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Decomposition of Anthraquinone Dye in the Aqueous Solution by Ozone, Hydrogen Peroxide or UV Radiation

Abstract

The results into investigations of the decomposition of an anthraquinone dye (polan blue E2R) in an aqueous solution induced by ozone, hydrogen peroxide and UV radiation are discussed in the paper. The effect of the ozone dose and concentration, as well as the temperature on decolouration at different initial dye concentrations, were investigated. Low ozone concentrations enabled its better utilisation in the reaction with the dye, although the process rate was slow. An increase of gas flow rate and ozone concentration at the reactor inlet caused a decrease of ozone consumption from 80% to 49%. The hydrogen peroxide concentration from 0.05 to 0.1 mol/dm³ allowed us to obtain a 45% decolouration degree after 48 hours, with the process proceeding most quickly in the first 2 hours. It was found that the yield of anthraquinone dye photolysis in the aqueous solution was affected by both the power of the UV lamp and the character of the light which it emitted. It appeared most advantageous to use mono-energetic radiation in the UV range below 310 nm.

Key words: decolouration, ozone, hydrogen peroxide, UV radiation, hydroxyl radicals, anthraquinone dye.

especially the treatment of wastewater coming from both chemical plants producing dyes and textile factories which used them have gained importance.

A specific feature of wastewater containing dyes is a strong colouration at low concentrations which is revealed by small values of such parameters as chemical oxygen demand (COD), biological oxygen demand (BOD₅) and total organic carbon (TOC), at high values of the colour threshold number (CT).

Textile wastewater is the main source of all types of surface water pollution connected with drastic colour changes. Beside strong colour, this wastewater is characterised by a high content of surfactants, various auxiliary agents and strong mineralisation (mainly chloride and sodium carbonate). Despite the relatively small content of dyes in the wastewater, their variety and stability hampers or even prevents treatment. In the case of the textile industry, reactive or direct dyes prevail; there is also a large percentage of dispersed dyes (pigments) as well as basic and acid dyes. The dyes used at present are usually non-toxic to humans, although the infiltration of coloured dyes into surface water prevents its recreational or economic use. The dyes in the water strongly absorb sunlight, which decreases the intensity of its assimilation by water plants and phytoplankton and reduces the self-purification capacity of water reservoirs. Additionally, dyes used in the textile industry may be toxic to organisms living in the surface water, and

can be resistant to natural biological degradation.

The treatment of coloured wastewater, including that from textiles, is a difficult and costly task because of its quantity and composition. Usually it is necessary to apply a three-stage treatment technique, which includes mechanical, biological, physico-chemical and chemical processes.

On the basis of theoretical data, the most advantageous method for wastewater decolouration appeared to be chemical oxidation, and next, if required, the elimination of decomposition products from water by biological methods. First, studies on dye oxidation were carried out using chlorine and potassium permanganate. In the first case, a very negative feature was the formation of carcinogenic low-molecular chlorine compounds soluble in water; in the other, the obtained results were not encouraging as far as both the final effect and total cost of the method were concerned. Recently, there are firstly the processes of oxidation based on ozone, hydrogen peroxide and oxidation combined with UV irradiation, and also all types of combination of these processes. Low selectivity, high capacity and the rate of the process, as well as the lack of by-products, are the main features that encourage further detailed studies and work on pilot-plant and semi-commercial scale installations.

A brief review of the application of ozone for textile wastewater treatment

Introduction

Dyes are a class of compounds that have been used extensively in various branches of industry, in particular in the textile and clothing industries. The industrial revolution of the 18th century caused an abrupt increase in demand for various goods and semi-finished products, including dyes, and hence natural dyes were replaced by synthetic ones. The first synthetic dye was produced by Natanson around the year 1855. The production of dyes in Poland was started by Jan Śniechowski, the founder of the 'Boruta' dye factory in Zgierz, which is still in operation.

In the initial period of production no attention was paid to environmental pollution. As the dye industry developed and the volume of production, product range and applicability increased, dye neutralisation and

should make the reader realise how advanced works in this field are and how competitive they are in relation to the classical methods [1,2].

The first wastewater decolouration system was installed in Nagahama Plants (Kanebo, Japan) in 1974. In that system, a combined action of activated carbon and ozone (a so-called GAC) was used. Coloured wastewater was supplied to a 600 m³ tank. The amount of ozone fed to the tank was 7.2 kg/h. The tank in the form of a column was filled with granulated activated carbon. As a result of the processes that took place in the column, the wastewater was decoloured and oxidised. The values of COD and BOD₅ decreased by about 60-80%.

Beside the installation presented above, in Japan there are at least seven systems that use ozone for wastewater treatment in the textile industry. The method they employ is mainly the GAC technique. The biggest of these systems was built in 1978. Its capacity was 12,000 m³ of textile wastewater per day.

Textile wastewater ozonation was used also on an industrial scale in Leek, Great Britain. The installation reached full capacity in June 1992. Beside coagulation and biological treatment it was necessary to apply ozonation as well. Capacity of the installation was 17,424 m³/day at an ozone consumption of 6.6 kg/h.

Studies on wastewater decolouration technologies by means of ozone have been carried out in Spain and Canada. They concern the wastewater from plants producing azo dyes, while in Canada they cover dispersed dyes.

Ozonation of wastewater from three dyehouses of textile factories was carried out in Taiwan. In a laboratory-scale system ozonation, coagulation, sedimentation and purification with active sludge were applied. In the full-scale installation design the capacity of 5400 tons/day was assumed. Laboratory-scale investigations in this field were also carried out in Germany and the United States.

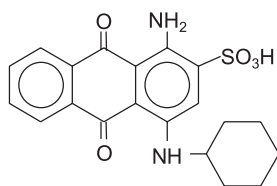
As for a separate application of hydrogen peroxide or UV radiation in the decolouration or treatment of textile wastewater, besides bench-scale investigations there has been no information on the use of these factors on a larger scale.

The UV radiation itself may be used for water disinfection, especially in swimming pools where it is applied instead of chlorination. Hydrogen per-

oxide is used mainly in the textile industry for bleaching. Recently, the growing applicability of H₂O₂ has been observed in the Fenton process applied to the purification of industrial wastewater and leachates [3,4].

Material

The investigation covered an anthraquinone dye, polan blue E2R (Acid Blue 62, C.I. 62045). Because of their chemical structure, the anthraquinone dyes are often the subject of studies on decolouration in water solutions [5-9]. In the textile industry it is applied in dyeing polyamide fibres and wool. Its chemical structure is presented below:



Experimental Procedure and Analytical Methods

The system in which experiments on dye decomposition in water solutions using ozone, hydrogen peroxide and UV radiation were conducted has been presented in our previous studies dedicated to advanced oxidation processes [10,11].

Dye oxidation processes were carried out in a Sovirel glass reactor 1.5 dm³ in volume, equipped with a thermostating jacket. In the centre of the reactor there was a quartz tube with light sources. The mixture of oxygen and ozone flowing to the reactor was supplied to the solution by means of a porous plate which enabled fine gas bubbling.

The solutions were analysed by a spectrophotometric method using a Hewlett Packard HP8452A device. The spectra were collected in the range from 190 to 800 nm. For quantitative determinations, absorption data at 620 nm with the maximum of the dye spectrum in the visible range were used. Because of the intensity of solution colouration, the length of optical path was 1 mm. Dye solutions of the concentrations 100 and 200 mg/dm³ (0.25 and 0.5 mmol/dm³) were tested. The initial pH value was natural (pH=3.8). The solution volume was 1.2 dm³.

The experimental procedure was as follows:

1. In the case of ozonation, the oxygen flow rate ranged from 3 to 40 dm³/h, and the ozone concentration in the

gas at the reactor inlet was within the range from 6 to 36 mg O₃/dm³. The process time usually did not exceed 60 minutes; samples for analysis were taken every 5 minutes. The temperature applied ranged from 288 to 333 K (15-60°C).

2. The treatment with hydrogen peroxide consisted in adding 5 or 10 cm³ of 30% H₂O₂ solution (perhydrol) to 1 dm³ of dye solution at the concentration of 100 mg/dm³. Total hydrogen peroxide was added at the beginning of the reaction. The treatment time was 48 hours. The solution was mixed by oxygen flowing at the rate of 20 dm³/h. The process temperature was 298 K (25°C).
3. In the case of UV irradiation of wastewater, the reaction time was up to 5 hours. Oxygen passed through the orifice at a rate of 17 dm³/h. The following lamps were used: a low-pressure mercury-discharge lamp TNN 15/32 of 15 W power, medium-pressure lamp of type TQ150 of 150 W electric power, and a high-pressure Q-400 burner (Hanau, Germany) of 400 W power. The experiments were carried out at the dye concentration of 100 mg/dm³; the volume of solution was 1 dm³ and the temperature 298 K (25°C).

Actinometric measurements were made in order to set oxidised UV doses supplied to wastewater samples and to calculate the quantum yields of pollutant decomposition.

To determine light intensity, a uranyl-oxalate actinometer was used. This was an aqueous solution of uranyl sulphate at the concentration of 0.01 mol/dm³ and oxalate acid at the concentration of 0.05 mol/dm³. The actinometer was sensitive to the wavelengths from 208 nm to 436 nm. Light acting on the actinometric solution caused decomposition of the oxalic acid sensibilised with uranyl ion.

The reaction was measured by determining the losses of oxalate acid at specified time intervals by means of titrating the samples with potassium permanganate at the concentration of 0.2 mol/dm³. Analyses were made in an acid medium and at an elevated temperature (~310 K).

The aqueous dye solutions were treated with ozone, hydrogen peroxide and UV radiation. All these processes were used to decolour the water solutions. The results obtained can be also used in comparison with other versions of oxidation, particularly advanced oxi-

dation with a simultaneous use of these two or three oxidising agents. More attention was given to the process of ozonation, which was considered efficient, simple and economical.

Ozonation

The polan dye decomposition with ozone was tested with respect to on the flow rate of the oxygen-ozone mixture, ozone concentration in the gas, the process temperature and dye concentration.

The effect of ozone concentration at the inlet to the reactor was investigated for ozone concentrations equal to 6, 17, 20 and 36 mg O₃/dm³ gas, at the flow rate of the oxygen-ozone mixture of 17 dm³/h, which gave ozone doses 85, 240, 283 and 510 mg O₃/h×dm³ solution respectively. The initial dye concentration in the solution was 100 mg/dm³. The volume of the solution was 1.2 dm³ at the process temperature of 298 K (25°C). The changes in the dye concentration during ozonation are shown in Figure 1.

The process of decolouration depends clearly on the ozone concentration in the gas at the inlet to the reactor. At a constant flow rate, this corresponds to the amount of ozone supplied to the reaction mixture (dosing rate of O₃). At determined parameters of ozone transfer to the aqueous solution, ozone self-degradation rate and reaction with the dye, the rate of ozone consumption during the decolouration of polan blue can be defined.

With an increase of the reaction time and ozone dose, a growing degree of solution decolouration is observed. As expected, the process rate is the fastest for the highest ozone concentration applied, equal to 36 mg O₃/dm³. For the lowest ozone concentration, i.e. 6 mg O₃/dm³, there was an induction period of about 5 minutes connected with setting up the reaction parameters (mainly saturating the solution with ozone). At concentrations 17 and 20 mg O₃/dm³ the induction period is very short, while at 36 mg O₃/dm³ it does not occur in practice.

Distinct induction periods are observed when dye decomposition curves are presented not as a function of time but as a dose of reacted ozone. The ozone dose corresponding to the induction period is from 10 to 25 mg O₃/dm³ solution, and increases with an increase in ozone concentration in the gas (this corresponds to time from 7 to 3 minutes).

A decrease of dye concentration in the solution depending on the ozone content in the gas mixture supplied to the reaction for three reaction times is illustrated in the inset in Figure 1. An evident increase may be observed in dye decomposition yield with a growing ozone concentration, especially for the reaction times of 15 and 30 minutes.

These relationships undoubtedly derive from the applicability of ozone supplied to the reactor in the decolouration process. Low ozone concentrations in the gas phase ensure better use of the ozone, while at high ozone content in the gas its penetration to the solution and participation in the decolouration reaction are much worse. Unfortunately, if we wish to use ozone in the most efficient way, the reaction time is prolonged significantly and the economic efficiency of the process decreases. In our system, the amount of unreacted ozone increased in time from about 20% to 60% in the gas exit.

The effect of gas mixture flow rate was investigated in the range from 10 to 40 dm³/h at a constant ozone concentration in the gas equal to 16 mg O₃/dm³ and the dye concentration of 100 mg/dm³. The process temperature was 298 K (25°C) and the reaction time was 60 minutes. A diagram of colour changes in time is shown in Figure 2. It is seen that with an increase of the flow rate (ozone dose supplied), the degree of solution decolouration increases. The relations illustrated in the inset (Figure 2, inset) show this effect even more distinctly. For the given reaction times (15, 30 and 45 min) there is a sudden decrease of dye concentration in the solution with an increase of gas flow rate.

If the dye concentration is presented not versus reaction time but versus the doses of reacted ozone, the kinetic curves for different gas mixture flow rates will not overlap. Thus, there is an impact of gas flow rate, with the worst results of the solution decolouration in the experimental conditions appearing at 30 dm³/h. This can be explained by the fact that there are at least two effects in different directions. On the one hand, with an increase of the gas flow rate, the time of contact decreases and hence the conditions of ozone penetration to the solution deteriorate; on the other hand, mixing is more intensive, which has a favourable effect on the reaction rate. As a result, there is a relationship between gas flow rate and decolouration yield at a constant dose of the reacted ozone.

The amount of unreacted ozone in the process conditions increases (with an increase in the gas flow rate) from about 24% to 70%.

The effect of temperature on dye decomposition during ozonation was investigated for the gas flow rate of 17 dm³/h and at a low ozone concentration in the gas of 6 mg O₃/dm³. A slight effect of temperature on the process of decolouration was found. The best results were obtained at 318 K (45°C). The dye decomposition rate close to optimum was obtained at 323 K (60°C). A slightly slower rate was reported at 288 and 203 K (15 and 30°C) (Figure 3). It is well known that gas solubility in liquid (ozone in water) decreases with temperature growth, while the rate of ozone decomposition in water and that of reaction with the dye increases. The latter effect probably prevails, as is reflected by the results obtained. This dependence is clearly visible in the inset in Figure 3, where the decolouration degree depends only slightly on temperature for the three selected reaction times of 20, 30 and 45 min.

In the case of elevated temperature, the amount of unreacted ozone in the outlet gas decreased to 8.3% at 318 K (45°C) and 5.3% at 333 K (60°C). This is undoubtedly related not so much to its reaction with the dye particles as to the process of ozone self-degradation.

When increasing the dye concentration up to 200 mg/dm³, a decrease in percentage yield of the dye decomposition was observed, as expected. This effect is visible when dye degradation is considered with respect to the reacted ozone dose (Figure 4). The absolute reaction rate increases with an increase in the dye concentration.

Summing up the process of polan blue decomposition in the water solution by means of ozone, it is concluded that the choice of optimum process parameters would require a decision concerning optimisation criteria. We should decide whether the main criterion should be the process duration or the dose of reacted ozone (most advantageous in the decolouration process). In both cases the results will be different. In technological conditions a compromise should be made between these two parameters, because both are very important for the process' economy. A decrease in the ozone supply rate reduces the direct costs of ozone (electrical energy and exploitation costs), but prolongs the process duration, thus increasing the residence time of the wastewater

and the installation capacity (investment costs).

The application of hydrogen peroxide

The conditions and technique of carrying out the decomposition of the anthraquinone dye by means of hydrogen peroxide have been discussed earlier. In this study, high concentrations of hydrogen peroxide were used bearing in mind the poor results of colour reduction at low concentrations obtained in our previous investigations on the decomposition of non-ionic detergents, Tritons and Tergitols [12-14] and textile wastewater - both in a model and in reality [15,16].

Figure 5 shows the decolouration degree in time up to 48 hours for two different initial concentrations of H_2O_2 . In this time (48h), hydrogen peroxide in the solution at temperature of 298 K is wholly degraded. In the first case the amount of H_2O_2 added to the solution was $5 \text{ cm}^3/\text{dm}^3$, which corresponded to the peroxide concentration equal to $0.049 \text{ mol}/\text{dm}^3$, while in the second case the concentration was twice as high and amounted to $0.098 \text{ mol}/\text{dm}^3$.

It follows from the relationships obtained that the main course of the reaction takes place in the first 2 hours when the decolouration degree increases linearly. This may be explained by the assumption of a constant concentration of hydroxyl radicals, products of hydrogen peroxide decomposition in the first phase of the reaction course, when the H_2O_2 concentration is high enough. The second stage, in which decolouration of the solution is much slower, is probably related to secondary processes involving colour products of polan blue oxidation. Processes leading to the reconstruction of dye molecules are also possible [17-19]. The yield of decolouration does not depend strictly on the initial concentration of hydrogen peroxide, because when it is doubled, the decolouration degree increases from 5.8% to 7.8% after 45 minutes and from 17.4% to 20.1% after 130 minutes. Hence, even a doubled concentration of H_2O_2 caused only a slight (ca. 4%) increase in the polan dye's degree of degradation.

Photochemical process

The decomposition of polan blue E2R in the aqueous solution by means of UV radiation (photolysis) was carried out using three types of lamps. They differed both in the type of emitted spectrum and in their power. The maximum photo-reaction time was 5

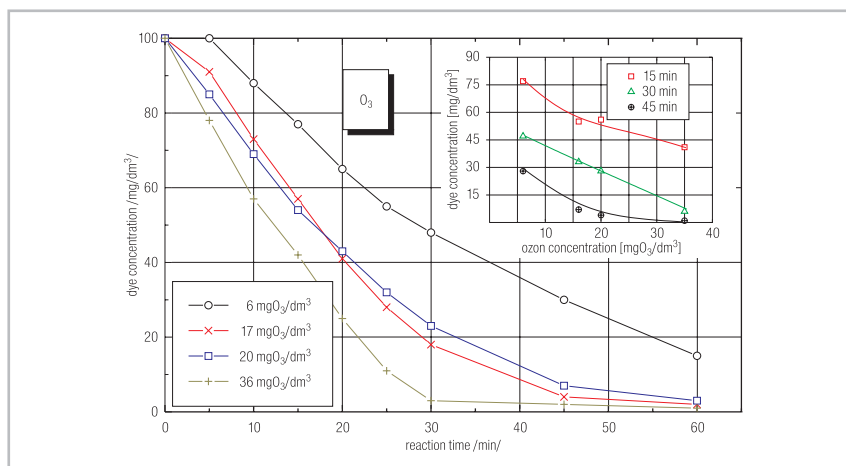


Figure 1. The effect of ozonation time on changes in dye concentration (solution colouration) for different ozone concentrations (6, 17, 20 and $36 \text{ mg O}_3/\text{dm}^3$) in the oxygen-ozone mixture. Temperature 25°C . Flow rate of the gas mixture $17 \text{ dm}^3/\text{h}$. Dye concentration in water $100 \text{ mg}/\text{dm}^3$. Inset: Dependence of dye concentration on ozone concentration in the oxygen-ozone mixture for three reaction times 15, 30 and 45 min.

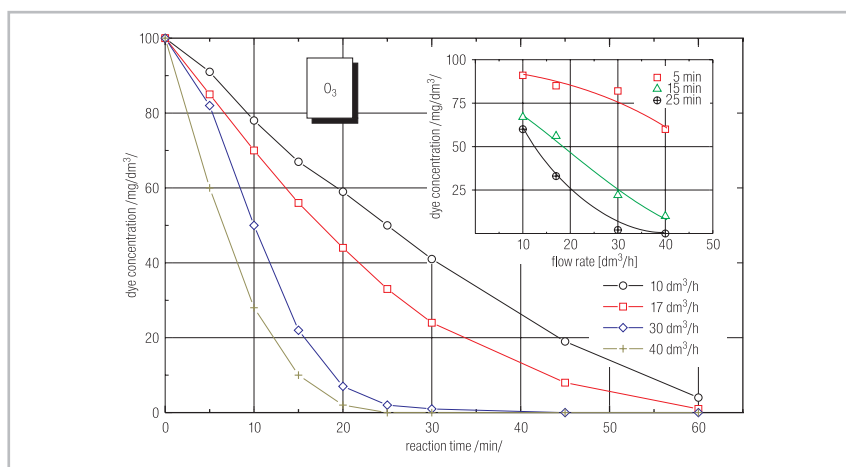


Figure 2. The effect of ozonation time on changes in dye concentration (solution colouration) for different gas mixture flow rates (10, 17, 30 and $40 \text{ dm}^3/\text{h}$). Ozone concentration in the oxygen-ozone mixture $17 \text{ mg O}_3/\text{dm}^3$. Temperature 25°C , dye concentration $100 \text{ mg}/\text{dm}^3$. Inset: Dependence of dye concentration on gas mixture flow rate for three reaction times 15, 30 and 45 min.

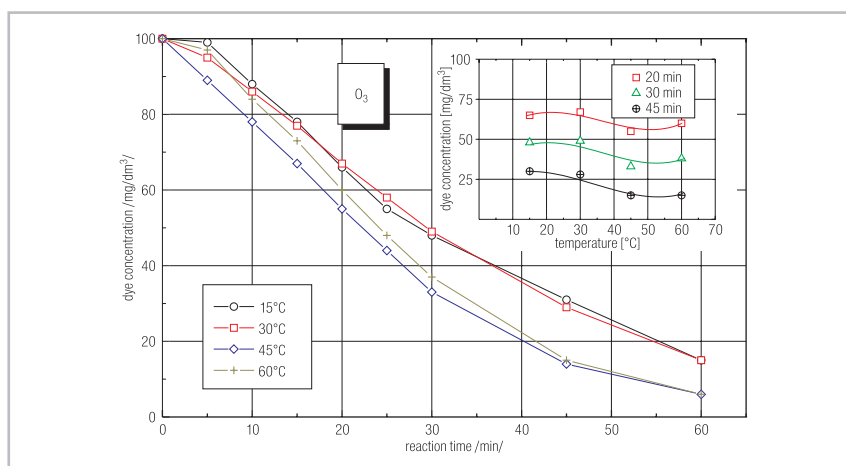


Figure 3. The effect of ozonation time on changes in the dye concentration (solution colouration) for different temperatures of the solutions (15, 30, 45 and 60°C). Gas flow rate $17 \text{ dm}^3/\text{h}$, ozone concentration in the oxygen-ozone mixture $6 \text{ mg O}_3/\text{dm}^3$, dye concentration in the solution $100 \text{ mg}/\text{dm}^3$. Inset: Dependence of dye concentration on the solution temperature for three reaction times 20, 30 and 45 min.

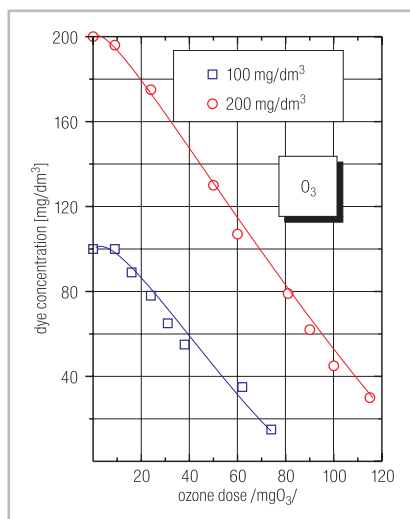


Figure 4. Decomposition of alizarin blue in the aqueous solution for two initial concentrations 100 and 200 mg/dm³ depending on ozone dose.

hours. The relevant data are given in Table 1.

For instance, a low-pressure mercury-discharge lamp TNN 15/32 of 15 W power emitted light of intensity $1.97 \cdot 10^{22}$ quantum/dm³h, which corresponded to $3.27 \cdot 10^{-2}$ Einstein/dm³h. 75.9% of the quanta were radiation at $\lambda < 310$ nm with maximum at $\lambda = 254$ nm. The number of quanta is given per time unit and volume unit of the reactor in which experiments were carried out. The quantum yield of the decomposition is calculated on the assumption that the whole colour change in the solution is related to the decomposition of dye molecules. Photochemical capacity is the amount of decomposed molecules per 100 eV of absorbed light energy. The power emitted by the

lamp in the form of light was 3.33 W/dm³.

When comparing the applied lamps and taking into account light emission below 310 nm only, the most efficient appeared to be the TQ150 ($2.61 \cdot 10^{-2}$ Einstein/dm³h) and TNN15/32 ($2.48 \cdot 10^{-2}$ Einstein/dm³h) lamps, while the Q-400 burner was about 3 times less efficient ($7.4 \cdot 10^{-3}$ Einstein/dm³h).

The results of polan dye decomposition by photolysis have no direct connection with the determined intensity of light emitted by particular lamps. The 15 W lamp, emitting the most monoenergetic light in the UV range ($\lambda = 254$ nm), gives very good results of decomposition both in the aspect of time and quanta absorbed. On the other hand, the medium-pressure mercury-discharge lamp, (despite its 10 times higher electric power) with a similar number of quanta in the UV range, gives much worse results of dye decomposition both in time and when analysing decolouration as a function of the absorbed quanta.

It is interesting that the quantum yield of polan blue decomposition is almost identical for the TQ150 lamp and the Q-400 burner, despite significant differences in the emission spectrum of both light sources.

The character of decomposition curves reveals that in the aqueous solution the dye is degraded as a result of photolysis and photo-oxidation when UV radiation is used. The UV radiation that is emitted with light impedes photodegradation. The significant contribution of UV radiation to the process may lead to a serious hampering of the

reaction, whose rate is then at least 10 times lower than when monoenergetic radiation from the UV range is used.

Conclusions

The experiments revealed the high susceptibility of polan blue E2R to degradation by ozone, hydrogen peroxide and photolysis. The best results of decolouration were obtained using ozone as an oxidant. The degradation rate was affected by ozone dose and concentration in the gas mixture, its flow rate through the solution, and by temperature.

The process may be optimised by taking into account the dose of reacted ozone or the time of reaction. High degrees of ozone utilisation and low doses of the reacted ozone required a slow gas flow rate and a low ozone concentration therein.

This in turn caused the reaction time to be longer, which as a consequence required a larger reactor volume. In industrial conditions, a compromise is necessary and the decolouration should be optimised, taking into account the process' economy.

The use of hydrogen peroxide as an oxidant, or to carry out dye photolysis in the presence of oxygen, does not bring about satisfactory results. Reaction times are long, and the required light intensity or H₂O₂ concentration is high. From the economic point of view, these two factors have no practical importance.

In the case of industrial coloured solutions, which do not require very accurate treatment but only a complete

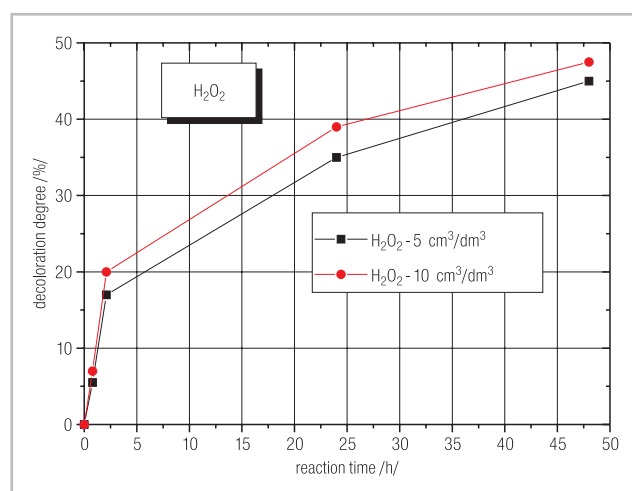


Figure 5. Dependence of decolouration degree of the solution on reaction time for two doses of hydrogen peroxide added (5 cm³ and 10 cm³ H₂O₂/dm³ solution). Initial dye concentration 100 mg/dm³, temperature 25°C.

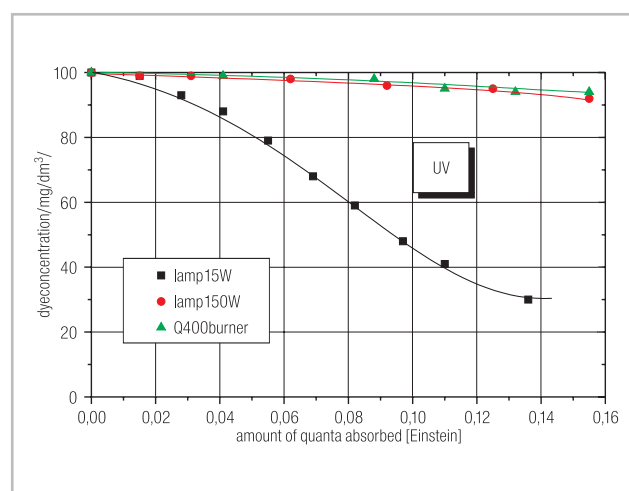


Figure 6. Kinetics of alizarin blue photolysis depending on the absorbed dose of radiation for three different radiation sources: low-pressure lamp 15 W, medium-pressure lamp 150 W and Q-400 quartz burner. Initial dye concentration 100 mg/dm³, temperature 25°C.

Table 1. Comparison of parameters and results of photochemical decomposition of anthraquinone dye by light emitted by different lamp types.

Quantity	Low-pressure lamp 15 W	Medium-pressure lamp 150 W	Q-400 quartz burner
Amount of quanta emitted per time unit for the whole UV-VIS range	$1.47 \cdot 10^{22}$ quantum/dm ³ h	$0.975 \cdot 10^{23}$ quantum/ dm ³ h	$5.52 \cdot 10^{22}$ quantum/dm ³ h
Quantum yield calculated for total absorbed light	$1.72 \cdot 10^{-3}$ molecules/quantum	$1.29 \cdot 10^{-4}$ molecules/quantum	$9.68 \cdot 10^{-5}$ molecules/quantum
Amount of quanta emitted per time unit for light at $\lambda < 310$ nm	$2.244 \cdot 10^{20}$ quantum/min	$2.107 \cdot 10^{20}$ quantum/min	$8.94 \cdot 10^{19}$ quantum/min
Quantum yield calculated for light at $\lambda < 310$ nm	$2.27 \cdot 10^{-3}$ molecules/quantum	$1.19 \cdot 10^{-3}$ molecules/quantum	$1.19 \cdot 10^{-3}$ molecules/quantum
Power of light emitted in the whole UV-VIS range	3.33 W	15.52 W	8.35 W
Photochemical quantum yield calculated for the whole absorbed light	$4.07 \cdot 10^{-2}$ molecules/100eV	$4.34 \cdot 10^{-3}$ molecules/100eV	$3.32 \cdot 10^{-3}$ molecules/100eV
Power of light emitted in the range of $\lambda < 310$ nm	2.92 W	2.48 W	1.00 W
Photochemical quantum yield calculated for light at $\lambda < 310$ nm	$4.64 \cdot 10^{-2}$ molecules/100eV	$2.71 \cdot 10^{-2}$ molecules/100eV	$2.77 \cdot 10^{-2}$ molecules/100eV

decolouration which would enable their re-use in the technological process, one-stage processes of chemical oxidation through ozonation can be used successfully.

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