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Investigation of the Efficiency of the UV/H₂O₂ Process on the Removal of Dye Acid Green 16 from Aqueous Solutions: Process Optimization and Toxicity Assessment

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Abstract

The effects of the removal of Acid Green 16 (100 mg AG-16/dm³, COD = 111 mg O₂/dm³) from aqueous solutions by the UV/H₂O₂ process in UV reactors: low pressure lamp (LP, 15 W) and medium pressure lamps (MP, 150 W) are presented. The best results of AG-16 removal were obtained for H₂O₂ 250 mg/dm³ (99.85%, AG-16 = 0.15 mg/dm³) and 200 mg/dm³ (99.80%, AG-16 = 0.20 mg/dm³) for LP and MP lamps, respectively, with the same parameters, i.e. 30 min reaction time and pH 6. Under these conditions, the AG-16 solution was completely discolored and the COD removal efficiency was 57.3% (LP lamp) and 63.4% (MP lamp). However, at optimum conditions of decolorisation, no decrease in the toxicity of solutions (Microtox test) was observed. For the MP lamp, the toxicity of solutions remained at the same level as in the initial solutions (Toxicity Unit, TU = 3), whereas in the case of the LP lamp, the TU value after the process increased to 6. In conclusion, the AOPs for toxic pollutants should also be optimised from the point of view of toxicity.

Key words: Acid Green 16, decolorization, toxicity, UV/H₂O₂ process.

and mutagenic properties in relation to aquatic organisms, and in addition they can cause irritation and allergic dermatitis, as well as being able to act as genotoxins (e.g. Acid Green 16) for mammals [2-4]. For the purification of colour wastewater, chemical and physicochemical methods are used such as coagulation, sorption, nanofiltration, reverse osmosis and chemical oxidation. The main advantages of coagulation are ease of use and low investment and operating costs, while the disadvantage is the large amount of sludge generated [5]. Different types of sorbents, both natural and waste, are used in the sorption process (apple pulp, wheat pods, zeolite, chitosan, sawdust, fly ash) [6-8], as well as synthetic [9]. The sorption process is highly effective, but it requires prior removal of the suspension from the effluent, which is usually associated with higher process costs. Alternative methods of removing dyes from wastewater are membrane techniques such as nanofiltration and reverse osmosis [10, 11]. Their main disadvantage is high operating costs, and in addition they do not degrade the dyes that remain in the retentate. Generally most of the dyes are found to be resistant to the conventional treatment process, and the disposal problem of precipitated wastes is the main disadvantage of precipitation methods [12]. Also chemical oxidants (e.g. ozone or hydrogen peroxide) used alone are not effective at higher concentrations of dyes because of low rates of reaction at economically acceptable con-

centrations of oxidants. Therefore advanced oxidation processes (AOPs) are increasingly used to remove many dyes from wastewater [13, 14]. As a result of oxidation with OH[•] radicals, the dyes are substantially degraded, as opposed to the methods previously mentioned. AOPs such as UV/H₂O₂ [15, 16] and the Fenton reagent [17, 18] are used for the purification of colour wastewater. There are also studies conducted on, inter alia, UV/TiO₂ and UV/Fe⁰ methods [19, 20] as well as those using alternative sources of hydrogen peroxide [21, 22]. Sewage containing dyestuffs can be treated applying biological methods using activated sludge or a biofilter, which require long retention times in the system. Only certain synthetic dyes undergo aerobic decomposition. In the case of aromatic compounds, it is effective to use biological anaerobic methods. Integrated systems operating under anaerobic-aerobic conditions [23-25] are also used.

This paper presents the results of a study on the removal of Acid Green 16 (AG-16) acid dye from aqueous solutions by the UV/H₂O₂ method. Optimum parameters of the UV/H₂O₂ process (pH, H₂O₂ dose, reaction time, lamp type) were chosen, for which the toxicity of the aqueous solutions was determined, because AG-16 negatively affects mammals and may cause long-term adverse effects in an aquatic environment. It is not permissible for both solid and liquid forms to enter drains or natural water reservoirs

Introduction

Dyes are one of many organic pollutants which get into industrial wastewater due to their wide application in the textile, printing, cosmetics, pharmaceutical, food, and other industries. In the course of trade, there are more than 100 000 commercial products containing dyes, and their annual production is estimated at over 7 · 10⁵ Mg [1]. Even small amounts of dyes present in wastewater give it a specific colour. Colored wastewater introduced into the aquatic ecosystems may cause the inhibition of photosynthesis and lead to toxic effects on aquatic organisms. Some dyes have carcinogenic

Table 1. Classification of sample toxicity [32].

TU	Classification
< 0.4	Non-toxic
0.4 < TU < 1	Moderately toxic
1 < TU < 10	Toxic
10 < TU < 100	Very toxic
TU > 100	Extremely toxic

[26]. Therefore it is necessary to remove it from the wastewater before introducing it into aquatic ecosystems.

Materials and methods

The solutions simulated were prepared by diluting commercial dye Acid Green 16 (C.I. 44025, Boruta-Zachem S.A., Zgierz, Poland) in distilled water. The dye was of high purity and used without further purification. The concentration of the dye was 100 mg/dm³ due to the fact that in their earlier dye removal studies the authors had used an initial dye concentration equal to 100 mg/dm³ each time. Thus prepared, aqueous solutions of the dye were characterised by an intense dark green color of high clarity, with COD = 111 mg O₂/dm³ and pH = 7.2. The concentration of AG-16 was determined by the spectrophotometric method by absorbance at wavelength λ=638 nm using Nanocolor Linus[®] spectrophotometer (Macherey-Nagel, Germany). Measurement of absorbance for each solution was conducted twice, the average value calculated, and the AG-16 concentration was read from the calibration curve previously prepared. pH was measured by the electrometric method using an inoLab[®] pH/ION/Cond 750 pH-meter (WTW, Germany) equipped with a SenTix[®] 81 electrode [27].

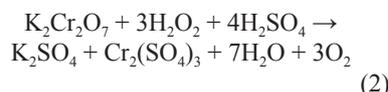
The UV/H₂O₂ process was carried out using two flow-through photo-reactors with recirculation of treated solutions. One of the reactors (Titanium, type AOP 0.5, Titanium-Wega S.A., Poland, reactor working volume = 0.5 dm³, dimension = 45 mm, height = 420 mm) was equipped with a 15W low pressure UV lamp (LP lamp) and the second reactor (Heraeus, UV-RS-2, UV-Consulting Peschl, Germany, reactor working volume = 0.5 dm³, dimension = 60 mm, height = 270 mm) with a medium pressure (MP lamp) UV TQ 150 with a power of 150W. Aqueous solutions with a volume of 0.8 dm³ were adjusted to a certain pH value with 1 mol/dm³ NaOH or 9 mol/dm³ H₂SO₄ (Chempur, Poland), and then an appro-

appropriate amount of H₂O₂ (30%, Chempur, Poland) was added to them. Aqueous solutions of the AG-16 dye were then introduced into a reaction vessel (2 dm³) placed next to the reactor on a magnetic stirrer, and then fed with a peristaltic pump to a UV reactor at a capacity of 50 cm³/min, from where they were returned to the tank. Three reaction times (10, 20, 30 min) were used. Oxidation reactions were carried out successively at pH 3, 4, 5, 6, 7, 8 & 9, and H₂O₂ concentrations were used in the range of 100-250 mg/dm³. For optimum UV/H₂O₂ process parameters, extended studies were performed that additionally included COD and toxicity. COD was determined by the standard dichromate method [28] using a CR 4200 thermoreactor and Spectroflex 6100 spectrophotometer (WTW, Germany). The COD-determined value was corrected for residual H₂O₂ according to **Equation (1)**, because the H₂O₂ increased the COD value since it acts as a reductant (reaction 2), especially in the dichromate-based analysis of COD [29]:

$$COD_{corr.} = COD_m - f \times [H_2O_2];$$

$$f = 0.4706 \times [H_2O_2] - 4.06 \times 10^{-5} \times [H_2O_2]^2 \quad (1)$$

where: $COD_{corr.}$ – correct COD value, mg O₂/dm³, COD_m – measured COD value, mg O₂/dm³, f – correction factor, $[H_2O_2]$ – concentration of residual H₂O₂, mg/dm³



The residual amount of H₂O₂ was determined by the manganometric method [30]. Toxicity tests were performed using the Microtox[®] system (MicroBioTest Inc., USA) [31] with bioluminescent bacteria *Vibrio fischeri* as toxicity indicators. The contact of *Vibrio fischeri* with a toxic substance causes variations in the intensity of light produced directly proportional to the biological activity (toxicity) of the test substance. Test results indicate the concentration of the test substance causing 50% inhibition of the bioluminescence of the test organisms (EC₅₀). Tests for all samples were performed in 3 replicates, after earlier total removal of residual H₂O₂, using Na₂SO₃ (POCH, Gliwice, Poland), as even very low concentrations of H₂O₂ resulted in the inhibition of vital activity of bacteria *Vibrio fischeri*. Wastewater toxicity is classified by the US EPA scale, in which the toxicity index is TU (Toxic Unit). This ratio is obtained by converting the toxicity test result (EC₅₀) to acute toxic-

ity units TU according to the relationship (3). The classification of wastewater toxicity is shown in **Table 1** [32].

$$TU = \frac{100\%}{EC_{50}\%} \quad (3)$$

Results and discussion

The aim of the study was to determine the most favorable UV/H₂O₂ process parameters (i.e., H₂O₂ dose, initial pH, reaction time) to achieve the high removal efficiency of AG-16 and visual discoloration of aqueous solutions of this dye with a concentration of 100 mg AG-16/dm³. Preliminary tests showed that visual discoloration occurred when the concentration of AG-16 dye was reduced to <0.4 mg/dm³. Then for the process parameters appointed, an extended analysis was performed including COD removal and toxicity tests.

Effect of H₂O₂ dose

In the studies concerning the impact of H₂O₂ on the efficiency of AG-16 removal, three different irradiation times (10, 20 and 30 min) were used at an initial pH value of 3. In the case of the LP lamp for all reaction times, as the H₂O₂ dose (range 100-250 mg/dm³) was increased, the AG-16 removal efficiency (**Figure 1**) also increased. With the MP lamp, enhancement of dye removal efficiency for all reaction times was observed only in the range of 100-200 mg H₂O₂/dm³ (**Figure 2**). For larger doses of H₂O₂ (225 and 250 mg/dm³), the effectiveness of AG-16 removal was reduced compared to the dose of 200 mg/dm³. Probably the excess H₂O₂ in this case acted as a radical scavenger OH[•] (reaction 4), which contributed to the reduction in the process efficiency.



For both types of lamps, for each H₂O₂ dose, AG-16 removal efficiency was increased by increasing the reaction time. The greatest effect of the doses of H₂O₂ used on the efficiency of the process was noted for a reaction time of 10 minutes. In such an irradiation time, using the LP lamp (**Figure 1**), increasing the H₂O₂ dose in the range of 100-250 mg/dm³ resulted in a decrease in AG-16 concentration to 6.07 mg/dm³ (93.9%) and 1.05 mg/dm³ (99%) for doses of 100 and 250 mg H₂O₂ /dm³, respectively. A similar tendency was observed for the MP lamp (**Figure 2**). Increasing the H₂O₂ dose in the range

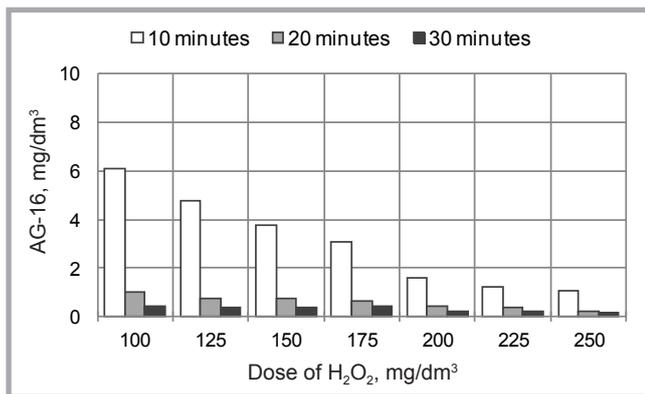


Figure 1. Changes in AG-16 concentration at different H₂O₂ doses (LP lamp, pH 3).

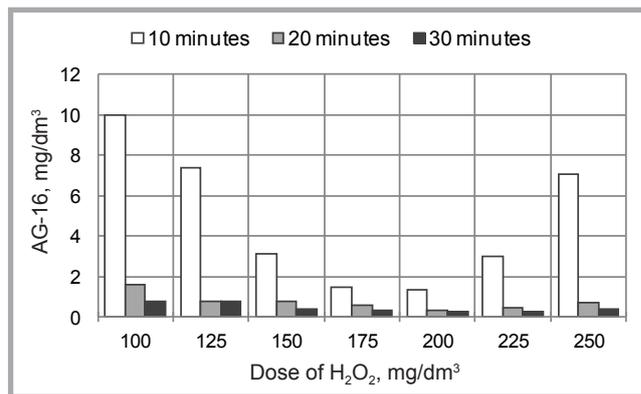


Figure 2. Changes in AG-16 concentration at different H₂O₂ doses (MP lamp, pH 3).

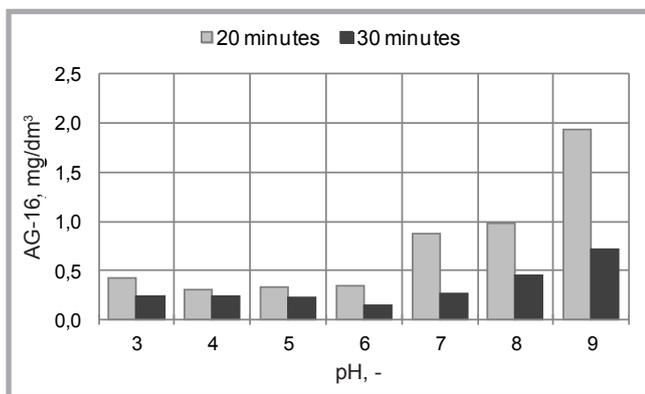


Figure 3. Changes in AG-16 concentrations at different pH values (LP lamp, H₂O₂ = 250 mg/dm³).

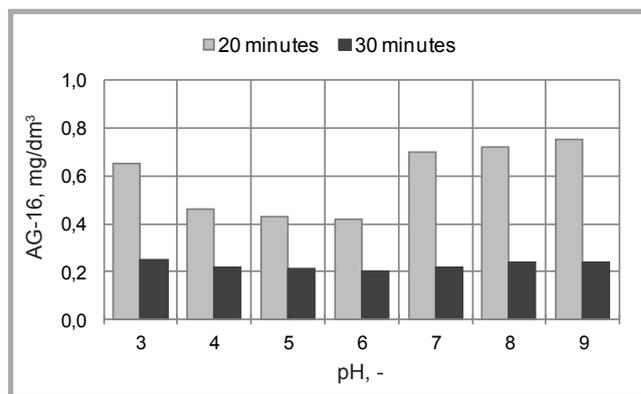


Figure 4. Changes in AG-16 concentrations at different pH values (MP lamp, H₂O₂ = 200 mg/dm³).

of 100-200 mg/dm³ (reaction time of 10 min) resulted in a reduction in AG-16 concentration to 9.99 mg/dm³ (90.1%) and 1.36 mg/dm³ (98.6%), respectively. For both types of lamps and the remaining periods of irradiation time (20 and 30 min), significantly better decolorisation effects were observed with an increase in the H₂O₂ dose, compared with the 10 min reaction time; however, for the MP lamp it was only in the range of 100-200 mg H₂O₂/dm³. The results showed that the reaction time had more impact on the UV/H₂O₂ process effectiveness than the dose of H₂O₂. In addition, for a 10 minute irradiation time, no visual decoloration of the test solutions was obtained, regardless of the dose of H₂O₂ and type of UV lamp used. On the other hand, extending the reaction time to 20 and 30 minutes resulted in a significant increase in AG-16 removal effects, even if the lowest doses of H₂O₂ were used. Decoloration of the solutions at 20 min reaction time was achieved at 250 and 200 mg H₂O₂/dm³ for LP and MP lamps, respectively. On the other hand, 30 min decoloration of the solutions was achieved at 200 and

150 mg H₂O₂/dm³, respectively, for LP and MP lamps. Based on the results obtained for further experiments (initial pH selection), 250 and 200 mg H₂O₂/dm³ for LP and MP lamps were selected, respectively.

Effect of pH

In the second stage of the study for both types of UV lamps, a similar tendency of changes in discoloration effectiveness was observed depending on the initial pH, H₂O₂ dosage and reaction time. Generally, in all cases, the use of initial pH values greater or less than pH 6 resulted in a deterioration or a lack of improvement in decolorising efficiency. For LP lamps in the pH range of 3-6, slight changes in AG-16 concentration were observed in the range of 0.30-0.42 and 0.15-0.24 mg/dm³ for 20 and 30 min, respectively. At pH 6 for the LP lamp and 250 mg H₂O₂/dm³, the AG-16 concentration decreased to 0.35 and 0.15 mg/dm³ for 20 and 30 min, respectively. On the other hand, increasing the pH above 6 resulted in a rapid increase in AG-16 concentration (**Figure 3**).

In the case of the MP lamp, the effect of the pH on the decolorization efficiency occurred mainly at 20 min of the reaction time. In the pH range of 3-6 the concentration of AG-16 decreased from 0.65 to 0.42 mg/dm³. At pH >6 and 20 min, a decrease in AG-16 removal efficiency was observed, as was achieved in the case of the LP lamp. In contrast, for a 30 min irradiation time, the AG-16 concentrations (0.2-0.25 mg/dm³) were almost similar throughout the pH range. At a dose of 200 mg H₂O₂/dm³ and pH 6, the concentration of AG-16 after 20 and 30 min of reaction decreased to 0.42 and 0.22 mg/dm³, respectively (**Figure 4**).

The rapid decrease in process efficiency above pH 6 could be due to the fast spontaneous decomposition of H₂O₂ to H₂O and O₂ under these conditions, and thus the formation of much fewer OH[•] radicals. Based on the results, it was found that for both lamps, the most favorable initial pH value was equal to 6. This choice was primarily due to economical reasons, since a lower amount of reagents was required to reach pH 6 compared to lower pH val-

Table 2. Changes in COD and toxicity of solutions in the UV/H₂O₂ process.

Parameter	Model solution	Low pressure lamp, LP	Medium pressure lamp, MP
Dose of H ₂ O ₂ , mg/dm ³	–	250	200
Toxicity unit, TU	3	6	3
COD, mg O ₂ /dm ³	111	47.4	40.6
COD removal, %	–	57.3	63.4

ues. In addition, a reaction time equal to 30 min was chosen for further studies as it provided a much greater degree of dye removal compared to 20 min.

COD and toxicity analysis

In the third stage of the study, an extended analysis of the AG-16 removal process including dye concentration, COD removal efficiency, and toxicity tests was conducted. The UV/H₂O₂ process was carried out at initial pH of 6, for 30 min reaction time and H₂O₂ 250 and 200 mg/dm³, respectively for LP and MP lamps. The analysis of COD changes showed that for both types of UV lamps, COD values of the aqueous solutions after the UV/H₂O₂ process decreased in comparison with the initial solution (Table 2). In the MP lamp reactor, better COD removal was achieved (63.4%, COD = 40.6 mg O₂/dm³) compared to the LP lamp reactor (57.3%, COD = 47.39 mg O₂/dm³). Despite the use of a lower H₂O₂ in the MP lamp reactor, a higher degree of removal of organic compounds, expressed as COD, was observed, which may indicate that the UV lamp power is one of the most important factors influencing the efficiency of the UV/H₂O₂ process. The results of toxicity tests showed that initial solutions of 100 mg/dm³ of AG-16 dye were classified as toxic [32] as the toxicity measured (TU) equal to 3 was in the range of 1-10 (Table 2). AG-16 degradation in the UV/H₂O₂ process using a UV lamp with an MP lamp (for predetermined optimum decolorization conditions) did not change the toxicity of the aqueous solution of the dye. On the other hand, in the case of the LP lamp system, the value of TU doubled compared to the initial dye solution. In both cases, these solutions could still be classified as toxic because the TU obtained was in the range of 1-10.

Despite the effective removal of AG-16 dye and the decolourisation of aqueous solutions, the toxicity of the solutions tested was not reduced. This was probably due to the formation of intermediates of AG-16 as a result of the dye decomposition. The possibility of toxic intermedi-

ate formation (that sometimes have even greater toxicity to primary substrates) during the decomposition of pollutants (estrogens and dyes, among others) when AOP methods are used is confirmed by the literature [33, 34]. Moreover Barbusiński [35] studied the toxicity of four types of industrial wastewater treated by Fenton's reagent and showed that the high efficiency of organic component degradation (as COD value) was not always followed by a reduction in toxicity to a very low level. In order to achieve the total reduction of toxicity in the Fenton process, it was necessary to increase both the H₂O₂ dose and reaction time.

Conclusions

- The best effect of the removal of AG-16 dye from aqueous solutions using a LP lamp (99.85%, concentration AG-16 = 0.15 mg/dm³) was obtained for H₂O₂ 250 mg/dm³, a reaction time of 30 min and initial pH of 6. In the case of the MP lamp, a similar effect (99.80% concentration AG-16 = 0.20 mg/dm³) was obtained for H₂O₂ 200 mg/dm³ and the same values for other parameters, i.e. reaction time and pH. In both cases a visual discoloration of the solutions was observed, while better removal of COD was achieved with the MP lamp (63.4%) compared to the LP lamp (57.3%).
- For the best effects of AG-16 removal, no toxicity reduction of the solutions was achieved, and in the case of the LP lamp, there was even a double increase in the value of TU relative to the initial solutions. In order to reduce the toxicity of the solutions, the H₂O₂ dose and/or the duration of the reaction time should be increased, and a UV reactor with MP lamp should be used.
- On the basis of the results, it can be concluded that in the case of the implementation of AOPs in specific wastewater treatment processes, these should be optimised not only for the removal of pollutants but also for toxicity reduction. This requires an extension of the scope of analysis, also for

toxicological tests. This is especially important when, for example, wastewater treated by AOP methods is directly discharged into natural waters.

- For particularly toxic wastewater, the possibility of introducing a wider range of toxicity tests should be considered, e.g. based on *Daphnia magna*, as it is highly sensitive to pollutants.

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