



Photocatalytic Fenton oxidation of sodium dodecyl sulfate solution using iron-modified zeolite catalyst

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ABSTRACT

The heterogeneous photo-Fenton oxidation of sodium dodecyl sulfate (SDS) solutions was investigated in a quartz batch reactor using an artificial UV light source. Fe-modified zeolite was used as a heterogeneous catalyst in the process. The effect of various process variables on SDS removal performance was evaluated by examining temperature, pH, H₂O₂ dosage, catalyst loading, initial SDS concentration and light intensity. The optimal operational parameters were found as follows: temperature 35°C, solution pH 7.5, 15 mmol H₂O₂ dosage, and 1 g/L catalyst loading. Stability and the reuse of the catalyst were also tested. Comparison with homogenous photo-Fenton process was also performed by analyzing SDS removal rate. Kinetic investigation of removal process based on different kinetic models was evaluated. The results showed that the first-order kinetic model was in good correlation with experimental data.

Keywords: Heterogeneous photo-Fenton; Fe-modified zeolite; SDS; Advanced oxidation

1. Introduction

Sodium dodecyl sulfate (SDS), the crystalline sodium salt (C₁₂H₂₅NaO₄S) of sulfated laurels alcohol, is an anionic surfactant and used as a detergent, wetting, and emulsifying agent in household detergents and also many personal care items such as shampoos and toothpastes [1]. Surfactants can also be used in paper, food, polymer, pharmaceuticals, and oil recovery industries. Surfactants are usually nontoxic and harmless in low concentrations; however, their degradation products have potential health and environmental effects. Surfactants produced by household and industry have significant impacts by formation of toxic or nuisance foams in receiving media and treatment plants. The excess foam generated by surfactants

can cause operational problems in the treatment plants [2]. Therefore, removal of SDS from aqueous effluents is a significant environmental issue. The efficiency and simplicity of advanced oxidation processes can make a suitable choice for the removal of surfactants from wastewaters [3,4].

There are a number of studies regarding to the removal of surfactants by advanced oxidation and other methods in the literature. SDS removal using agricultural by-product as an adsorbent was investigated to optimize the adsorption process [5]. The electrochemical oxidation on boron-doped diamond of synthetic waste polluted with surfactant, sodium dodecylbenzenesulfonate (SDBS), was studied by Louhichi et al. [6]. Peroxi-electrocoagulation process was investigated in the treatment of SDS-containing wastewater [7]. The results showed that SDS in aqueous phase was

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effectively removed by the peroxi-EC method. Wet air oxidation for removal of SDBS was studied by Suarez-Ojeda et al. [8]. The photocatalytic degradation of the SDBS was performed using TiO_2 as catalyst in the work by Zhang et al. [9]. The work by Bandala et al. investigated SDS degradation using Fenton and solar-driven Fenton and compared these results with cobalt/peroxymonosulfate process [10]. Their results showed that degradation efficiency of Fenton and Co/PMS was increased considerably with the use of solar radiation.

The Fenton-like processes have been used as a powerful source of hydroxyl radicals with H_2O_2 in the presence of metal cations, with effective reaction conditions [3,11]. The use of homogenous Fenton systems ($\text{Fe}^{2+}/\text{Fe}^{3+}/\text{H}_2\text{O}_2$) with UV was extensively reported [12,13]. Due to some disadvantages of these systems, such as pH range of reaction and sludge production, heterogeneous photocatalytic processes were developed using zeolite-like minerals. Iron ions can either be adsorbed or exchanged onto the surface of zeolites, then form $\text{Fe}^{2+}/\text{Fe}^{3+}$ complexes on the surface of zeolite which can react with H_2O_2 or by the UV radiation, thus allowing iron ions to participate in the Fenton catalytic cycle [14–16]. Zeolites are hydrated aluminosilicate minerals having the large internal and external surface areas and cation exchange capacities with formula of $(\text{Na,K})_x\text{Si}_{30}\text{Al}_6\text{O}_{72}\cdot n\text{H}_2\text{O}$ [17]. Zeolites are also ideal catalytic materials because they have nanostructure on the same scale as the molecules that react within their pores [18,19]. Fe-exchanged zeolites, as a heterogeneous catalyst, were used both in wet peroxide oxidation systems [20] and photochemically enhanced Fenton reactions [21,22].

The aim of this paper was to investigate the use of Fe-modified zeolite as an heterogeneous Fenton-type catalyst using UV source lamps for the removal of SDS in solutions. The efficiency of the heterogeneous oxidation process was evaluated by examining temperature, pH, H_2O_2 dosage, catalyst loading, initial SDS concentration, and light intensity and compared with homogenous Fenton reaction. Kinetic investigation of the removal process was also evaluated by applying first- and second-order kinetic reaction models.

2. Materials and methods

2.1. Materials

SDS ($\text{SDS}-\text{C}_{12}\text{H}_{25}\text{SO}_4\text{Na}$) and analytical grade H_2O_2 solution (30%) were provided by Merck and used without purification. Deionized water was used throughout the experiments. The pH of the solution was adjusted using 0.1 N NaOH or 0.1 N H_2SO_4 to explore the effect of the pH, otherwise the experiments were carried out as such at the solution pH.

Natural zeolite samples used in this work were obtained from Bigadiç region in Turkey. The main characteristics of the zeolite were described previously [23]. Mineral identification showed that the zeolite consisted of mainly clinoptilolite (80%). All zeolite samples were grounded to 40–80 mesh after washing and drying. Catalysts were then prepared by modifying the zeolites with 0.02 M $\text{Fe}(\text{NO}_3)_3\cdot 9\text{H}_2\text{O}$ solutions and amount of iron concentration on zeolite samples was found as 28 mg/g after four times conditioning experiment according to the procedure performed previously [24].

2.2. Photoreactor and chemical analyses

A 500 mL capacity batch photoreactor was used in the experiments [24,25]. This laboratory-scale quartz photoreactor was designed in a column of 34 cm height and 4.6 cm diameter. Air was blown into the reactor with a flow rate of 150 mL/min to saturate the solution with oxygen during the reactions. Stirring was applied to disperse the catalyst particles and gas bubbles. Three types of lamp were used as follows: UVC (mainly emits at 254 nm) (Philips TUV8 W FAM), UVA white (predominantly emits at 365 nm) (Philips TL8 W/05 FAM), and UVA blue (predominantly emits at 365 nm) (Philips TL 8 W/08 FAM). The reactor has six lamp sockets which are appropriate for each type of lamp. The lamps were located equally in an aluminum sealed tube in a hexagonal position to enhance the radiation by reflection. The UVA white-type lamps were used throughout the experiments unless otherwise stated. The heat effect of the lamps was sufficient for temperatures needed over 20 °C. Chilled air was blown to eliminate the heat effect of the lamps for lower temperatures. The sensor of the light meter was placed in the quartz reactor inside the lamp assembly to measure the light intensity of the lamps (Lutron UVA-365 and UVC-254). Intensity measurements were performed as following: after switching all lamps on, six measurements were taken by directing the sensor to each lamp hexagonally and a mean value was recorded. Lamps were switched off one by one and measurements were recorded in the same manner and then values were evaluated and, consequently, linear model was achieved according to the number of lamps. The total intensity of each set of six lamps was obtained as 3.17 mW/cm² for UVC, 2.30 mW/cm² for UVA white, and 3.55 mW/cm² for UVA blue.

Samples were collected at 30 min intervals and centrifuged to remove catalyst particles. The remaining SDS concentration was analyzed using a titrimetric (ISO 2271) method [26].

3. Results and discussion

3.1. Effect of the reaction temperature

The influence of temperature on the SDS removal process using 50 mg/L SDS concentration, pH 7.5, 15 mmol H_2O_2 , and 1.0 g/L Fe^{3+} -Z was performed at 25, 30, and 35°C. Fenton reactions preferably take place over 25°C and reach the optimum at 40°C. Over 40°C, it is not convenient for aqueous solution due to water loss and economy. Therefore, the temperature values between 25 and 35°C were chosen to study in the experiments. Fig. 1 shows the effect of temperature on SDS removal efficiency. The removal rate was increased when temperature was raised from 25 to 35°C. Increase in the temperature can usually provide additional energy to the reactants for overcoming the activation energy barrier. Due to higher temperature effect on the reaction between hydrogen peroxide and the catalyst increases the rate of generation of OH radicals. The previous heterogeneous Fenton studies have also proved the importance of temperature on the removal processes [27]. The SDS removal increased with temperature but was not rigorously effected by an increase up till 35°C and had reached a plateau level at around 30°C. Heat effect of the lamps was already convenient for this level of temperature and, therefore, 30°C was selected for further experiments.

3.2. Effect of the initial pH values

The set of experiments was performed in order to determine the effect of initial pH for SDS removal in the present system (50 mg/L SDS concentration, temperature 30°C, 15 mmol H_2O_2 , 1.0 g/L Fe^{3+} -Z). The

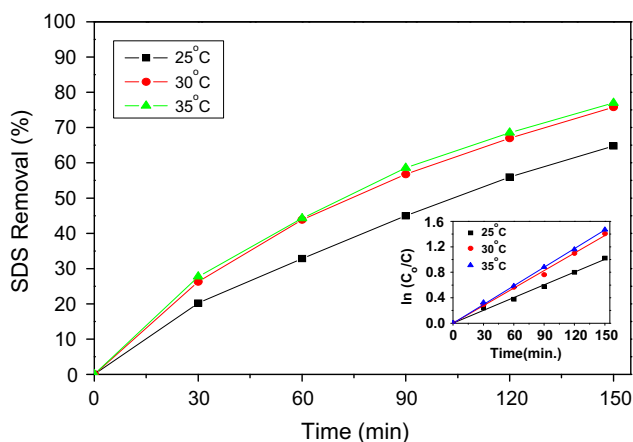


Fig. 1. Effect of temperature on SDS removal efficiency (500 mL, 50 mg/L SDS, 15 mmol H_2O_2 , 1 g/L Fe^{3+} -Z, and pH 7.5).

experiments were carried out in the range of pH 3–9 and SDS solutions were adjusted to the desired pH values with an addition of 0.1N NaOH or 0.1N H_2SO_4 . In early stages of the reaction, the initial pH of the solution was effective at 7.5 and at lower values, the removal rate was increased (Fig. 2). As the nature of Fenton processes, the pH improves Fenton reagent performance [7,28] and it was also reported that the leaching of Fe cations from zeolite was enhanced at lower pH values [24]. However, due to process competence, further experiments were performed at initial solution pH (7.5) without the pH value.

3.3. Effect of the initial SDS concentration

The influence of various initial SDS concentrations on the removal process was investigated between 25–150 mg/L SDS solutions (Fig. 3). The experimental conditions were as follows: pH 7.5, temperature 35°C, 1.0 g/L catalyst and 15 mmol H_2O_2 . The removal efficiencies were in the range of 62–92% for all concentrations studied. Similar removal efficiency was reported for SDS degradation using solar-driven Fenton process in the work by Bandala et al. [10]. The removal rates were decreased by increasing initial concentrations due to reaction stoichiometry. The increase in SDS concentration increases the number of SDS molecules and competition with the OH \cdot radical concentration and the removal rate decreases. In photo-Fenton process at higher SDS concentration, the penetration of photons entering into the solution decreases thereby, lowering the hydroxyl radical concentration [14]. Hence, oxidation process requires more OH \cdot radical concentration and longer reaction times for higher SDS concentrations.

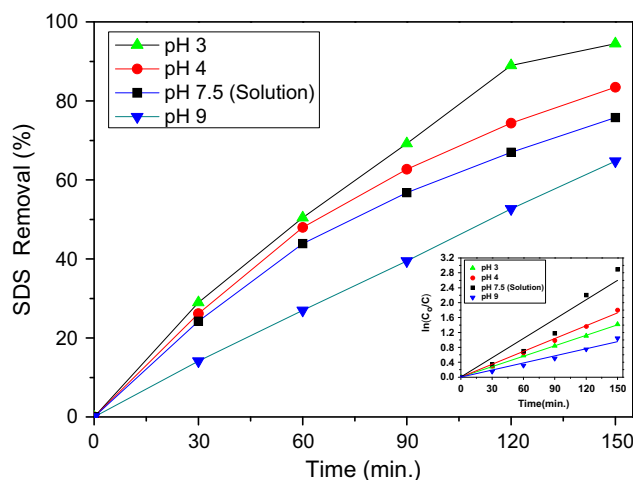


Fig. 2. Effect of pH on SDS removal (500 mL, 50 mg/L SDS, 15 mmol H_2O_2 , 1 g/L Fe^{3+} -Z, and 30°C).

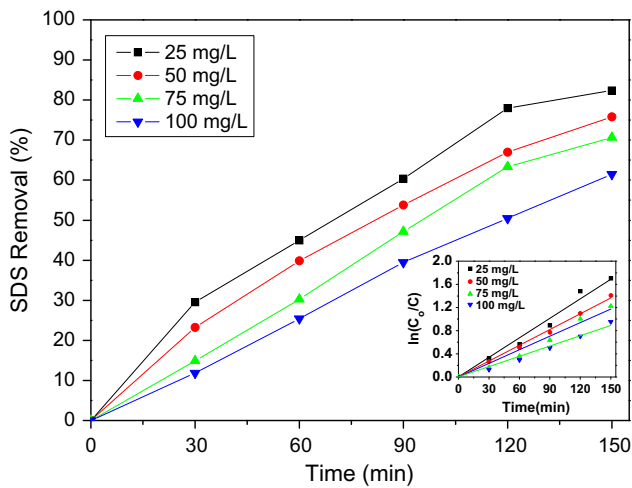


Fig. 3. Effect of initial SDS concentration on the removal efficiency (500 mL, 15 mmol H₂O₂, 1 g/L Fe³⁺-Z, 30 °C, and pH 7.5).

3.4. Effect of H₂O₂ dosage

The effect of H₂O₂ dosage on the removal process was performed using following experimental conditions: 50 mg/L SDS concentration, pH 7.5, temperature 30 °C and 1.0 g/L catalyst. Increasing H₂O₂ concentration with a dosage more than 15 mmol improves the removal process (Fig. 4). The enhancement of the removal rate by addition of H₂O₂ is due to increase in the OH[•] radicals [10,14]. However, the optimum H₂O₂ concentration was chosen as 15 mmol as this dose produces enough OH[•] radicals to complete the oxidation process. Therefore, it can be concluded that an H₂O₂ dosage higher than 15 mmol corresponds to unnecessary consumption of hydrogen peroxide.

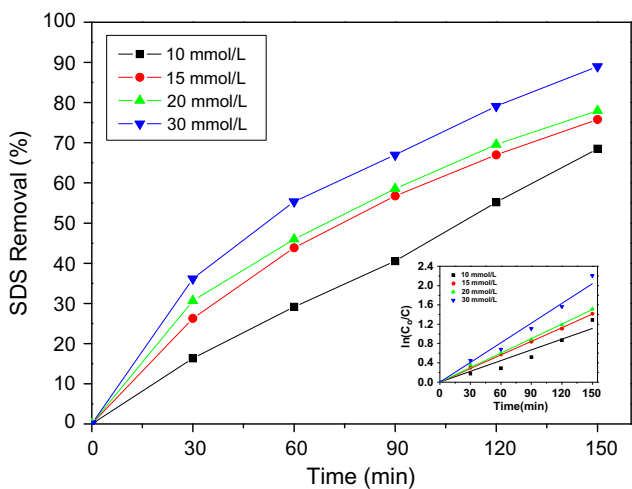


Fig. 4. Effect of H₂O₂ concentration on SDS removal (500 mL, 50 mg/L SDS, 1 g/L Fe³⁺-Z, 30 °C, and pH 7.5).

3.5. Comparison of process reactants

Heterogeneous Fenton-type photocatalytic reactions were carried out in the absence of four main process reactants as follows: UV, H₂O₂, Fe³⁺-Z, and oxygen using the experimental conditions as follows: pH 7.5, temperature 30 °C, 1.0 g/L catalyst, 15 mmol H₂O₂, and 50 mg/L initial SDS concentration. The removal rates were negligible if no UV or H₂O₂ was used (Fig. 5). This indicates the importance of UV and H₂O₂ in the heterogeneous photo-Fenton catalytic reactions. The reactions are initiated by the photoreduction of Fe³⁺ on the surface of [Fe³⁺-Z] to Fe²⁺ under illumination of UV light. Then, this formed Fe²⁺ accelerates the decomposition of H₂O₂ in solution generating highly oxidative OH[•] radicals. Meanwhile, H₂O₂ itself produces limited number of OH[•] radicals with light and O₂. These OH[•] radicals attack SDS molecules leading to reaction intermediates and finally, the reaction intermediates are mineralized into CO₂ and H₂O Eq. (1–6) [29,30]. Meanwhile, feeding air to the reaction solution in order to give oxygen to the system increased the removal rate particularly in the initial stages of the reaction. The absence of UV caused the restricted production of hydroxyl radicals leading to partial degradation since the usual Fenton-like (H₂O₂, Fe³⁺-Z) oxidation has already occurred.

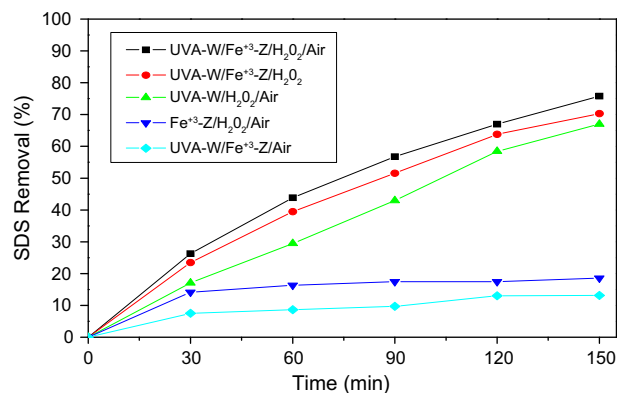
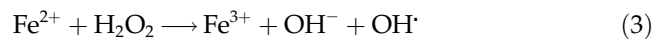
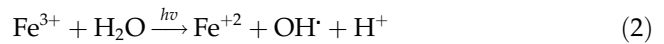
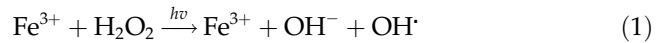
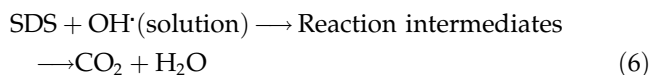
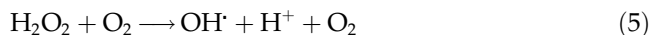


Fig. 5. Comparison of process reactants on SDS removal efficiency. (500 mL, 50 mg/L SDS, 15 mmol H₂O₂, 1 g/L Fe³⁺-Z, 30 °C, and pH 7.5).



3.6. Effect of catalyst loading

Experimental studies of catalytic oxidation with different catalyst loadings were conducted using the following experimental conditions: 50 mg/L SDS concentration, pH 7.5, temperature 30°C and 15 mmol H₂O₂ (Fig. 6). The removal rates were slightly changed by increasing catalyst loading. However, due to catalyst cost efficiency, the catalyst concentration of 1 g/L was selected for further experiments. Similar observations were reported for acid orange degradation of photocatalytic wet peroxide oxidation using iron-doped silica as a catalyst [31].

3.7. Effect of lamp type and UV light intensity

The effects of UV lamp type and light intensity on the process were observed using following experimental conditions: 50 mg/L SDS concentration, pH 7.5, temperature 35°C, 15 mmol H₂O₂, and 1.0 g/L catalyst. UVA-White-, UVA-Blue-, and UVC-type lamps were used to explore the effect of lamp type on the removal process (Fig. 7(a)). The photochemically active region of photo-Fenton processes lies in the UV region which is generally classified as UVC spectral band (mainly emits at 254 nm), whereas the solar radiation part of

UV wavelength reaching the earth is generally defined between 315–380 nm [32]. Although the highest removal rate was achieved using UVC lamp type, UVA emitting lamp types in the region of solar radiation were selected as a light source in this study. The SDS removal rate by light intensity was given in Fig. 7(b). Photogenerated OH[·] radicals were increased by increasing the light intensity, hence SDS removal rates were efficiently enhanced as expected.

3.8. Stability and reuse of catalyst

The reuse tests were performed in order to evaluate the catalytic activity of iron-modified zeolite during successive sequential experiments and, thus, to observe the possibility of catalyst reuse. The catalyst was used in three consecutive experiments by using fresh dye solutions at optimum conditions (50 mg/L dye concentration, pH 5.2, temperature 35°C, 15 mmol H₂O₂ and 1.0 g/L catalyst). Between each experiment, the catalyst was removed by filtration and then washed with deionized water for several times and

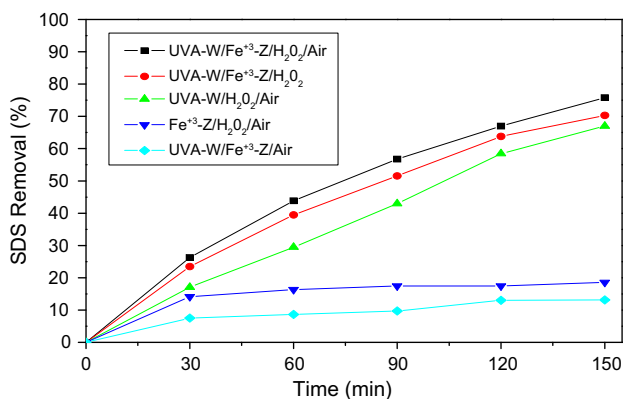


Fig. 6. Effect of catalyst loading on the SDS removal (500 mL, 50 mg/L SDS, 15 mmol H₂O₂, 30°C, and pH 7.5).

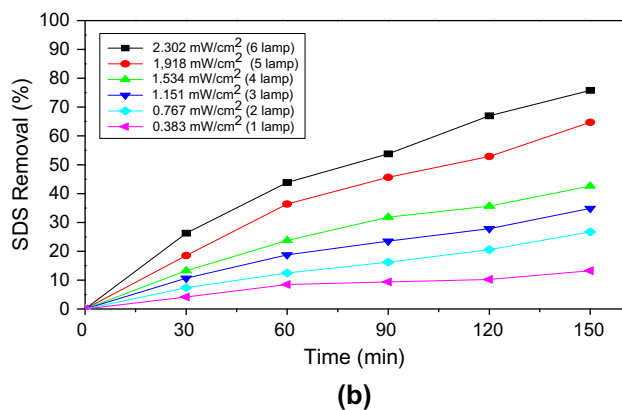
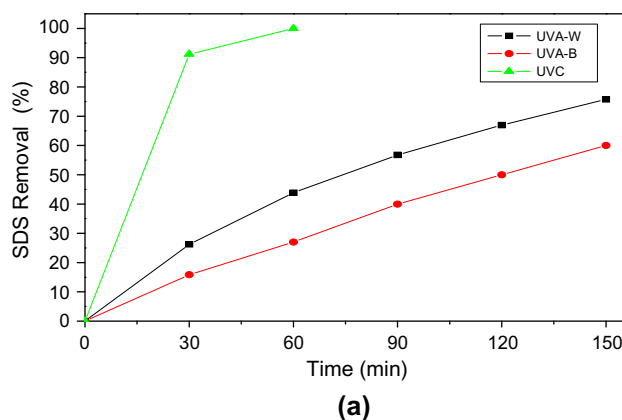


Fig. 7. SDS removal using lamp types (a) and light intensity (b) (500 mL, 50 mg/L SDS, 15 mmol H₂O₂, 1 g/L Fe³⁺-Z, 30°C, and pH 7.5).

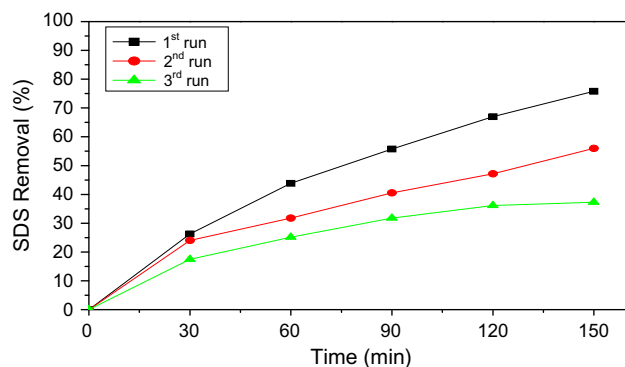


Fig. 8. Reuse of catalyst (500 mL, 50 mg/L SDS, 1 g/L Fe^{3+} -Z, 15 mmol H_2O_2 , 30 °C and pH 7.5).

dried at 50 °C for overnight. It is obvious that the initial activity decreased gradually during consecutive runs (Fig. 8). At longer reaction times, the removal rate is rather comparable for all the runs. The decay of active catalytic sites is considered as the reason for this initial loss in activity because small amounts of leached iron from the catalyst surface are observed [33]. To demonstrate that during the first run, the leached iron concentrations in the solution were measured in 150 min, the usual run time, and were found less than 0.55 mg/L. Hence, this amount was very

small considering the iron concentration on the exchanged zeolite which was 28 mg/g. As a result, the process can be considered as heterogeneous and no iron hydroxide sludge is created. These values are compatible with those measured in the some other work published [22,34].

3.9. Kinetic evaluation of processes

First- and second-order reaction kinetics were applied to the experimental data since different steps may take place in the catalytic photo-Fenton reactions.

The reaction kinetic constants, k_1 (min^{-1}) and k_2 (L/mg min) were calculated from the slope of the straight line of related kinetic models and given in Table 1. The kinetic constants were favorable with increased temperature as higher temperature affects the rate of generation of OH^\cdot radicals. The kinetic constants also increased with increasing H_2O_2 concentrations and higher catalysis loading, due to increased production of OH^\cdot radicals. The kinetic constants were decreased with higher initial SDS concentration, as expected. The average regression coefficients were also given for comparison in the same table. It can be seen from Table 1 that the first-order kinetic model was indicated as in somewhat good correlation with experimental data than the second-order kinetic

Table 1
The reaction kinetic constants for SDS removal at different reaction conditions

		First order		Second order	
		$k \times 10^{-3}$ (min^{-1})	R^2	$K \times 10^{-3}$ (L/mg min)	R^2
Temperature	25 °C	6.7	0.998	0.24	0.960
	30 °C	9.2	0.998	0.40	0.944
	35 °C	9.7	0.999	0.43	0.942
Initial concentration	25 mg/L	11.3	0.992	1.30	0.893
	50 mg/L	9.1	0.998	0.41	0.920
	75 mg/L	7.8	0.989	0.21	0.917
	100 mg/L	5.9	0.992	0.10	0.931
H_2O_2 concentration	10 mmol/L	7.4	0.966	0.27	0.868
	15 mmol/L	9.4	0.999	0.40	0.944
	20 mmol/L	10	0.999	0.46	0.933
	30 mmol/L	13.6	0.991	0.98	0.787
Catalyst concentration	0.5 mg	7.7	0.999	0.30	0.954
	1.0 mg	9.2	0.999	0.41	0.923
	1.5 mg	9.9	0.997	0.48	0.890
	2 mg	13.6	0.991	1.04	0.794
pH	3	9.4	0.999	2.20	0.704
	4	11.5	0.998	0.70	0.880
	7.5	17.3	0.969	0.41	0.944
	9	6.4	0.988	0.24	0.901

model. Therefore, first-order kinetic model curves were placed in the related figures as inset graphics (Figs. 1–4, and 6).

4. Conclusions

In this work, SDS removal was investigated using the heterogeneous photo-Fenton oxidation process. The role of various operating parameters on the removal process such as temperature, pH, H₂O₂ dosage, catalyst loading, initial SDS concentration, light intensity, and process reactants were explored. The optimum process conditions were selected from variable parameters. The optimal operational parameters were found as follows: temperature 30°C, pH same as solution pH, that is, 7.5, 15 mmol H₂O₂ dosage, and 1 g/L catalyst loading. The first-order kinetic model was indicated as in good correlation with experimental data. Stability and reuse of the catalyst were demonstrated up to three consecutive experiments showing that the reaction was essentially heterogeneous. The use of UVA-type light source has also proved that the heterogeneous photo-Fenton reaction was successful in the UVA region of solar radiation. This makes the UVA-assisted heterogeneous Fenton oxidation a promising technology to treat wastewater as it makes use of natural solar energy for power efficiency. This technology also uses fewer chemicals and does not produce toxic waste.

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