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Study of the impact of combination of ZnO nanoparticles with ultraviolet radiation (photocatalytic process) on the removal of anionic surfactant linear alkyl benzene sulfonate (LAS) from aqueous solutions using Taguchi statistical method

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ABSTRACT

Surfactants or surface-active agents are molecules with hydrophilic head and hydrophobic tail. They have been widely used as active ingredient in soap and detergent formulations. Detergents are synthetic, organic materials with cleansing and dissolution properties. Among the anionic surfactants, linear alkyl benzene sulfonate (LAS) has the highest consumption and is used in high concentrations in household detergents as washing powders, dishwashing liquids, and other household cleaners. LAS can have an impact of inhibiting the biological activity in the soil, which plays an important role in soil fertility and nutrient cycles. Removing pollutants in advanced oxidation processes is mainly based on producing the hydroxyl free radicals with potential high oxidation as well as the fact that many organic compounds can be converted into minerals. In the past two decades, photocatalyzing by semiconducting materials such as zinc oxide (ZnO) has received much attention due to its high sensitivity, high stability, non-toxic nature, wide energy gap, and higher efficiency in the production of electrons. The properties of the energy gap may cause the absorption of UV portion of the spectrum by these nanoparticles. In this study, initial concentrations of LAS including 0.5, 14, and 21 mg/L, ZnO concentration of 50, 100, and 150 mg/L, pH 3, 7, and 11, duration of 8, 16, and 24 min were used. Also, the Taguchi statistical method was used. The result showed that, in the LAS concentration of 21 mg/L, duration of 24 min, pH 3, and ZnO concentration of 150 mg/L, the highest percentage of removal could be seen.

Keywords: LAS; ZnO; Semiconductor; Photocatalyzing

1. Introduction

Surfactants extracted from municipal and industrial sewage are indeed xenobiotic. Therefore, their entrance into the environment may provide pollutions leading to low degradation of these materials [1,2]. Surfactants or surface-active agents are indeed molecules with a hydrophilic head and a hydrophobic tail which have been widely used as an active ingredient

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in soaps and detergent formulations [3,4]. Detergents are in fact synthetic organic materials with cleansing and dissolution properties [5]. These chemical compounds were made up of different materials such as the active substance or surfactant, builder, additives, and fillers. Furthermore, surfactants can be divided into four groups: nonionic, anionic, cationic, and amphoteric [6,7]. Ionic surfactants are accounted for two-thirds of the whole surfactants which more than ninety percent of ionic surfactants comprise anionic surfactants [8]. Among the anionic detergents, linear alkyl benzene sulfonate (LAS) has the highest consumption and is used largely in the household detergents as washing powder, dishwashing liquid, and other household cleaners [5]. LAS is also used as emulsifier agent in industrial applications such as agricultural pesticides, food and cosmetics, printing inks, and paints [9,10]. In fact, LAS contains a sulfonated aromatic ring in the para position and is attached to a linear alkyl chain at any position other than the position of the terminal that is shown in Fig. 1 [11].

LAS can have an inhibition impact on the biological activity in the soil, which may play an important role in the soil fertility and nutrient cycles [12]. LAS could also have a toxicity effect on aquatic organisms in low concentrations, i.e. about 1 milligram per liter; thus, due to its resistance against biodegradation, it can accumulate in the bodies of aquatic organisms [10,13,14]. Furthermore, if LAS enters the water, it can produce negative ions and may cause the rapid growth of aquatic plants in water resources and could lead to further eutrophication, decomposition of aquatic plants reducing the dissolved oxygen for aquatic life and water and sewage oxidation as well [15]. Moreover, average concentrations of surfactants in household waste might be about 1–10 mg/L and could reach above 21 mg/L [16,17]. The LAS values are usually between 3 and 21 mg/L in domestic sewage [18]. In 2009, The Institute of Standards and Industrial Research of Iran determined the maximum permissible amount of detergent in drinking water about 200 μg/L [19]. Anionic surfactants, which are less filterable due to their exposure to the stable materials, are resistant to biological treatment. In addition, chemically resistant materials may refer to substances whose rate of degradation or cellular uptake is less compared to ordinary bacteria in case of entering the acceptor areas or sectors such as wastewater treatment sectors [20]. It should be noted that it cannot be completely removed by conventional treatment and may create environmental problems [21]. In fact, the conventional methods of surfactants' removal from water, reported so far, may include chemical precipitation, adsorption, membrane technology, and biological methods, which may require more time and higher costs for sludge production [9,22]. Furthermore, in recent decades, advanced oxidation technologies have been used, among other processes, as the most efficient option for the destruction of toxic materials, especially in effluent wastewater treatment plants, which have features such as chemical stability or low biodegradation ability [21]. The removed pollutants in advanced oxidation processes, based on producing hydroxyl free radicals with high oxidation potential in many organic compounds, can indeed be converted into minerals [23]. Moreover, the free radicals are powerful for oxidizing and quick attacking of the organic molecules. Thus, one hydrogen atom can be removed from the structure, which may increase the organic matter decomposition [24]. The photocatalytic method using nanoparticles is in fact one of the advanced oxidation processes which have been considered in recent years, applicable for water and wastewater treatment [25]. In the past two decades, photocatalyzing by semiconducting materials such as zinc oxide (ZnO) has received more attention due to its high sensitivity, high stability, non-toxic nature, wide energy gap, and higher efficiency in the production of electrons [25]. The properties of the energy gap can cause the absorption of the ultraviolet (UV) portion of the spectrum by these nanoparticles [26]. The nanoparticles of metal oxides include TiO_2, ZnO, ZrO_2, Fe_2O_3, and SnO_2 [25]. However, the catalyst cost and toxicity consist of the restrictions in the use of some of these substances in the water; it is desirable to consider the ZnO nanoparticles for use in wide development [27]. The mechanism of this process includes irradiating the ultraviolet light onto the semiconductor material and prompting an electron from the valence band to the conduction band electrons, which can stimulate the production of the hydroxyl radicals [25]. ZnO has a band gap of 2.3 eV and

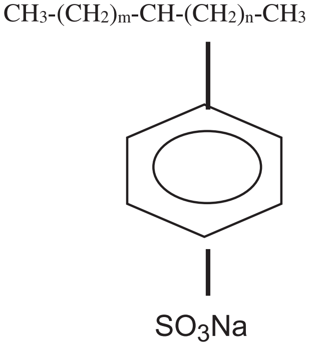

Fig. 1. LAS structure.

irradiates a wavelength of 387 nm and is excitable with UVA light in the 320–380 nm wavelength [26]. In addition, when ZnO nanoparticles are under ultraviolet irradiation in visible environment, the irradiation may cause the excitation and activation of electrons in the valence band and the electrons may go to the conduction band. This can create a hole in the valence band which is very active and can be directly absorbed by organic pollutants and then can react on the catalyst surface, or can do it indirectly through hydroxyl radical [26,28]. The hydroxyl radical formation caused by radiation to ZnO surface is shown in Eqs. (1)–(4) and Fig. 2 [29].

$$\mathrm{ZnO} + hv \longrightarrow \mathrm{ZnO}(e^-_{\mathrm{Cb}} + h^+_{\mathrm{Vb}}) \qquad (1)$$

$$h^+_{\mathrm{Vb}} + \mathrm{LAS} \longrightarrow \mathrm{LAS}^{0+} \longrightarrow \text{oxidation of the LAS} \qquad (2)$$

$$h^+_{\mathrm{Vb}} + \mathrm{H_2O} \longrightarrow \mathrm{H}^+ + \mathrm{OH}^0 \qquad (3)$$

$$h^+_{\mathrm{Vb}} + \mathrm{OH}^- \longrightarrow \mathrm{OH}^0 \qquad (4)$$

ZnO has three major advantages: It is a semiconductor with a 3.37 eV band gap, its excitation energy is high (60 meV), and also it is close to UV emission. On the other hand, ZnO is safe and biocompatible [29,30]. The biggest advantage of ZnO is its ability to absorb a range of the electromagnetic and photocatalytic capabilities under UVA radiation. In fact, ZnO is non-toxic with chemical stability at high temperatures and able to produce chemical oxidation [31,32]. Due to the high cost of the chemical processes and the possible formation of toxic byproducts during water treatment, the applications of photocatalytic process have been increased in recent years [33]. In this study, the photocatalytic oxidation using ultraviolet radiation was investigated on the treatment and removal of LAS. The objective of this research was to assess the effect of pH, time, the initial LAS concentration, and the nanoparticle concentration in LAS removal efficiency from aqueous solution.

2. Materials and methods

This semi-experimental was conducted to investigate the possibility of eliminating LAS from the synthetic solutions using ultraviolet radiation and ZnO nanoparticles in laboratory scale. The spectrophotometer model DR-2000 with 652 nm wavelength was used for measuring the LAS concentration. The test procedure was as follows:

1. Preparation of stock solutions of LAS: One gram of pure LAS was poured in a one-liter flask, and a liter of distilled water was added until the volume reached one liter.
2. Preparation of LAS standard solution: 10 mL of the stock solution was added to distilled water until the solution reached the volume of 1,000 mL.
3. Preparation of concentrations of 0.5, 14, and 21 mg/L from the LAS standard solution.
4. LAS prepared at each concentration level including 0.5, 14, and 21 mg/L was combined with the amounts of ZnO nanoparticles containing 50, 100, and 150 mg/L. Then, each of these prepared solutions was placed on a magnetic mixer and put in the dark place for 20 min to reach equilibrium in the adsorption and desorption process in these solutions.

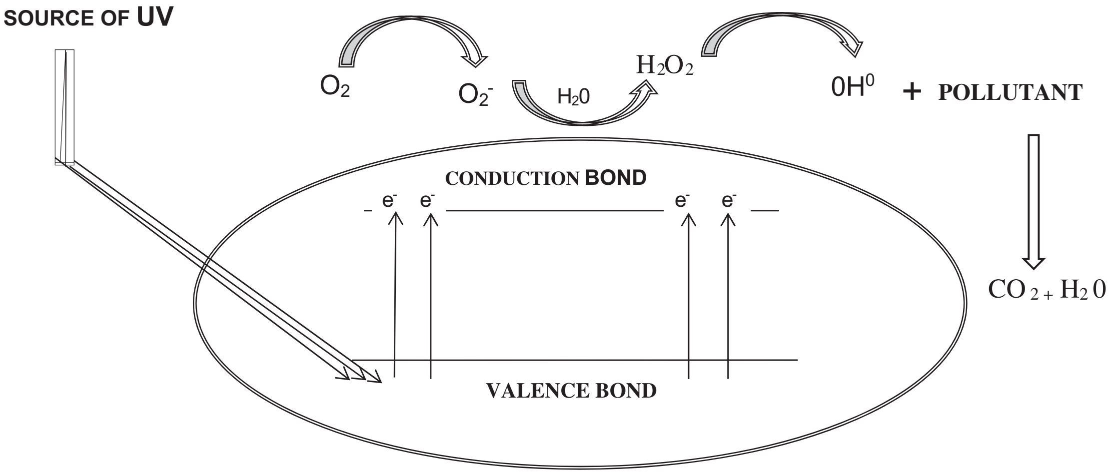

Fig. 2. The mechanism of photocatalytic process.

The use of ultraviolet radiation on solutions included three durations of 8, 16, and 24 min. The specification of UV lamp included power of 150 W, wavelength of 254 nm, and medium pressure. The pH values were set on 3, 7, and 11 using a pH-meter made in Germany by the amount of sodium hydroxide and hydrochloric acid to evaluate the degradation of LAS after a period of time. The samples were drawn out from the reactor in order not to interfere with the LAS separation. A 0.45-micron filter was used to separate ZnO nanoparticles; thus, the remaining amount of LAS was denoted by the methylene blue method with the presence of chloroform solvent (in this way, LAS reacts with methylene blue and builds up a blue-colored salt. The salt is dissolved in chloroform, and the resulting color intensity is proportional to that of the LAS concentration). Finally, to obtain the removal rates of LAS in the tested samples, a UNIQUE spectrophotometer made in America model 2100 with the power of 250.3 and 652 nm wavelength was used [30]. The properties of ZnO nanoparticles are shown in Table 1.

2.1. Statistical method

Experimental design was based on Taguchi statistical method. The factors were divided into two sets of experiments to be controlled (signal elements S) and uncontrollable (or noise disturbance factors N). In this method, in order to determine the best performing test, the signal-to-noise ratio analysis was used to achieve optimal response. The calculation of this ratio depends on what kind of optimization is required. Since the response factor used in this study was the percentage of LAS removal, the objective of the study was to maximize the response and the signal-to-noise ratio which can be determined using Eq. (1).

$$S/N = -10\ \log\frac{\frac{1}{y_1^2}+\frac{1}{y_2^2}+\dots\frac{1}{y_n^2}}{n} \qquad (5)$$

Eq. (5). SN Ratio Equation.

Y_n is the response which was measured for each experiment and is the number of occurrences of each test. In this study, the more efficient the signal-to-noise ratio, the more efficient the LAS removal is. In Taguchi method, variance analysis is used to determine error variance and the relative importance of each factor [31].

For analysis, the Minitab 16 software was used. The experimental design has been explained in Table 2 with 9 tests (L_9) including the value of each factor in each experiment.

3. Results and discussion

3.1. Effect of the initial concentration of nanoparticles in the UV/ZnO system efficiency of the LAS removal

In order to evaluate the different doses of ZnO nanoparticles, the samples prepared of LAS were combined with different doses of ZnO nanoparticle (50, 100, and 150 mg/L) in the presence of UV radiation. Then, after spending the required time for the absorbance of the samples, the spectrophotometer was used for measuring the absorbance. It was observed that with the increase in the nanoparticle concentration, the decomposition of LAS increased. This phenomenon can be explained by photocatalytic oxidation reactions on the catalyst surface. In addition, with the increase in nanoparticles, the catalyst surface and its active centers may also increase. All LAS molecules were adsorbed on the catalyst; thus, the LAS removal was done faster.

In the study conducted in 2004 on the removal of insecticide diazinon from contaminated water in the presence of the ZnO nanoparticles, Fadaei found that the optimum concentration of ZnO was about 150 mg/L [25]. We also found that the optimal amount of ZnO concentration is 150 mg/L (Fig. 3).

3.2. Effect of pH on the decomposition of LAS

When ZnO contacts with an aqueous solution, it may show an amphoteric behavior and its pH can be equivalent to 9. In the pH of less than 9, the oxide surface is positively charged, while in pH level more than 9, it is negatively charged. Therefore, the ZnO nanoparticle surface is positively charged in acidic

Table 1
Properties of ZnO nanoparticle

Product name	Purity percentage	The grain size	Specific surface	Density
ZnO nanoparticle	99.8%	12 nm	40–150 m^2/g	105 kg/m^3

Table 2
Experimental design of Taguchi method

Number of experimental	ZnO concentrations (mg/L)	pH of the solution	Contact time (min)
1	50	3	8
2	50	7	16
3	50	1	24
4	100	7	8
5	100	11	16
6	100	3	24
7	150	7	8
8	150	3	16
9	150	11	24

medium. In addition, since anionic surfactant (LAS) has a sulfonate (SO_3^-) group in its structure, it results in more absorption and surfactant degradation rate [32]. Undoubtedly, the surfactants adsorbed on the surface of the nanoparticles can enhance the reaction between hydroxyl radicals and carbon atoms in the benzene ring [33,34]. In a study in 2009 on the photocatalytic degradation of metamitron in ZnO water suspensions, Mijin and Savic found that the optimum pH was 3 [35]. In this study, as shown in Fig. 4, the optimum pH was also 3.

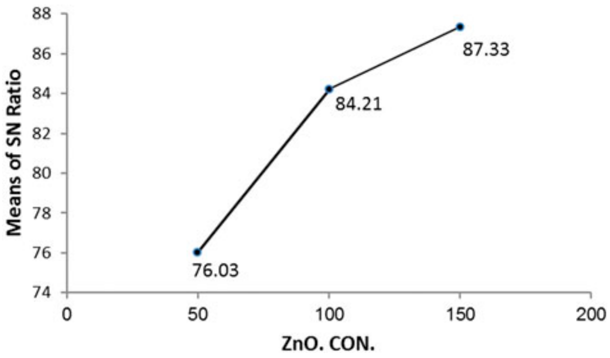

Fig. 3. Effect of ZnO concentration.

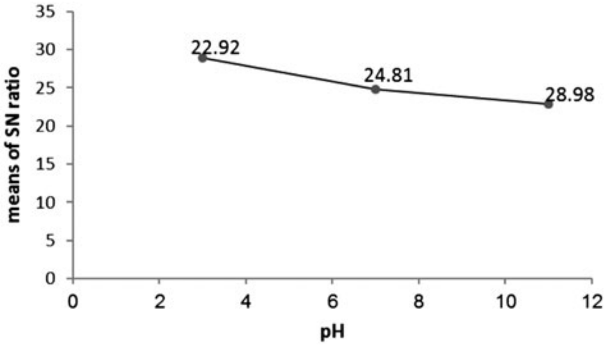

Fig. 4. Effect of pH in degradation of LAS.

3.3. Effect of contact time on LAS degradation

As shown in Fig. 5, by increasing the contact time, the rate of decomposition increases. In fact, by increasing the time, considerable levels of LAS degradation can be expected after 24 min. It was observed that the degradation efficiency can be enhanced by increasing the contact time [35,36]. In a study on the photocatalytic reduction of Cr(VI) in aqueous solutions by UV irradiation with the presence of titanium dioxide, Hidaka found that the degradation of Cr(VI) was increased by increasing the contact time [37].

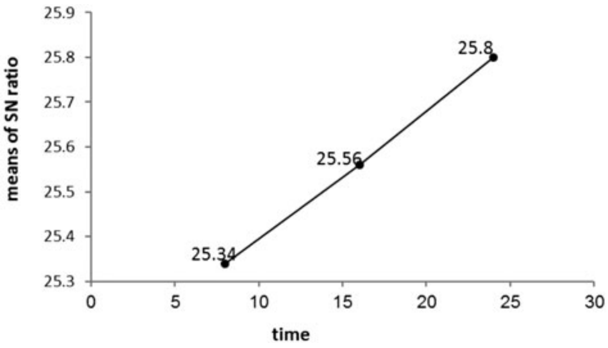

Fig. 5. Effect of contact time.

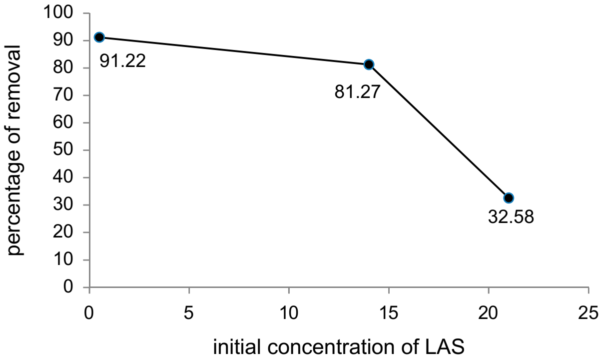

Fig. 6. Effect of initial concentration of LAS.

Table 3
Experimental number and there variables with removal efficiency

Experiment number	Variables ZnO-con (mg/L)	pH	time (min)	LAS removal efficiency (%)
1	150	7	8	74.12
2	150	3	16	81.27
3	150	11	24	78.94
4	100	7	8	75.14
5	100	11	16	71.14
6	100	3	24	78.00
7	50	11	8	66.52
8	50	7	16	68.16
9	50	3	24	71.22

3.4. Effect of the initial LAS concentration

Our findings showed that in the lowest concentration of 0.5 mg/L, the higher removal rate was achieved. However, in the highest concentration of 14 and 21 mg/L, the removal rate significantly decreased compared to the concentration of 0.5 mg/L (Fig. 6). Indeed, the high LAS concentration absorbs the most photons. Therefore, the available photons may decrease to activate the nanoparticles. Thus, the amount of conversion at the same time for samples with low concentration is more than the high concentration [37]. In a study in 2004 on the removal of insecticide diazinon from contaminated water in the presence of the ZnO nanoparticles, Dehghani found that the optimum LAS concentration was about 0.5 mg/L [29].

Table 3 shows the percentage of LAS removal at different times. The pH and ZnO concentration levels were tested at the selective LAS concentration of 14 mg/L. The highest removal rate was achieved at acidic pH, time of 24 min, and nanoparticle dose of 150 mg/L.

4. Conclusions

The results of this study showed that the removal efficiency rose by increasing the rates of ZnO nanoparticles, time, and initial LAS concentration. Furthermore, the removal rate in acidic solution was greater than other solutions. Three factors, time, ZnO nanoparticle concentration, and pH, had a significant effect on the removal rate. The highest removal efficiency was achieved upon maximum contact time in an acidic environment and when the amount of ZnO nanoparticles was 150 mg/L. Therefore, this method is suitable for the removal of surfactant because of its low cost, safety, and biocompatibility. Using the Taguchi statistical method reduced the number of tests; thus, it reduced the cost and testing time.

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