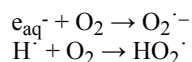
Figure 4. The influence of dose during gamma irradiation experiments on the spectrum of Triton X-100 in an N_2O -saturated aqueous system: $[\text{Triton X-100}] = 1.6 \cdot 10^{-4} \text{ mol dm}^{-3}$. Inset: influence of low doses on the optical density of the sample taken at 275 nm and 250 nm. Data from [17].

can be formed during the first primary step. The adducts decay by a second-order process leading to the radiolysis end-product described by an absorption spectrum similar to that shown in Fig. 4 (residual absorption after 15 kGy) [17]. Using high doses of ca. 30 kGy and more, the UV absorption disappeared, and the solution started to be transparent although not obviously 'pure'.

Oxygen Effect in the Radiolysis of Polluted Water

In practice, the ionising radiation is often applied in the continuous presence of air. Thus, the reducing species initially formed during the radiolysis of polluted water, i.e. H atoms and e_{aq}^- , as well as the organic radicals, are largely transformed into peroxy intermediates. The main reactions leading to the organic radicals are due to the reactivity of OH radicals, as was discussed previously.

In the presence of oxygen, the reducing species, i.e. the e_{aq}^- and H atoms, take part in the following reactions:



The rate constants of these reactions are very high, as previously discussed. The organic radicals $R \cdot$ can also be scavenged by oxygen:



The recombination of ROO radicals can generate various products, i.e. carbonyled and carboxylated compounds, alcohols and even H_2O_2 and O_2 molecules [19].

The Triton X-100 decomposition yield was dependent on the O_2 content in the irradiated system. The oxygen promoted surfactant decomposition in the aqueous solution containing Triton X-100 [20,21]. The G(-Triton X-100) for the oxygen-saturated, neutral aqueous system ($[Triton\ X-100] = 1.8 \cdot 10^{-4} \text{ mol dm}^{-3}$) was almost two times higher than for the argon-saturated solution.

It is interesting to note that the G(-Triton X-100)=0.12 for gamma-irradiated solutions where HO_2 radicals are generated ($[Triton\ X-100]=1.8 \cdot 10^{-4} \text{ mol dm}^{-3}$, $[tert\text{-butanol}]=0.01 \text{ mol dm}^{-3}$, $[HClO_4]=0.1 \text{ mol dm}^{-3}$, oxygen saturated) was lower compared with the argon-saturated system, G(-Triton X-100) = 0,34 [20].

The HO_2 radicals seem to be less reactive towards Triton X-100 [22] compared to

OH radicals. The importance of $HO_2 \cdot / O_2^{\cdot-}$ species in the radiation-induced degradation of water pollutants has been underlined by Getoff [23] in his paper devoted to the decomposition of biologically resistant compounds (chlorinated phenols, aliphatic and aromatic chlorinated compounds) in both drinking and waste water. The $HO_2 \cdot / O_2^{\cdot-}$ species and OH radicals initiate chain reactions in the systems; the products generated are further attacked by these intermediates in a prolonged irradiation procedure, and can be decomposed up to CO_2 and H_2O [23]. In combined radiation-ozone processing of water, the synergistic degradation effect of radiation and ozone was observed [23].

The $HO_2 \cdot / O_2^{\cdot-}$ species and OH radicals have been proposed as the main acting intermediates in the generally accepted mechanism of photo-initiated (and other) AOPs [24]. However, it must be conceded that the currently presented models and reaction schemes must still be treated as a 'radical chain mystery' [24] because of the complexity of the systems, and also because of the lack of directly measured reaction and kinetic data.

It is worth mentioning that the $HO_2 \cdot$ radicals have been applied in modelling the complete oxidation of organic compounds in an aqueous system at high temperatures and pressures. This technology is referred as a supercritical water oxidation. In the most advanced model of methanol oxidation in supercritical water [25, 26], $HO_2 \cdot$ radicals were used as intermediates in reactions which control the concentration of the OH radical, the main oxidant in this technology. The contribution of OH radicals to the understanding of textile wastewater treatment has been convincingly discussed in papers by Kos and Perkowski [27-32].

Summing up, it is obvious that OH radicals are the main species initiating and propagating reaction chains in AOPs.

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