



## Combination of hydrodynamic cavitation with oxidants for efficient treatment of synthetic and real textile wastewater

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### ABSTRACT

In this study, the degradation of direct red 89 (DR89) dye from synthetic wastewater was assessed by lab-scaled hydrodynamic cavitation (HC) combined with different oxidants such as periodate (PI), persulfate (PS), and oxalic acid (OA). The degradation tests were carried out by combining the three oxidants in the absence of HC, and using HC with only one of the oxidants (HC/OA, HC/PS, and HC/PI) or with all combined (HC/PS/OA/PI). In all processes, the degradation constants followed first-order reaction kinetics, being the highest one that of the HC/PS/OA/PI process, equal to  $2.01 \times 10^{-1} \text{ min}^{-1}$ . The degradation of DR89 was affected by pH, initial dye concentration, and reaction time. The degradation efficiency increased when pH decreased obtaining the best results at pH 3.0 due to favorable conditions for the generation and persistence of radical species. The degradation of the dye improved with increasing dose of OA, PS and PI. The best results were achieved using dosages of OA, PS, and PI of 100.0, 125.0, and 150.0  $\text{mg L}^{-1}$ , respectively. The quenching experiments revealed the presence of  $\cdot\text{OH}$ ,  $\text{SO}_4^{\cdot-}$ ,  $\text{IO}_3^{\cdot-}$ ,  $\text{O}_2^{\cdot-}$ , and  $^1\text{O}_2$  in the HC/PS/OA/PI process, while  $\cdot\text{OH}$ ,  $\text{SO}_4^{\cdot-}$  and  $\text{IO}_3^{\cdot-}$  were the dominant responsible radicals. The rate constants of the individual processes were used to calculate the value of the synergetic coefficient of HC/PS/OA/PI process. At the optimum conditions, the calculated synergetic coefficient was 15.39. The effectiveness of the HC/PS/OA/PI process in the removal of color and COD from real textile wastewater was also investigated and exhibited the  $96.23 \pm 1.92\%$  and  $93.84 \pm 0.94\%$  reduction of color and COD after 70 min reaction time, respectively. The results showed that the combined process can be a useful technology for the treatment of textile wastewater.

### 1. Introduction

In recent decades, with the population increasing and the development of industry, pollution has also significantly increased. Industrial wastewater contains hazardous substances, and consequently it must be treated before discharging it into the environment. Dyes are one of the most extended toxic chemicals found in industrial wastewater from different origin. The importance of removing dyes can be even greater than that of other colorless materials in wastewater because most of them contain azo bonds ( $-\text{N}=\text{N}-$ ), sulfonic groups, and aromatic structures which are mutagenic, teratogenic, and carcinogenic even at

low concentration levels [1,2]. Other problems produced by the presence of dyes in water include aesthetic nature, disruption of the transfer of sunlight into water bodies, decrease photosynthetic function, and thus a threat to aquatic life. In addition, these substances may also enter the food chain and threaten human health [3–5]. Conventional treatment processes such as coagulation, adsorption, chemical oxidation, membrane separation, and biological process are able to treat the toxic, non-biodegradable, and persisting nature of the dye. In this sense, more appropriate, lower-cost, and/or simpler treatment techniques are necessary for the effective treatment of azo dyes [6].

Among numerous available treatment processes, the advanced

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oxidation processes (AOPs) are being widely studied for the degradation of different toxic pollutants from wastewater. They involve the generation of reactive oxygen species (ROS), such as hydroxyl radicals ( $\bullet OH$ ) with an oxidation potential ( $E^0$ ) of 2.8 eV, that are non-selective species with high oxidation potentials [7,8]. One of the alternatives is hydrodynamic cavitation (HC), which is considered a good choice since it is environmentally friendly, easy to operate, economical and scalable, and particularly suitable in the treatment of industrial wastewater [9,10]. HC is produced by a cavitation device such as a venturi device, pump, jet nozzle, propeller, orifice plate, and so on. The formation, growth, and collapse of vapor cavities (bubbles) are obtained through the generating of low-pressure areas in a solution, with vapor pressures lower than that of the liquid [11–13]. The collapse of the cavity generates local extremely high pressures (10–500 MPa) and temperatures (1000–15,000 K) so-called “hot spots”, as well as intense turbulence [14]. Under such extreme conditions free radicals like  $\bullet OH$ ,  $H^*$ , and  $H_2O_2$  are produced due to the decomposition of water vapor (Eqs. (1) and (2)) and non-condensable gases inside the cavities. These species can produce the oxidation of dissolved pollutants [12,15].



HC process has been successfully used for the treatment of industrial wastewater, emerging pollutants like ibuprofen and para-nitrophenol, water disinfection [16]. The efficiency of HC can be improved using hybrid treatment systems where HC is combined with other oxidants. This approach can increase the degradation rate, reducing the reaction time [17]. Sulfate radical-based advanced oxidation processes (SR-AOPs) have recently gained increasing attention due to the longevity, high stability, and high reactivity of sulfate radicals ( $SO_4^{\bullet -}$ ;  $E^0 = 2.6$  V) [18]. Moreover, several studies confirmed that SR-AOPs can be used for the degradation of various non-biodegradable contaminants. Persulfate (PS) ( $S_2O_8^{2-}$ ) anions can be activated by UV [19] or ionizing irradiations [20], strong alkaline conditions [21], high temperatures [22], ultrasound (US) or acoustic cavitation [23,24], HC process [25] and so on. In this study, the heat generated in HC can activate PS, which saves energy and chemicals. Periodate (PI) is another well-known strong oxidant ( $E^0 = 1.60$  V). Under UV irradiations [26] or in the presence of US waves [27], it can be converted into highly reactive intermediates, such as  $IO_4^*$ ,  $IO_3^*$ ,  $\bullet OH$ , and  $O^*$ , which can oxidize the organic pollutants dissolved in water. Although PI is potentially an effective and selective oxidant, its activation is difficult because it requires special equipment and high energy consumption [28]. To the best of our knowledge, no study has previously reported the use of HC to activate PI. On the other hand, some studies have been conducted to evaluate the effect of addition of oxalic acid (OA) on the treatment efficiency [29,30]. Kwan and Chu [31] reported that OA addition to the Fenton process leads to faster and higher efficiency of oxidation process. Furthermore, the reaction of oxalate with the dissolved oxygen in the solution can produce superoxide radicals, and consequently, hydrogen peroxide and  $\bullet OH$  radicals [31].

No research has been devoted to investigating the characteristics of the degradation process induced by the HC/OA/PS/PI combined process. Hence, the goal and novelty of this study is the analysis of the effects of HC, OA, PS, and PI separately and combined on the kinetics of DR89 degradation at different operating conditions (reaction time, solution pH, oxidant concentration, and DR89 initial concentration). In the present study we try to combine different oxidants and supporting compounds in order to enhance the degradation efficiency and achieve high mineralization degrees. In addition, the combination of these oxidants decreases the reaction time and the total required dose of the oxidants. In addition, we analyzed also the degradation of both synthetic and real dye wastewater by systematically optimization of dose of each oxidant.

## 2. Materials and methods

### 2.1. Chemicals and reagents

DR89 dye (a double azo class,  $C_{44}H_{32}N_{10}Na_4O_{16}S_4$ , 1177 g/mol, CAS No: 12217-67-3, and  $\lambda_{max}$ : 494 nm) was obtained from Baharjin textile factory (Iran). Sodium peroxydisulfate ( $Na_2S_2O_8$ , CAS No: 7775-27-1, purity  $\geq 98$  %), oxalic acid ( $H_2C_2O_4$ , CAS No: 144-62-7, purity  $\geq 98$  %), and sodium (meta)periodate ( $NaIO_4$ , purity  $\geq 99.8$  %) were all analytical reagent grades received from Merck Co. (Germany). The pH of the solution was adjusted using NaOH and HCl solutions (0.1 N) (Merck Co.). All the solutions were prepared with deionized water.

### 2.2. Real textile wastewater

In this study, the wastewater of textile industry was collected from the receiving tank in the treatment plant of a textile industry (Isfahan, Iran). The main characteristics of textile wastewater are summarized in Table S1.

### 2.3. HC experimental set-up

The schematic of the HC reactor set-up used in the present study is shown in Fig. S1 [32]. It consists of a feed tank with 2 L of capacity, a centrifugal pump of power rating 0.37 KW, a pressure gauge to measure the effluent pressure, a venturi device, and a flow meter. The suction side of the centrifugal pump is connected to the bottom of a feed tank and the feed solution was recycled through the line using a circulating pump (Pentax, Italy). The inlet pressure and the recovered pressure were measured using pressure gauges. The venturi device was made of brass metal and installed in the mainline acting as a cavitation device had an inner diameter of the throat was 0.5 mm and its total length was 130 mm with the contraction and expansion angles both  $20^\circ$ . To maintain the feed temperature within 25–29 °C, venturi device was installed in the cooling water path. This operation was done by circulating cold water.

### 2.4. Experiment procedure and analytical methods

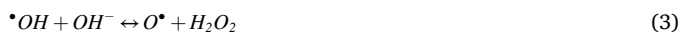
The prepared DR89 solution was introduced into the feed tank. The DR89 degradation was initially evaluated using different processes, namely non-combined HC, OA, PS, and PI oxidations, and combined, including HC/OA, HC/PS, HC/PI, PS/OA/PI, and HC/PS/OA/PI. Then, the effect of various parameters including solution pH (3.0–10.0), initial DR89 concentration (50–100 mg L<sup>-1</sup>), reaction time (5–120 min), OA dose (50.0–150.0 mg L<sup>-1</sup>), PS dosage (50.0–150.0 mg L<sup>-1</sup>), and dose of PI (50.0–150.0 mg L<sup>-1</sup>) on DR89 degradation efficiency were systematically evaluated. A pH adjustment was made at the beginning of each experiment using HCl and NaOH and monitored during the experiment, but not controlled. A stock solution of oxidants was prepared each day and a specified value was added to the feed solution. To determine the dominant free radicals in DR89 degradation, the experiments were carried out with separate addition of 15 mM of *tert*-butanol (TBA), ethanol (EtOH), phenol, p-Benzoquinone (p-BQ), and sodium azide ( $NaN_3$ ). In this study, the efficiency of the HC reactor in removing dyes and chemical oxygen demand (COD) from textile wastewater was also measured. All the degradation tests were performed in triplicate and obtained results were represented as Ave  $\pm$  STD. At the ends of experiments, 1 mL of  $Na_2S_2O_3$  solution (5.0 M) were added to the solution in order to stop degradation reactions and DR89 concentration was quantified with a visible spectrophotometer (Milton Roy Company 20D) at a wavelength ( $\lambda_{max}$ ) of 494 nm. The solution pH and conductivity, chemical oxygen demand (COD), and total suspended solid (TSS) were measured using a multi parameters device (HQ40D portable pH and conductivity meter, HACH Company, Germany), closed reflux colorimetric method, and gravimetric method [33].

### 3. Results and discussion

#### 3.1. Effect of solution pH

HC-assisted degradation of DR89 was performed at different initial solution pH with values ranging from 3.0 to 10.0, and maintaining a DR89 initial concentration of  $75.0 \text{ mg L}^{-1}$ , an inlet pressure of 2 bar, and doses of PS, OA, and PI adjusted to  $50.0 \text{ mg L}^{-1}$  in each experiment (Fig. 1).

It was observed that the extent of degradation increased with decreasing initial pH and the highest degradation percentage of DR89 was achieved at solution pH of 3.0 regardless of the process employed. It is also confirmed the best behavior of the HC/PS/OA/PI combined process in all the pH range analyzed. Lowering the pH from 10.0 to 3.0 increased the extent of DR89 degradation by an average of 3.5-fold, so subsequent experiments were performed at pH 3.0. This better behavior at acidic pH can be explained as follows: under acidic conditions, the formation of  $\bullet\text{OH}$  radicals by the HC is favored and the oxidation potential of these  $\bullet\text{OH}$  is higher. However under basic conditions, the recombination of free radicals leads to the formation of  $\text{H}_2\text{O}_2$  (Eq. (3)), which has a lower oxidation capacity [34]. This deactivation of  $\bullet\text{OH}$  radicals with hydroxyl ions is more important when the pH of the solution is higher. In addition, the self-decomposition rate of hydrogen peroxide (Eq. (4)) increases under alkaline conditions, leading to the decrease in the amount of  $\text{H}_2\text{O}_2$  available in the solution, which further supports the observed reduction in degradation observed at increasing initial pH values [35].



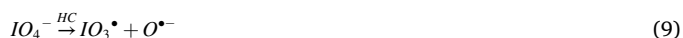
In addition, considering that the  $pK_a$  value of DR89 is 4.95 [32], the state of DR89 molecules at solution pH equal to 3.0 is neutral, and consequently hydrophobic, allowing them to be transported more rapidly toward the bubble interface of the collapsing cavities. On the other hand, under basic conditions (at pH higher than the  $pK_a$ ), DR89 molecules are predominantly in anionic state, becoming more hydrophilic and remaining in the bulk solution, and not at the interface between the cavity and water. Since only a small proportion of the generated  $\bullet\text{OH}$  radicals can be dispersed in the bulk solution, the degradation decreases [36].

The different oxidants also show particular characteristics at different pH values. In the case of PS, at pH 3 it can also react with electron ( $e^-$ ) to form  $\text{SO}_4^{\bullet-}$  (Eqs. (5) and (6)) [37], which increases the

amount available of this highly oxidative radical to degrade DR89. Thus, under acidic conditions,  $\text{SO}_4^{\bullet-}$  is the predominant radical specie and more reactive than  $\bullet\text{OH}$  [38]. In contrast, at alkaline conditions, the main radical is  $\bullet\text{OH}$  due to reactions between activated PS anions and  $\text{OH}^-$  (see Eq. (7)), so the degradation decreases because  $\bullet\text{OH}$  has a lower oxidation potential than  $\text{SO}_4^{\bullet-}$  [39]. Moreover, at basic conditions  $\bullet\text{OH}$  radicals can recombine to produce  $\text{H}_2\text{O}_2$  (see Eq. (8)), which in turn reduces even more the extent of the degradation of the dye, supporting the results represented in Fig. 1. These findings are in agreement with the previously reported by Monteagudo et al. and Wang and Zhou [40,41], who used sono-activated sulfate to treat diclofenac and carbamazepine, respectively, and whose optimal degradation was achieved at acidic pH.



In the case of oxidation with PI, it exists in form of  $\text{IO}_4^-$  in acidic medium, but it transforms to less reactive  $\text{H}_2\text{I}_2\text{O}_4^{4-}$  with increasing pH, which justifies the lower degradation of DR89 under alkaline conditions with this oxidant. In addition, at low pH, the formation of several highly reactive intermediate radicals such as  $\text{IO}_3^{\bullet}$ ,  $\bullet\text{OH}$ , and  $\text{IO}_4^{\bullet}$  (Eqs. (9)–(11)) justifies the increase of the DR89 degradation in the presence of periodate compared to that in alkaline conditions [42]. Yu-chi et al. [43] also used sonochemically activated periodate to remove perfluorooctanoic acid, and their results were consistent with those of this study. They obtained the highest removal (62.4 %) at an acidic pH of 3.9. When OA are present in the solution, OA can react with the oxygen molecule to produce superoxide radicals ( $\text{O}_2^{\bullet-}$ ) that lead to the production of  $\text{H}_2\text{O}_2$  [30], so, improve the degradation efficiency. In addition, OA can decrease the pH of the solution as a favorable condition for formation of  $\bullet\text{OH}$  radicals by the HC process.



#### 3.2. Effect of initial DR89 concentration and reaction time

In order to investigate the effect of the initial DR89 concentration and the reaction time on the degradation efficiency of DR89, different experiments were performed with initial concentrations of 50.0, 75.0 and  $100.0 \text{ mg L}^{-1}$  at a pH of 3.0 and with different oxidants. Figs. 2 and S2–S5 present the degradation profiles of DR89 by HC/PS/OA/PI process and different combined process, respectively. In all the processes the degradations of DR89 at a concentration of  $50.0 \text{ mg L}^{-1}$  were greater than those at  $100.0 \text{ mg L}^{-1}$ . The percentage of DR89 degradation at  $50.0 \text{ mg L}^{-1}$  using HC/PS, HC/OA, HC/PI, HC/PS/OA, and HC/PS/OA/PI processes were  $73.74 \pm 1.47 \%$ ,  $92.31 \pm 1.85 \%$ ,  $94.74 \pm 2.84 \%$ ,  $75.61 \pm 2.27 \%$ , and  $98.21 \pm 0.98 \%$ , respectively, after 130 min of reaction time. With increasing the initial DR89 concentration to  $100.0 \text{ mg L}^{-1}$ , the degradation efficiency of DR89 by mentioned process reduced by 17–22 % due to depletion of the availability of oxidizing species and also the formation of reaction intermediates increases, so the competition for the oxidizing species increases [44,45]. The lower degradation of DR89 by HC/PS/OA may be due to the fact that the pH of the solution is reduced with OA, allowing hydrogen ions ( $\text{H}^+$ ) to accumulate and act as scavengers of  $\text{SO}_4^{\bullet-}$  and  $\bullet\text{OH}$  radicals as described in the Eqs. (12) and (13) [46].

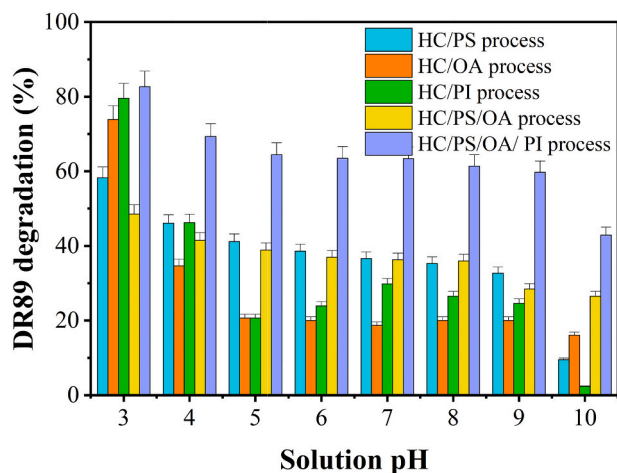


Fig. 1. Effect of solution pH on degradation of DR89 by different process. Experimental conditions:  $[\text{DR89}]_0 = 75.0 \text{ mg L}^{-1}$ ,  $[\text{PS}]_0 = [\text{OA}]_0 = [\text{PI}]_0 = 50.0 \text{ mg L}^{-1}$ , pH = 3.0–10.0, and reaction time = 30 min.

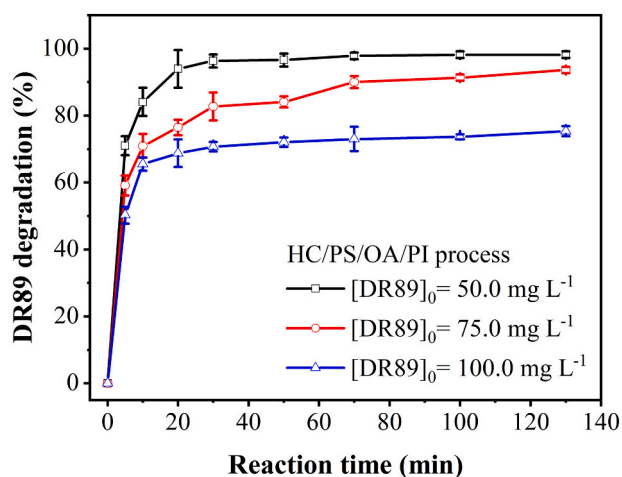


Fig. 2. Effect of reaction time on degradation of DR89 by HC/PS/OA/PI process. Experimental condition:  $[DR89]_0 = 50.0\text{--}100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = [OA]_0 = [PI]_0 = 50.0 \text{ mg L}^{-1}$ ,  $\text{pH} = 3.0$ , and reaction time = 5–130 min.



The degradation of DR89 increased more rapidly in the first few reaction minutes and then it almost stopped. This can be explained by the fact that in the first few minutes and in the presence of large amounts of oxidizing agents, decomposition occurs at a high rate, and after that the oxidizing agents have destroyed the dye molecules, the rate of decomposition remains almost constant [37]. It seems that the equilibrium time for the degradation experiments is ca. 70 min and this reaction time was selected for other experiments in order to save time and optimize energy consumption.

### 3.3. Effect of OA concentration

The effect of OA dosage (50.0 to 150.0  $\text{mg L}^{-1}$ ) was studied in HC/PS/OA/PI, HC/OA, and HC/PS/OA processes. The doses of PS and PI were fixed at 50.0  $\text{mg L}^{-1}$ , the initial concentration of DR89 was set at 100.0  $\text{mg L}^{-1}$  and the pH of the solution was adjusted to 3.0 as the optimal one. The effect of OA dosage on the dye degradation of combined processes are displayed in Figs. 3 and S6 and S7.

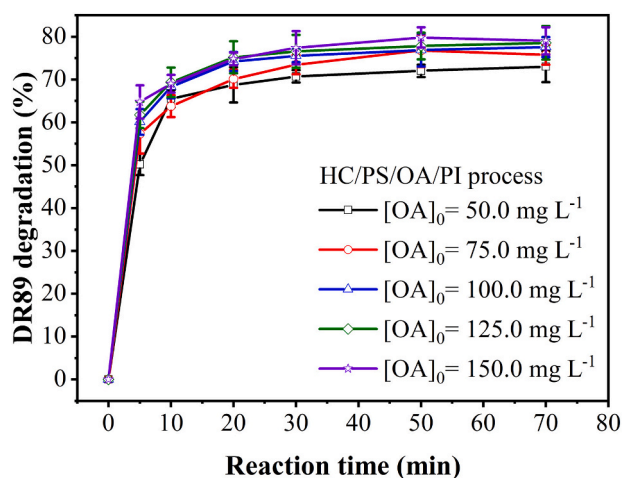


Fig. 3. Effect of addition of OA on degradation of DR89 by HC/PS/OA/PI process. Experimental conditions:  $[DR89]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = [PI]_0 = 50.0 \text{ mg L}^{-1}$ ,  $[OA]_0 = 50.0\text{--}150.0 \text{ mg L}^{-1}$ ,  $\text{pH} = 3.0$ , and reaction time = 70 min.

As can be seen, with increasing the OA dose from 50.0 to 100.0  $\text{mg L}^{-1}$ , the DR89 degradation by HC/PS/OA/PI, HC/OA, and HC/PS/OA processes improved from  $73.01 \pm 3.65 \%$  to  $77.59 \pm 2.33 \%$ ,  $72.72 \pm 3.64 \%$  to  $78.77 \pm 2.36 \%$ , and from  $51.40 \pm 2.57 \%$  to  $55.48 \pm 2.22 \%$ , respectively. This behavior is presumably related to a higher the generation of oxalate radicals ( $C_2O_4^{\bullet-}$ ),  $H_2O_2$ ,  $\bullet OH$  and superoxide ( $O_2^{\bullet-}$ ) during oxidation processes according to Eqs. (14)–(16) [29,30]. In addition, the present of OA in the solution led to decreasing solution pH, which favor the generation of active radicals like  $\bullet OH$ ,  $SO_4^{\bullet-}$ ,  $IO_3^{\bullet}$ , and  $IO_4^{\bullet}$  by HC process [42]. On the other hand, presence of OA in the acid solution can be attributed to its co-existing anionic role and presence of anions can improve the treatment efficiency of AOPs [32,47].



Further increase of OA dose above 100.0  $\text{mg L}^{-1}$  did not significantly enhance the degradation of DR89 using combined process. In overall, the increase of OA dose from 100.0 to 150.0  $\text{mg L}^{-1}$  enhanced the degradation by about 2%. This may be related to retardation of intermediate species decomposition at excess OA dose and also to the decomposition of  $C_2O_4^{\bullet-}$  radicals based on the Eq. (17) [30]. On the other hand, due to the organic nature of OA, the excess concentration of OA in the solution can compete with DR89 molecules for the available oxidizing species.



### 3.4. Effect of PS dose

The effects of PS dose (50.0–150.0  $\text{mg L}^{-1}$ ) in different combined processes (HC/PS, HC/PS/OA, and HC/PS/OA/PI) were also studied at constant dose of OA (100.0  $\text{mg L}^{-1}$  determined by previous experiments) and PI dose of 50.0  $\text{mg L}^{-1}$  and pH of 3.0. Figs. 4 and S8 and S9 depicts the influence of PS dose on degradation of DR89 by HC/PS/OA/PI process and other combined process, respectively.

As appears in Fig. 4, the PS dose significantly affects the decomposition of the dye molecules. When dosage of PS increased from 50.0 to 125.0  $\text{mg L}^{-1}$ , the DR89 degradation of HC/PS/OA/PI process promptly enhanced from  $77.59 \pm 2.33 \%$  to  $91.65 \pm 3.67 \%$ . The PS anion is classified as a strong oxidant ( $E^\circ = 2.01 \text{ V}$ ) that can be activated by light,

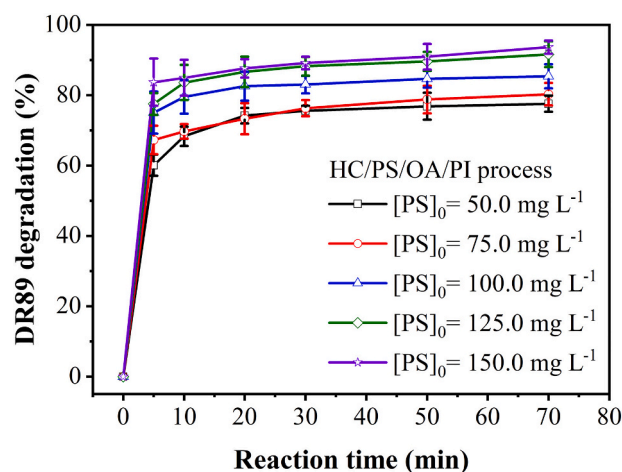
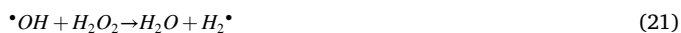


Fig. 4. Effect of PS as oxidant on degradation of DR89 in HC/PS/OA/PI process. Experimental conditions:  $[DR89]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 50.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 50.0\text{--}150.0 \text{ mg L}^{-1}$ ,  $\text{pH} = 3.0$ , and reaction time = 70 min.

heat or by other agents to generate the  $SO_4^{\bullet-}$  radicals ( $E^\circ = 2.6$  V) and as a results, the  $SO_4^{\bullet-}$  radicals is used as a treatment method for organic contaminants because of its long lifetime and high solubility [48]. In our case, the PS anion in the HC/PS/OA/PI process is activated by the heat generated by HC yielding  $SO_4^{\bullet-}$  (see Eqs. (1) and (18)). In addition, other reactive species like  $\bullet OH$  and  $H^\bullet$  radicals (see Eqs. (19)–(22)) and also non-radical species such as  $H_2O_2$  (Eq. (8)) can be produced and, consequently, facilitate the degradation of the dye molecules.



The increase of the PS dose above  $125.0 \text{ mg L}^{-1}$  did not significantly increase the degradation of the dye with the HC/PS/OA/PI process. A similar trend was reported by Choi et al. [25], who studied the decomposition of pentachlorophenol with HC, ferrous, and PS anions and demonstrated that an excess of PS anions no longer increases of pentachlorophenol degradation obtained. An overdose of PS anions could accelerate radical-scavenging because extreme  $SO_4^{\bullet-}$  could react with  $S_2O_8^{2-}$  (Eqs. (23) and (24)) and recombine to form  $S_2O_8^{2-}$  and  $SO_4^{2-}$  according to Eq. (25) [25,37]. Obviously, an appropriate dosage of PS would maintain the high degradation of DR89 while avoiding material waste.



### 3.5. Effect of PI dose

The initial concentration of DR89 was set at  $100.0 \text{ mg L}^{-1}$  and the dosage of PI was varied in the range of  $50.0$ – $150.0 \text{ mg L}^{-1}$  at a pH of 3.0. At this stage, the dose of PS and OA were equal to their optimal concentrations from the previous steps,  $125.0 \text{ mg L}^{-1}$  and  $100.0 \text{ mg L}^{-1}$ , respectively. The DR89 degradation by HC/PS/OA/PI and HC/PI

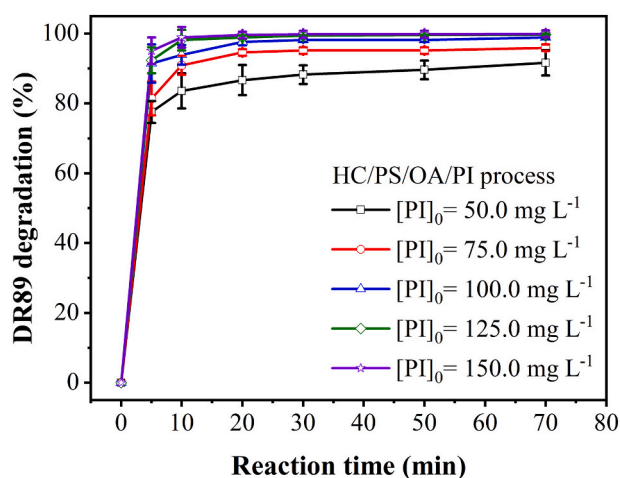


Fig. 5. Effect of PI dose on degradation of DR89 in HC/PS/OA/PI process. Experimental conditions:  $[DR89]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 125.0 \text{ mg L}^{-1}$ ,  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 50.0$ – $150.0 \text{ mg L}^{-1}$ , pH = 3.0, and reaction time = 70 min.

processes versus reaction time are shown in Figs. 5 and S10, respectively.

It can be clearly seen that the efficiency of DR89 degradation by both combined processes grows with increasing PI dose from  $50.0$  to  $150.0 \text{ mg L}^{-1}$ . The highest DR89 degradation by HC/PI and HC/PS/OA/PI process were achieved at a dose of  $150.0 \text{ mg L}^{-1}$  of PI, equal to  $99.90 \pm 0.09 \%$  and  $95.94 \pm 0.96 \%$ , respectively. As previously mentioned, the HC can generate strong oxidation species including  $\bullet OH$  and  $H^\bullet$  radicals and  $H_2O_2$ . It can also contribute to the activation of  $IO_4^-$  ( $E^\circ = +1.60$  V) with these reactive species yielding  $\bullet OH$  and periodyl radical ( $IO_4^\bullet$ ) (Eqs. (26)–(28)). The formed  $IO_3^-$  can initiate a chain of reactions to generate iodyl ( $IO_3^\bullet$ ) and  $\bullet OH$  radicals through the chemical reactions represented in Eqs. (29)–(31) [44,49]. These units react with the compounds and eventually enhance the rate of degradation. Iodine radical intermediates ( $IO_3^\bullet$  and  $IO_4^\bullet$ ) can interact and are believed to play the major role in the degradation of the organic pollutants. Moreover, the degradation of DR89 is more efficient in the first few minutes. This can be explained by the fact that the degradation not only relate to PI oxidant, but also attributed to the all-produced radicals and non-radical species ( $\bullet OH$ ,  $H^\bullet$  and  $H_2O_2$ ).



### 3.6. DR89 degradation by different processes

Experiments were performed to evaluate the DR89 degradation by various individual oxidation processes, including HC, OA, PS, and PI, and also combined processes, including HC/PS, HC/OA, HC/PI, PS/OA/PI, and HC/PS/OA/PI. The evolution of DR89 degradation with reaction time in the different processes is shown in Fig. 6.

After 70 min of reaction time (Fig. 6(a)),  $<3.0 \%$  of DR89 was degraded by the individual oxidation process with OA and PS, indicating that no free radicals were generated from PS and OA (in the absence of HC). The use of PI as oxidant results in a slight increase of the DR89 removal, achieving a degradation of  $5.46 \pm 0.27 \%$  after the 70 min of reaction. In contrast, to the observed with OA and PS, it seems that PI can generate a relatively low amount of radicals able to degrade the dye to a certain extent. The use of HC process results in a significantly higher DR89 degradation ( $56.88 \pm 1.7 \%$  at 70 min), which can be a consequence of the generation of micro-bubbles, microfluidics and  $\bullet OH$  radicals when using HC process [37,50]. The combination of HC process with the different oxidants (Fig. 6(b)) depicts significant increases of the degradation percentages, which achieved  $77.13 \pm 3.86 \%$ ,  $78.77 \pm 2.36 \%$ , and  $95.94 \pm 0.96 \%$  of DR89 with HC/PS, HC/OA, and HC/PI processes, respectively. These results confirm the synergetic effect produced by the combination of the oxidants with HC process. By combining PS, OA, and PI oxidants with the HC process, the DR89 degradation was improved and reached  $99.90 \pm 0.09 \%$ ,  $>95 \%$  within the first 5 min of reaction. This significant improvement could be due to the generation of large amounts of free radicals in the solution, which significantly increases the rate of degradation in these first few minutes.

The degradation of the DR89 by different process was fitted to a first-order kinetics (Eq. (32)), as shown in Fig. 7.

$$\ln C/C_0 = -k_1 t \quad (32)$$

where  $C_0$  and  $C$  are the initial and effluent concentration of the dye at

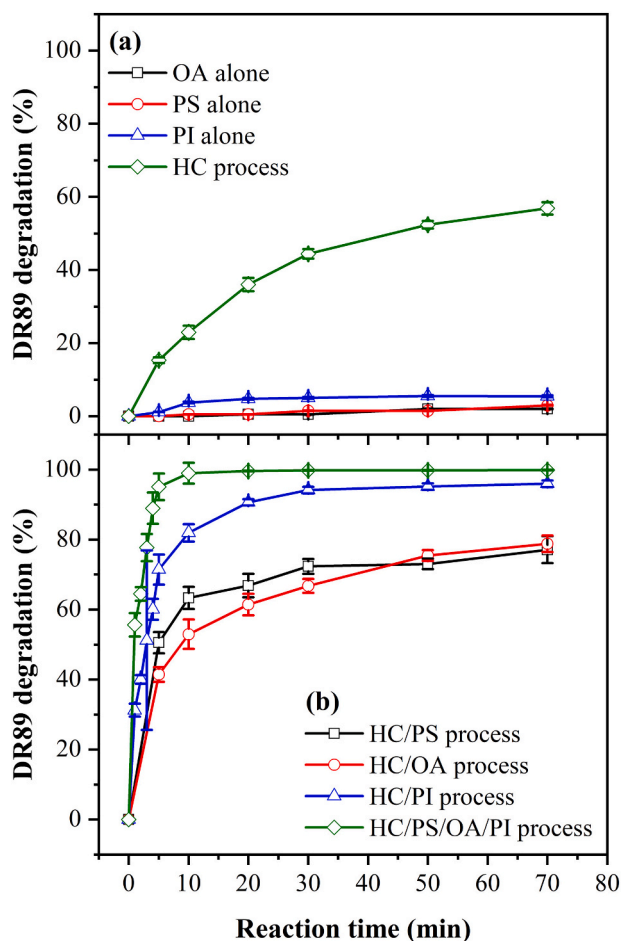


Fig. 6. DR89 degradation under individual process (a) and combined process (b). Experimental conditions:  $[DR89]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 125.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 150.0 \text{ mg L}^{-1}$ ,  $\text{pH} = 3.0$ .

time  $t$  ( $\text{mg L}^{-1}$ ),  $k$  is the first-order rate constant ( $\text{min}^{-1}$ ) and  $t$  is the reaction time (min). Table 1 summarizes the values of the first-order reaction rate constants. The highest constant rate was  $2.01 \times 10^{-1} \text{ min}^{-1}$ , obtained with HC/PS/OA/PI process, significantly higher than those of other individual and combined oxidation processes. The synergistic effect of the combined oxidation processes can be quantified as the normalized difference between the rate constants obtained in each combined oxidation process and the sum of the rate constants of the individual oxidation processes (Eq. (33)).

$$\text{Synergetic coefficient} = \frac{k_{com,p}}{k_{ind,p(1)} + k_{ind,p(2)} + k_{ind,p(n)}} \quad (33)$$

where  $k_{com,p}$  is the constant rate of the combined process and  $k_{ind,p}$  is the constant rate of individual processes. The values of this parameters for the different processes analyzed are also summarized in Table 1. The order of synergistic action of the combined oxidation processes is as follows: HC/PS/OA/PI (15.39) > HC/PI (7.24) > HC/OA (1.58) > HC/PS (1.29), which confirms the fastest degradation obtained when using HC/PS/OA/PI process.

### 3.7. Identification of possible oxidizing species and degradation pathway

In order to determine the possible oxidizing species in DR89 degradation by HC/PS/OA/PI process, several quenching experiments were conducted. For this purpose, different chemicals including EtOH, TBA, phenol, p-BQ, and  $\text{NaN}_3$  were used for quenching of  $\cdot\text{OH}$  and  $\text{SO}_4^{\cdot-}$ ,  $\text{SO}_4^{\cdot-}$ ,  $\text{IO}_3^{\cdot}$ ,  $\text{O}_2^{\cdot-}$ , and  $^1\text{O}_2$ , respectively [30,49,51]. Under the optimal

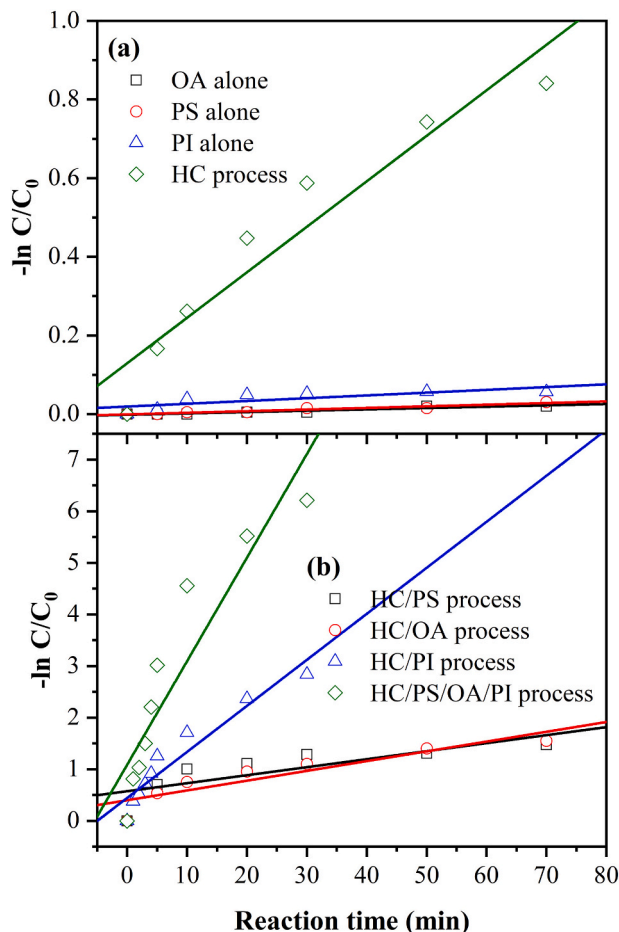


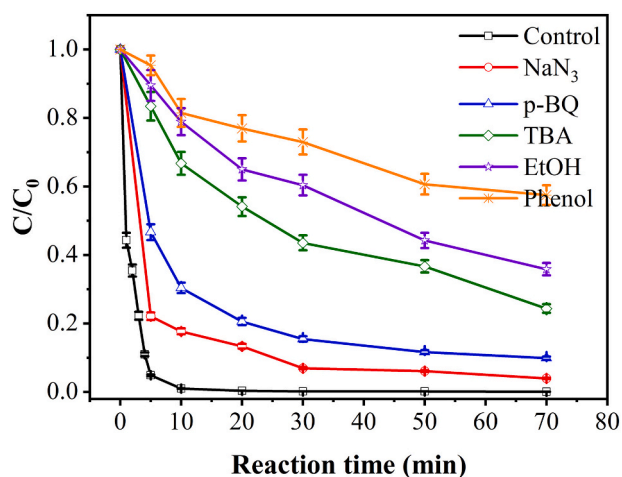
Fig. 7. First-order kinetic in (a) individual process and (b) combined processes.

Table 1  
Summarized reaction rate constants of the individual and combined processes.

Process type	$k_t$ ( $\text{min}^{-1}$ )	Synergetic effect
OA	$3.41 \times 10^{-4}$	–
PS	$4.12 \times 10^{-4}$	–
PI	$7.07 \times 10^{-4}$	–
HC	$1.16 \times 10^{-2}$	–
HC/PS	$1.55 \times 10^{-2}$	1.29
HC/OA	$1.89 \times 10^{-2}$	1.58
HC/PI	$8.92 \times 10^{-2}$	7.24
HC/PS/OA/PI	$2.01 \times 10^{-1}$	15.39

degradation conditions, the experiments were performed with addition of 15 mM of each quenching agent into test solution and obtained results are displayed in Fig. 8.

The DR89 degradation was not significantly decreased by p-BQ and  $\text{NaN}_3$ , which suggest that the degradation of DR89 was not controlled by  $\text{O}_2^{\cdot-}$ , and  $^1\text{O}_2$  species. Hu et al. [51] studied the levofloxacin (LVF) degradation by PI system and demonstrated that addition of quenching agents lead to low reduction of LVF removal rate from 95 % to 73.2 %, which indicating the possibility of the presence of  $\text{O}_2^{\cdot-}$ , and  $^1\text{O}_2$  species in the system. On the other hand, when EtOH or TBA was added to the system, the degradation of DR89 decreased very significantly. TBA addition resulted in a decrease in degradation from  $99.90 \pm 0.09 \%$  to  $75.58 \pm 2.27 \%$ , while EtOH reduced the degradation down to  $64.13 \pm 1.92$ . This confirmed the very significant role of  $\cdot\text{OH}$  and  $\text{SO}_4^{\cdot-}$  radicals in the HC/PS/OA/PI process. Hu et al. [49] used an iron-manganese oxide loaded biochar for the activation of PI anions in thiacloprid (TCP) degradation and reported that TCP degradation rate declined



**Fig. 8.** Degradation of DR89 in HC/PS/OA/PI process without and with scavenger. Experimental conditions:  $[DR89]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 125.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 150.0 \text{ mg L}^{-1}$ ,  $\text{pH} = 3.0$ ,  $[sc \pm nger]_0 = 15 \text{ mM}$ , and reaction time = 70 min.

from 94.1 % to 48.3 %, indicating an indispensable contribution of  $\cdot\text{OH}$  in the degradation system. As displayed in Fig. 8, the degradation efficiency of DR89 in the HC/PS/OA/PI process markedly inhibited with addition of phenol as a selective quencher of  $\text{IO}_3\cdot$  radicals. At this time, the degradation efficiency reduced from  $99.90 \pm 0.09 \%$  to  $42.47 \pm 1.27 \%$ , which demonstrates that  $\text{IO}_3\cdot$  were the main radicals in the HC/PS/OA/PI degradation process.

Based on the previous studies [14,52], the degradation pathway of DR89 proceeds through the formation of free active radicals ( $\cdot\text{OH}$ ,  $\text{SO}_4\cdot^-$  and  $\text{IO}_3\cdot$ ) and the oxidation of DR89 molecules by the attack of these free radicals. The dye molecules may undergo cleavage of C–N bond or symmetrical cleavage of azo bond, between naphthalene ring and azo bond or between triazolyl group and substituted aniline, respectively, resulting in the formation of intermediates like phenolic compounds, and further mineralization, to  $\text{CO}_2$ ,  $\text{NO}_3^-$ ,  $\text{SO}_4^{2-}$  and water.

### 3.8. Comparison with treatment process

The HC/PS/OA/PI process as the best combined process was compared to other treatment process for DR89 degradation in literature. Table 2 shows different studies for the treatment of DR89. As can be

**Table 2**  
Comparison of the performance of different process for treatment of DR89.

Process type	Reaction conditions	Removal	Ref.
HC/PS/OA/PI process	$C_0 = 100.0 \text{ mg L}^{-1}$ , $OA = 100.0 \text{ mg L}^{-1}$ , $PS = 125.0 \text{ mg L}^{-1}$ , $PI = 150.0 \text{ mg L}^{-1}$ , $\text{pH} = 3.0$ , and time = 70 min	99.9 %	Present study
Sulfate based AOPs	$C_0 = 50.0 \text{ mg L}^{-1}$ , $\text{pH} = 5.0$ , $ZVI = 300.0 \text{ mg L}^{-1}$ , sulfite dose = $400 \text{ mg L}^{-1}$ , and time = 60 min	99 %	[56]
Hydrodynamic cavitations	$C_0 = 80.0 \text{ mg L}^{-1}$ , $\text{pH} = 3.0$ , inlet pressure = 4.0 bar, and time = 130 min	75 %	[32]
Biosorption	$C_0 = 50.0 \text{ mg L}^{-1}$ , $\text{pH} = 1.0$ , adsorbent = $2.0 \text{ g L}^{-1}$ , and time = 90 min	46 %	[54]
Adsorption (wood sawdust)	$C_0 = 10.0 \text{ mg L}^{-1}$ , $\text{pH} = 2.0$ , adsorbent = $8.0 \text{ g L}^{-1}$ , and time = 40 min	90 %	[53]
Adsorption (activated carbons from sewage sludge)	$C_0 = 100.0 \text{ mg L}^{-1}$ , $\text{pH} = 3.0$ , adsorbent = $4.0 \text{ g L}^{-1}$ , and time = 90 min	90 %	[57]

seen, all the treatment process used capable to degrade DR89 from the dye solution, with degradation efficiency ranging from 46 % to 99 %. The use of adsorption process is an effective method for DR89 removal, but with highly consumption of sorbents [53–55]. Generally, HC/PS/OA/PI process provides high degradation efficiency (99.9 %), while the residual of oxidant agents is the most important problem of HC/PS/OA/PI process. In addition, the cost evaluation of HC/PS/OA/PI process is presented in the Text S1.

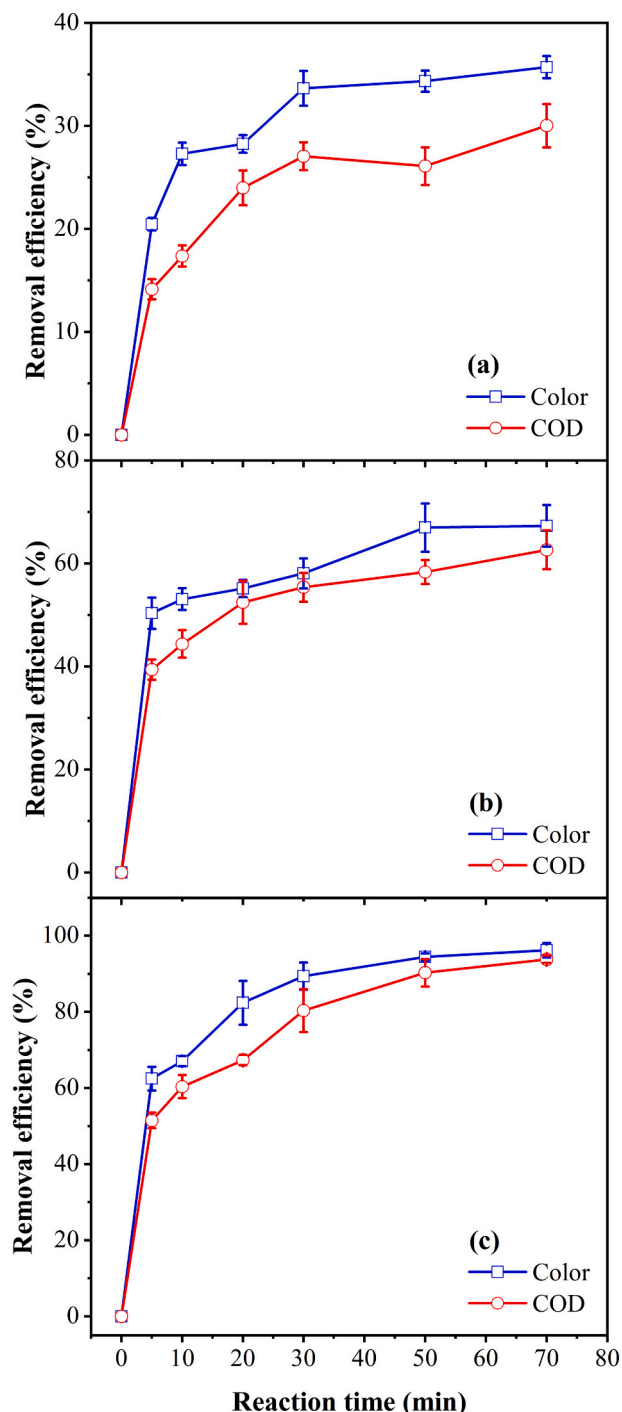
### 3.9. Treatment of real textile wastewater

The wastewater from textile industry is classified as a complex aquatic matrix which is considered as high organic-content wastewater. In the present work, the effectiveness of the HC/PS/OA/PI process in removing color and COD from real textile wastewater was also investigated for the first time. Fig. 9 illustrates the evolution of color and COD removal with reaction time of the real textile wastewater using a HC/PS/OA/PI process at different experimental conditions.

As depicts in Fig. 9, under the optimal conditions obtained from degradation experiments with pure water, the color and COD removals with real textile wastewater were equal to  $35.71 \pm 1.07 \%$  and  $30.03 \pm 2.10 \%$ , respectively. As expected, these degradation values are much lower than those previously discussed when using pure water. This situation is related to the much higher color and COD content of real textile wastewater compared to those of synthetic wastewater (theoretical COD of 100.0 mg of DR89 is equal to 170 mg). By doubling and tripling the doses of PS and PI, a considerable higher removal of color and COD content with the textile wastewater were obtained, equal to  $67.32 \pm 4.04 \%$  and  $96.23 \pm 1.92 \%$  for color and  $62.66 \pm 3.76 \%$  and  $93.84 \pm 0.94 \%$  for COD, respectively. Therefore, higher doses of oxidants were required to achieve acceptable removal percentages with the textile wastewater when using HC/PS/OA/PI process. Kiani et al. [58] studied real textile wastewater treatment by a  $\text{SO}_4\cdot^-$  radical-based AOP and reported that under optimum conditions ( $\text{pH} = 7.0$ , PS dose =  $570.0 \text{ mg L}^{-1}$ ,  $1.0 \text{ g L}^{-1}$  of  $\text{CuO@AC}$  and 60 min reaction time), about 95 % of color and 72 % of COD were removed. In another study, Asgari et al. [59] treated the real textile wastewater using ZnO/PS and reported that about 96.6 % of COD removed after 5 h reaction time at solution pH of 6.0, and by using ZnO dose =  $880 \text{ mg L}^{-1}$  and PS dose =  $233.2 \text{ mg L}^{-1}$ . Furthermore, Ghanbari et al. [60] studied the electrochemical activation of peroxides for treatment of contaminated water from landfill leachate and reported under the optimal conditions (hydrogen peroxide = 6 mM, applied current = 200 mA, and time = 80 min) about 83 % of the COD was removed (initial COD =  $1175 \text{ mg L}^{-1}$ ). Comparing to those previous studies, we used higher doses of PS and PI due to the higher COD content of the analyzed real textile wastewater. Regardless the oxidant doses, it should be remarked that the combined process was able to achieve an almost complete color (96 %) and COD (94 %) removal when treating a high-polluted real wastewater, which supports the high interest of the presented approach.

## 4. Conclusion

The present work studied the degradation of DR89 dye by combination of HC process with different oxidizing agents (OA, PS, and PI) and also the feasibility of combined HC/PS/OA/PI process for treatment of synthetic and, more important, real textile wastewater. The degradation experiments analyzed the effects of different factors on the dye degradation, including solution pH, dye concentration, reaction time and dosage of the oxidants. The HC/PS/OA/PI process achieved the highest DR89 degradation compared to the rest of individual or combined processes. The optimal conditions when using the HC/PS/OA/PI process were initial DR89 concentration =  $100.0 \text{ mg L}^{-1}$ ,  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 125.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 150.0 \text{ mg L}^{-1}$ ,  $\text{pH} = 3.0$ , and reaction time = 70 min. Using these conditions, the degradation of DR89 was close to complete ( $99.90 \pm 0.09 \%$ ). Overall, the obtained synergistic



**Fig. 9.** Color and COD removal from for textile wastewater by HC/PS/OA/PI process. (a) Experimental conditions:  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 125.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 150.0 \text{ mg L}^{-1}$ , (b) experimental conditions:  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 250.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 300.0 \text{ mg L}^{-1}$ , (c) experimental conditions:  $[OA]_0 = 100.0 \text{ mg L}^{-1}$ ,  $[PS]_0 = 500.0 \text{ mg L}^{-1}$ ,  $[PI]_0 = 600.0 \text{ mg L}^{-1}$ .

effect in HC/PS/OA/PI process was related to generation of powerful oxidants, mainly including  $\cdot OH$ ,  $SO_4^{\cdot -}$  and  $IO_3^{\cdot}$ . The treatment of a real textile wastewater using the combined HC/PS/OA/PI process showed that after 70 min,  $96.23 \pm 1.92\%$  of color and  $93.84 \pm 0.94\%$  of COD were removed. This study provides a new insight into the treatment of textile wastewater based on combined advanced oxidation process.

In this study, the cooling water bath was used to maintain constant temperature and lead to further energy and resource consumption. In

future, the DR89 removal experiments can be carried out with free heating for better understanding of the possible pathways for DR89 removal and also economical evaluation. In addition, further research should be conducted to model the obtained data.

#### Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

#### Data availability

Data will be made available on request.

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#### Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.jwpe.2022.103143>.

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