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Influence of microwave irradiation on hypochlorite decolorisation of synthetic dyes

ABSTRACT

In this study three synthetic dyes were treated using hypochlorite or combination of hypochlorite and microwave irradiation in order to investigate factors which determine decolorisation of the investigated compounds. An azo dye (C.I. Reactive Orange 16 – RO16), an azo methine dye (C.I. Basic Yellow 28 – BY28) and an anthraquinone dye (C.I. Acid Blue 111 – AB111) were used for experiments. Hypochlorite stability as well as the influence of microwaves on hypochlorite stability was also investigated.

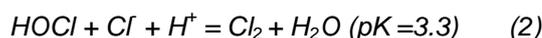
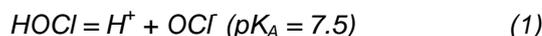
Keywords: Dye decolorisation, sodium chloride, azo dye, anthraquinone dye, azomethine dye, waste water, microwave reactor

1. INTRODUCTION

In many countries, as a result of production and use of dyes, the effluents of a variety of industries contain synthetic organic dyes. These industries include textile, leather, paper, food and hair coloring industry [1-5]. Dyes, which are colored, cause not only aesthetic pollution but can be serious health-risk factors [6,7]. Dyes can be classified by their application to substrate or by their chemical structure. The first classification includes direct, reactive, disperse, vat, azoic dyes etc. The second one includes azo, anthraquinone, nitro and nitroso, di – and triarylmethine, polyene and polymethine, carbonyl, sulfur dyes etc. [8,9]. Azo dyes represent the most important class of colorants. More than 50% of all dyes and pigments contain azo group. The second commercially important group belongs to anthraquinone dyes [10,11].

Dyes can be removed by chemical and physical processes, which include adsorption, filtration, ozonation, bleaching, oxidative degradation photochemical processes and electrochemical degradation [7,12-14]. Bleaching, as one of the process for

pollutant removal, includes use of oxidizing agents like sodium hypochlorite, hydrogen peroxide or peracids. Hypochlorite (HOCl) is the most aggressive and most efficient oxidant. It is very reactive towards dyes. The active species in bleaching are aqueous chlorine (at low pH) and undissociated hypochlorite (pH 4–10.5) [15]:



Microwave (MW) radiation is an electromagnetic radiation placed between infrared radiation and radio frequencies, with wavelength of 1 mm to 1 m. This corresponds to the frequencies of 300 GHz to 300 MHz, respectively. In order to avoid interferences with telecommunication devices, household and industrial microwave ovens are operated at either 12.2 cm (2.45 GHz) or 32.7 cm (915 MHz). Usually domestic microwave ovens operate at 2.45 GHz, and for that reason all commercial microwave reactors operate at the same frequency [16].

In comparison to conventional heating, MW irradiation has certain advantages. It exhibits higher heating rates, increased reaction rates, reduced activation energies, improved energy efficiency and reduced equipment size. MW irradiation is also selective and has excellent controllability, while application in sample preparation, organic and inorganic synthesis, food processing, sludge processing, medical waste treatment, contaminated soil remediation, wastewater treatment etc were observed [17-19].

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Organic pollutants which have been treated by microwave technology include dyes [20-22], phenolic compounds [23], pharmaceuticals [24], volatile organic compounds [25]. MW irradiation is usually combined with oxidants [17,26,27], or adsorbents [28]. Also different catalyst were used like nickel nanoparticles [29], fly ash zeolites [30], NiFe_2O_4 [31], polyaniline [32] and TiO_2 [33].

To the our best knowledge, until now, hypochlorite has not been combined with microwave irradiation. So, in this study, combined application of hypochlorite and microwave irradiation in the decolorisation of the synthetic dyes from the water solution were performed. As synthetic dyes, an azo dye (C.I. Reactive Orange 16 – RO16), an azo methine dye (C.I. Basic Yellow 28 – BY28) and an anthraquinone dye (C.I. Acid Blue 111 – AB111) were used. The structures of the studied dyes are given in Figure 1. The influence of microwaves on the hypochlorite stability was also investigated.

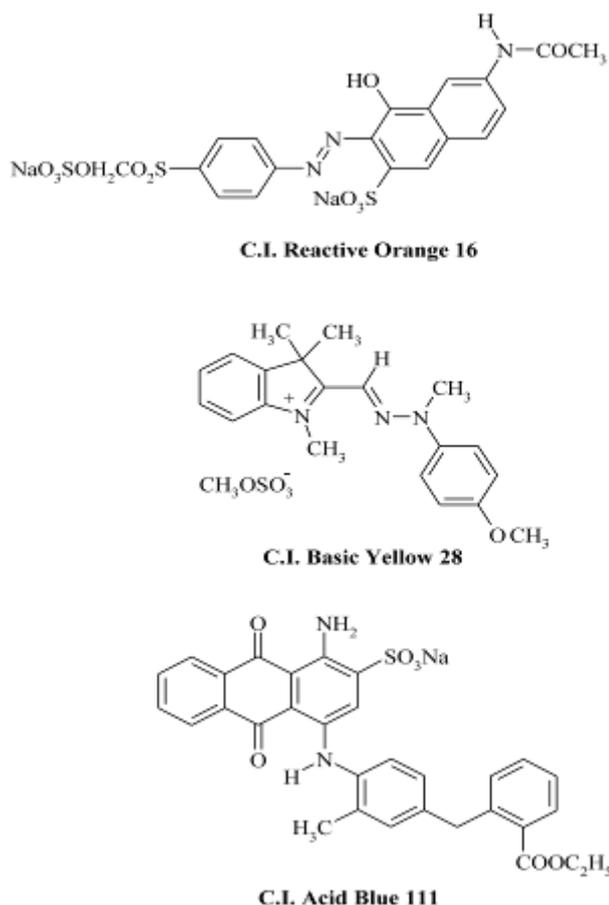


Figure 1 - The structures of the studied dyes

2. EXPERIMENTAL

2.1. Materials and methods

All starting materials were obtained from Aldrich and Fluka, and were used without further purification. The textile dyes were obtained as

follows: CI Reactive Orange 16 from Aldrich (dye content 50%); CI Basic Yellow 28 from Bezema (commercial name Bezacryl Goldgelb GL 200) and C.I. Acid Blue 111 from Hoechst (commercial name Fast Blue FGLL). The dyes were used without purification. Sodium chloride was p.a. grade (Merck). Deionised water was obtained from a Milipore Waters Milli-Q purification unit.

2.2. Preparation of hypochlorite

Preparation of hypochlorite was performed in the cylindrical glass electrochemical cell with electrolyte volume of 500 cm^3 . Electrolyte was prepared from deionised water and NaCl . As anode 5 cm^2 DSA Ti/PtOx , obtained by thermal decomposition of H_2PtCl_6 in 2-propanol with 1 mg cm^{-2} of platinum loading, was used, while cathode was 10 cm^2 plate made from austenite 18Cr/8Ni stainless steel series 304 [34]. Electrode with the gap of 3 mm was immersed at the top of the electrolyte. For the electrolysis of solution galvanostatic mode of PAR M273 potentiostat/galvanostat was used. Mixing of the electrolyte by magnetic stirrer was accomplished. During the electrolysis, at 5 min interval time 3 cm^3 of solution had been taken with pipette and UV-Vis spectra was instantly recorded. The concentration of the hypochlorite was followed by measuring the absorption of solution using UV-Vis spectrophotometer (Shimadzu model 1700 spectrophotometer). The concentration of hypochlorite was calculated using Faraday law [35].

2.3. Decolorization using hypochlorite

The decolorization process in thermostated cylindrical glass with reaction mixture volume of 100 cm^3 was investigated. The reaction mixture was prepared by mixing of dye solution with hypochlorite solution to obtain desired concentrations. Mixing of the reaction mixture was accomplished by magnetic stirrer at 600 rpm. During the decolorisation at certain times 3 cm^3 of solution was taken with pipette and UV-Vis spectra was instantly recorded. The concentration of dye was followed by measuring the absorption of solution using UV-Vis spectrophotometer.

2.4. Decolorization using hypochlorite under microwave irradiation

The decolorization process in a microwave reactor (Anton Paar Monowave 300) with reaction mixture volume of 6 cm^3 was investigated. The reaction mixture was prepared by mixing of dye solution with hypochlorite solution to obtain desired concentrations. The reaction mixture was stirred at 600 rpm and irradiated for a certain period of time at appropriate temperature. The progress of the reaction was followed by UV-Vis spectrophotometer.

3. RESULTS AND DISCUSSION

3.1. Hypochlorite generation

In the first part of the presented study, the sodium hypochlorite solution by electrolysis of NaCl was prepared, as described in Experimental part. The formation of hypochlorite was followed by UV-Vis spectrophotometer at 292 nm, and the concentration of hypochlorite solution was determined according to Faraday law (Figure 2) [35]. The obtained solutions were kept in dark bottles at 4°C.

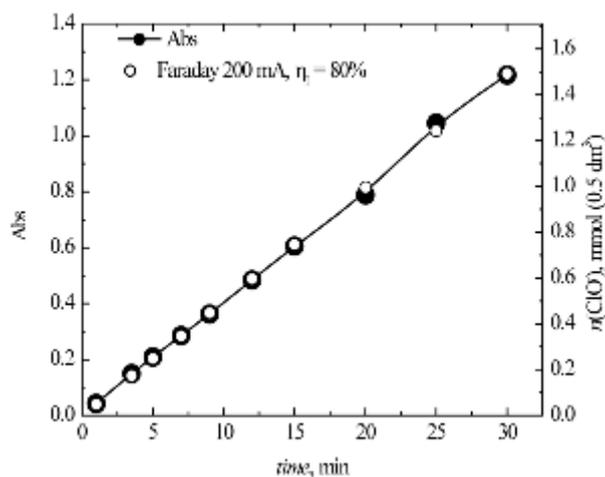


Figure 2 - Dependence of absorbance and hypochlorite concentration of electrolysis time (sodium chloride concentration = 20 g dm⁻³, current = 200 mA).

3.2. Stability of hypochlorite

Used NaOCl solution was prepared daily, since the results of hypochlorite stability (Figure 3) indicated that NaOCl solution was quite unstable.

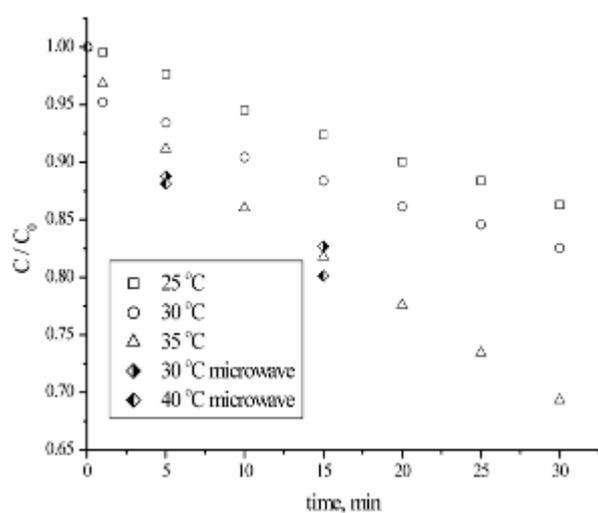


Figure 3 - Stability of hypochlorite at different temperatures (hypochlorite concentration = 0.375 mmol dm⁻³)

The main reason of instability is catalytic disproportionation with trace impurities of hypochlorite, especially at elevated temperatures, according to equation [36]:



The pH of solution was around 9 after electrolysis. Obviously, the diluted NaOCl solutions were degraded rapidly, and the degradation rate increased with temperature. Generally, around 12.5% of NaOCl was lost after 30 min at 25 °C, while at 35 °C more than 30% was degraded for the same period of time. Application of microwave irradiation did not have significant influence on the degradation of hypochlorite in comparison to conventional heating (around 12.5% after 5 min, and around 20% after 15 min).

3.3. UV-Vis spectra

The UV-Vis spectra changes, during decolorization of the studied dyes, are presented in Figure 4. Figure 4a shows UV-Vis spectra changes of RO16 during reaction with hypochlorite. The peak at 295 nm in the spectrum of the RO16 corresponds to the gamma acetylated acid structure, a precursor in RO16 synthesis. The peaks at 254 and 386 nm are indicative of benzene ring and azo group, respectively. At the end, due to an azo-hidrazone tautomerism in RO16 molecule, the peak at 493 nm corresponds to the hydrazone form of the dye [37]. The decolorization of RO16 dye can be easily observed by decrease of the absorption peaks at 386 and 493 nm.

The UV-Vis spectra of BY28 (Figure 4b) is characterized by three peaks: at 257, 300 and 436 nm. The first absorption band (at 257 nm) is due to the ($\pi \rightarrow \pi^*$) transition and is localized in the aromatic rings. The absorption band at 300 nm also corresponds to a ($\pi \rightarrow \pi^*$) transition. The third absorption band at 436 nm is assigned to a ($n \rightarrow \pi^*$) transition [38]. The decrease of absorption peak actually indicates a decolorization of BY 28 dye.

The UV-Vis spectra of AB111 (Figure 4c) is characterized by absorption peak at 634 nm (and a shoulder at 589 nm) which is result of amino groups presence. Amino groups as electron-donor cause an ($\pi \rightarrow \pi^*$) absorption band which is assigned to a charge transfer transition. The position of this band is the result of the conjugation of the 2p orbital of the nitrogen with the anthraquinone π electronic system [39]. The absorption band at 391 nm can be assigned to a ($n \rightarrow \pi^*$) transition. The absorption band at 254 nm corresponds to the ($\pi \rightarrow \pi^*$) transition (aromatic rings) as given before. The decrease of absorption peaks in the visible region indicates a decolorization of AB111 dye.

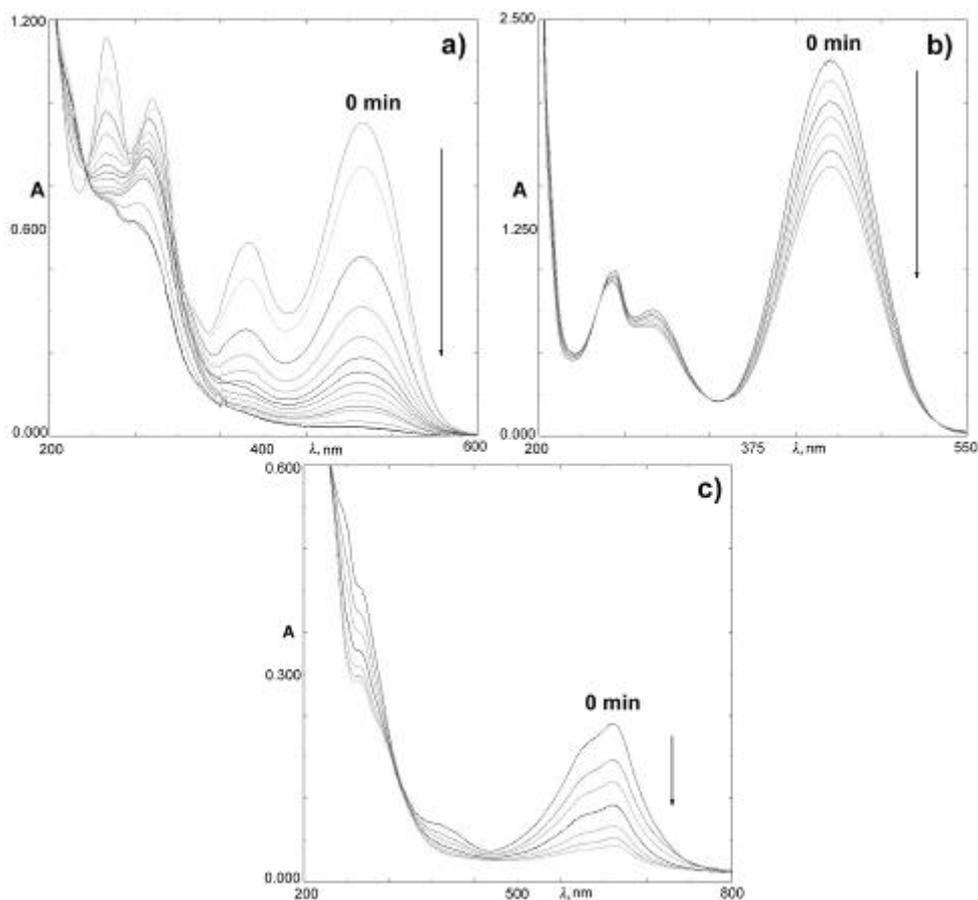


Figure 4 - a) UV-Vis absorption spectra of RO16 (25 °C, hypochlorite concentration = $0.375 \text{ mmol dm}^{-3}$), b) UV-Vis absorption spectra of BY28 (40 °C hypochlorite concentration = 1.5 mmol dm^{-3}), c) UV-Vis absorption spectra of AB111 (30 °C, hypochlorite concentration = $0.375 \text{ mmol dm}^{-3}$).

3.4. Dye decolorisation using only hypochlorite

It was mentioned earlier that azo-hidrazon tautomerism exists in RO16 molecule. The mechanism of decolorisation of one such molecule includes the conversion of hydrazone tautomer to hydrazone chloride ($\text{N}(\text{Cl})\text{-N}=\text{}$) which is considered to be the rate determining step of the reaction [40]. In addition, it was suggested that the rate-determining step is the electrophilic attack of the chloronium ion (Cl^+) on the nitrogen atom in the hydrazone tautomer [41]. The reaction then proceeds by OCl^- attack on the imino group of hydrazone chloride which gives single bond between two nitrogens. The other mechanism was also proposed which includes attack of deprotonated hydrazone form (N^-) on hypochlorous acid giving Cl^- and hydrazone hydroxide ($\text{NOH-N}=\text{}$). Hydrazone hydroxide then reacts with hydroxide anion which produces asymmetrical cleavage of azo bond [15]. Interruption of conjugation in molecule leads to the decolorization. Figure 5a shows the influence of reaction temperature on decolorisation. As it can be seen, decolorisation of around 90% can be achieved at 25 °C after 15 min,

while an increase in temperature increases the decolorisation rate and slightly increases the percentage of decolorisation.

The similar behavior was observed when AB111 dye was concerned, but the decolorisation was less effective. It is well known that 1,4-diaminoanthraquinone exists as amino-imino tautomers [42]. This implies that here the reaction can also proceed by the electrophilic attack of the chloronium ion on the nitrogen atom. However, due to the structure of anthraquinone molecule (condensed rings) AB111 is less susceptible to hypochlorite oxidation in comparison to RO16. Here, also an increase in temperature increases the decolorisation rate. The achieved percentage of decolorisation increases from ~60% to more than 80% (Figure 5b).

The lowest reactivity of the studied dyes was observed in the case of BY28 decolorisation. This dye is azomethine dye and, as such, is less reactive since the electrophilic attack of the chloronium ion on the nitrogen atom in BY28 is hindered by the conjugation which exists in the molecule (Figure 1).

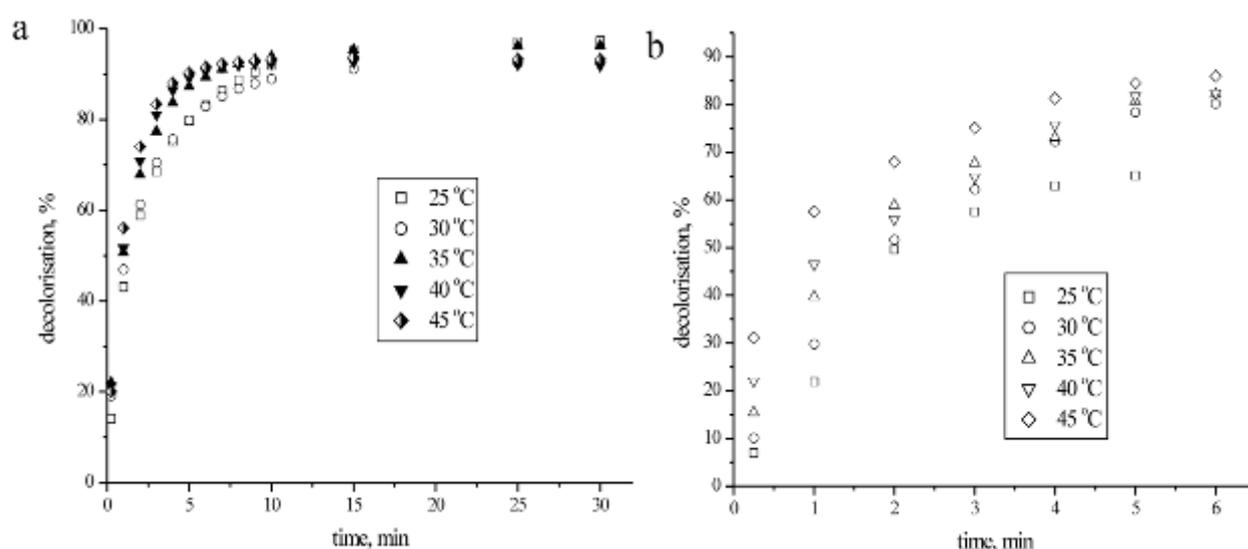


Figure 5 - Temperature effect on decolorisation of RO16 (a) and AB111 (b) dyes using only hypochlorite (dye concentration = $97 \mu\text{mol dm}^{-3}$, hypochlorite concentration = $0.375 \text{ mmol dm}^{-3}$)

Due to this low reactivity of BY28 [38], only reaction at 40°C was recorded and compared to RO16 and AB111 decolorisation under the same reaction conditions (Figure 6). It can be seen that the reactivity order is in accordance with the aforementioned, i.e. RO16>AB111>BY28.

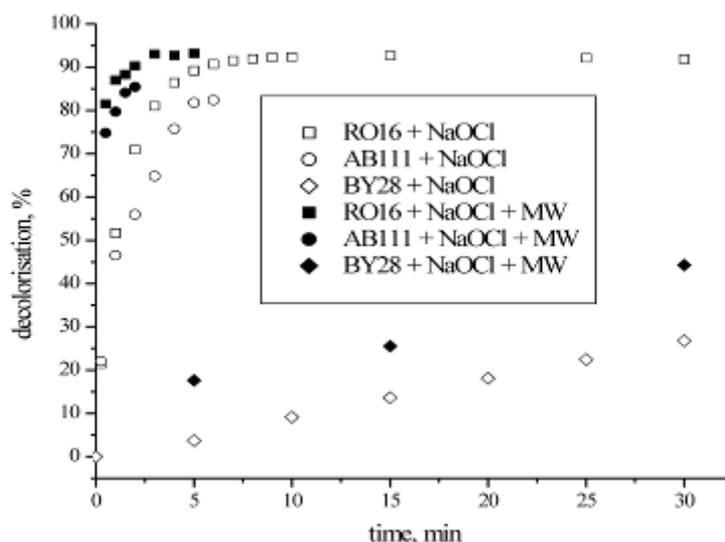


Figure 6 - Influence of dye structure on decolorisation using hypochlorite (empty symbols) and hypochlorite and microwave irradiation (full symbols) at 40°C (hypochlorite concentration = $0,375 \text{ mmol dm}^{-3}$ (RO16 i AB111) and 1.5 mmol dm^{-3} (BY28), dye concentration = $97 \mu\text{mol dm}^{-3}$).

3.5. Dye decolorisation using hypochlorite and microwave irradiation

In the final part of this research, influence of microwave irradiation on decolorisation of investigated dyes was studied (Figure 6). The microwave irradiation alone of aqueous solution of the studied dyes had no effect unless combined with hypochlorite. Microwave irradiation in combination with hypochlorite increases the reaction rate and

efficiency of decolorisation. This effect is smaller in the case of RO16 dye but much pronounced in the case of AB111 dye. The microwave effect was also observed in the case of BY28 decolorisation. It was shown in the previous section that thermal effect was observed only when hypochlorite was used. Here, we have seen that the combination of microwave irradiation and hypochlorite enhanced the decolorisation process. This enhancement was

observed in each microwave treatment of the studied dyes. The microwaves can have thermal and nonthermal (or specific) effects on the reaction. Thermal effects are caused by the heating rate, superheating, "hot spots" and the selective absorption of irradiation by polar substances [43]. So, the thermal and nonthermal effects were reported in the microwave degradation of Rhodamine-B photo-degradation [44]. As nonthermal effect is quite hard to prove [45], it can be presumed that only thermal effect of microwave irradiation influence the rate and decolorisation efficiency of the used treatment. At the end, it should be pointed out that there is no difference in reactivity order of dyes in the reaction with pure hypochlorite or hypochlorite/microwave combination (Figure 6).

4. CONCLUSION

The sodium hypochlorite solution was prepared by electrolysis of NaCl and the concentration of hypochlorite solution was determined according to Faraday law. The diluted NaOCl solutions were degraded rapidly, and the degradation rate increased with temperature. Generally, around 12.5% of NaOCl was lost after 30 min at 25 °C while at 35 °C more than 30% was degraded for the same reaction time. Application of microwave irradiation did not have significant influence on the degradation of hypochlorite in comparison to conventional heating. The reactivity order of the studied dyes was in accordance with the structure of dyes and their susceptibility to hypochlorite oxidation, i.e. RO16>AB111>BY28. The microwave irradiation alone of aqueous solution of the studied dyes have no effect unless combined with hypochlorite. Microwave irradiation in combination with hypochlorite increases the reaction rate and efficiency of decolorisation. This effect is smaller in the case of RO16 dye but much pronounced in the case of AB111 dye. The microwave effect was also observed in the case of BY28 decolorisation. It was shown that thermal effect was observed when only hypochlorite was used. The enhancement of the decolorisation process was observed in each microwave treatment of the studied dyes. This difference is due to the microwave thermal effect on the rate and decolorisation efficiency of the used treatment. It should be pointed out that there is no difference in reactivity order of dyes in the reaction with pure hypochlorite or microwave/hypochlorite combination.

Acknowledgement

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IZVOD

UTICAJ MIKROTALASNOG ZRAČENJA NA OBEZBOJAVANJE SINTETSKIH BOJA HIPOHLORITOM

U okviru rada, tri sintetske boje tretirane su rastvorom hipohlorita ili kombinacijom rastvora hipohlorita i mikrotalasnog zračenja, da bi se ispitaio uticaj različitih faktora na obezbojavanje ispitivanih boja. Jedinjenja koja su korišćena u ispitivanju pripadaju azo (C.I. Reactive orange 16 – RO16), azometinskim (C.I. Basic yellow 28 – BY28) i antrahinonskim bojama (C.I. Acid Blue 111 – AB111). takođe je ispitivana stabilnost hipohlorita kako pri zagrevanju tako i pri mikrotalasnom zračenju.

Ključne reči: Obezbojavanje boja, natrijum-hlorid, azo boje, antrahinonske boje, azometinske boje, otpadna voda, mikrotalasni reactor.

Naučni rad

Rad primljen: 17.10.2015.

Rad prihvaćen: 30.11.2015.

Rad je dostupan na sajtu: www.idk.org.rs/casopis