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INTEGRATING SONO-PHOTO-FENTON-LIKE AND NANO ZERO-VALENT IRON (NZVI) IN TNT YELLOW WATER TREATMENT

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ABSTRACT: Yellow water (YW) generated from the 2,4,6-Trinitrotoluene (TNT) production contains high concentrations of nitroaromatic compounds that pose serious risks to human health, ecosystems, and the environment. In this study, the removal of nitrotoluene compounds in yellow water treatment using the Sono-Photo-Fenton-like process combined with zero-valent iron nanoparticles (nZVI) was evaluated. The parameters of hydrogen peroxide dosage, nZVI dosage, ultraviolet power, and ultrasonic power on TNT degradation in yellow water treatment were investigated using the response surface methodology (RSM) to determine the optimal conditions. The findings demonstrated that at a pH of 3.0, with an initial TNT concentration of 80.8 mg L^{-1}, an nZVI dosage of 2 mM, a hydrogen peroxide concentration of 80 mM, an ultrasonic power of 60 W, and an ultraviolet power of 30 W, a TNT removal efficiency of 88.19% was attained after a reaction duration of 60 minutes.

Keywords: 2-4-6-Trinitrotoluen, Yellow water, Sono-Photo-Fenton-like, Nano ZVI, RSM

1. INTRODUCTION

2,4,6-Trinitrotoluene (TNT), commonly known as TNT, is one of the most widely used blasting explosives. It is synthesized by reacting toluene with a mixture of concentrated nitric and sulfuric acids. The process of making it has three steps: first, toluene is nitrated with the acid mixture to make mononitrotoluene (MNT); next, MNT is nitrated again to make dinitrotoluene (DNT); and finally, DNT is nitrated a third time to make TNT [1]. Most impurities are removed by washing process with hot water to remove the free acids, thus generating acidic yellow water. The crude TNT product typically contains small quantities of undesired TNT isomers and DNT isomers in addition to the target 2,4,6-isomer. These impurities must be removed to achieve the purity required for use as a blasting explosive. To accomplish this, the crude TNT is treated with an aqueous sodium bisulfite solution, followed by a settling process. This bisulfite treatment generated a by-product waste liquors known as alkaline red water [2]. Thus, the overall TNT production process results in the discharge of substantial amounts of wastewater containing persistent organic pollutants (POPs). These waste streams are primarily classified by color: acidic yellow water (YW) and alkaline red water (RW). Both types are highly contaminated with stable organic compounds, including TNT, DNT, MNT, and various nitrobenzene byproducts [3]. Typically, yellow water contains traces of TNT, residual acids, and unreacted toluene, while red water is enriched with asymmetric TNT isomers and other nitrobenzene derivatives [4]. Nitroaromatic compounds are known to exhibit acute and chronic toxicity, primarily targeting the nervous system and impairing blood function by disrupting oxygen transport.

Numerous studies have been conducted to treat persistent organic pollutants in aquatic environments [5-7]. Macroporous polystyrene resin RS 50B was employed as an adsorbent to treat red water [8]. Moreover, there have also been a lot of materials used to investigate the removal of TNT from red water such as bamboo charcoal, modified carbon adsorbents prepared from pine wood in chemical process and layered double hydroxides synthesized from red mud and brucite [3, 9, 10]. In other studies, various advanced oxidation processes (AOPs), including $O_3/H_2O_2/UV$, O_3/Fenton /UV, Fenton/H_2O_2/UV, Fenton/TiO_2/UV, $TiO_2/H_2O_2/O_3$/UV, TiO_2/O_3 /Fenton/UV, TiO_2/H_2O_2/Fenton/UV systems, were evaluated for red water treatment [11]. Recently, both the conventional Fenton process and the UV-Fenton process have been applied to reduce chemical oxygen demand COD and color in red wastewater [12]. However, few studies have been published on yellow water treatment. A Fenton reaction combined with zero-valent iron (ZVI) as a pretreatment stage was investigated for the degradation of yellow water [13]. This study found that the ZVI dosage used prior to the Fenton reaction could remove 100% of TNT, 100% of the organic nitrogen and 95.4% of the COD. In addition, the homogeneous Photo-Fenton process was evaluated for TNT removal from yellow water [14]. The highest TNT treatment efficiency reached 97.70% after reaction time of 120 minutes, while the concentration of COD in the wastewater decreased by 82.71% and Color index by 92.6%. The results showed high efficiency in treating yellow water,

particularly in terms of meeting key environmental parameters.

In recent years, Sono-Photo-Fenton-like process has been studied and recognized as an effective method for treating wastewater containing nitroaromatic compounds as well as other persistent organic pollutants like azure-B, phenol and dyes…[15-17]. In Sono-Photo-Fenton-like process, various radicals such as ●OH, ●H, and ●OOH are generated through the direct and synergistic effects of ultrasound and light, enabling the rapid degradation of organic pollutants. In the Fenton-like process, zerovalent iron nano particles (nZVI) have commonly been studied as a catalyst. nZVI can serve multiple roles, including acting as a reducing agent in direct reactions with nitro (-NO_2) contaminants and as a source for Fe^{2+} regeneration in the Fenton reaction [18]. The heterogeneous Sono-Photo-Fenton-like process combined with nZVI had demonstrated high potential for degrading nitrotoluene compounds in wastewater [19-21]. This process effectively overcame the limitations of the conventional Fenton process, such as high chemical agent consumption, excessive sludge generation, and limited iron reusability [22-24]. Hence, the aim of this study to find the optimum conditions to treat yellow water by the Sono-Photo-Fenton–like process combined with nZVI and to optimize the TNT removal efficiency in yellow water treatment by using Design-Expert 13 software.

The findings of this paper are structured as follows: (i) Comparison of the removal efficiencies of TNT, DNT, and MNT in a Sono–Photo–Fenton-like process combined with nZVI; (ii) Evaluation of influencing factors on the removal efficiency of TNT in yellow water using a Sono–Photo–Fenton-like process combined with nZVI; (iii) Study on the optimal conditions for treating TNT in yellow water using a Sono–Photo–Fenton-like process combined with nZVI.

2. RESEARCH SIGNIFICANCE

This study contributes to advancing wastewater treatment technology by integrating Sono-Photo-Fenton-like processes with nanoscale zero-valent iron (nZVI) for the degradation of nitrotoluene compounds in TNT yellow water. The combined process enhances hydroxyl radical generation, reduces chemical consumption, and minimizes sludge formation compared with conventional Fenton methods. The findings not only optimize operational parameters for improved treatment efficiency but also provide a scientific basis for practical applications in treating refractory nitroaromatic pollutants from explosive manufacturing wastewater, supporting sustainable and environmentally friendly industrial practices.

3. MATERIALS AND MOTHODS

3.1 Chemicals

2,4,6-Trinitrotoluene, 2,4-Dinitrotoluene (95%, Vietnam), p-nitrotoluene, $FeCl_3.6H_2O$, $NaBH_4$, NaOH, H_2SO_4 98% (Xilong Scientific, China), H_2O_2 (30%, Analytical Reagent, China), de-ionized water (Milli Q).

Zero-valent iron nanoparticles were produced at the laboratory scale through a chemical reduction method under atmospheric conditions as described in our previous study [20]. The particle size of nZVI typically ranges from 30 to 60 nm, exhibiting a classic core-shell structure.

To comparison of the degradation rate of TNT, DNT, and MNT by using Sono-Photo-Fenton-like process combined with nZVI, synthetic wastewater containing each compound at a concentration of 50 mg/L was prepared under pH 3.0 conditions.

To evaluate and optimize the treatment of actual wastewater by Sono-Photo-Fenton-like process using RSM method, the samples of yellow water was collected at a Chemical Factory in Vietnam. The typical characteristics of wastewater are pH of 1.5, COD of 360 mgL^{-1}, Color-index of 2100 Pt-Co and 80,8 mg L^{-1} of 2,4,6-Trinitrotoluene.

3.2 Sono-photo reactor

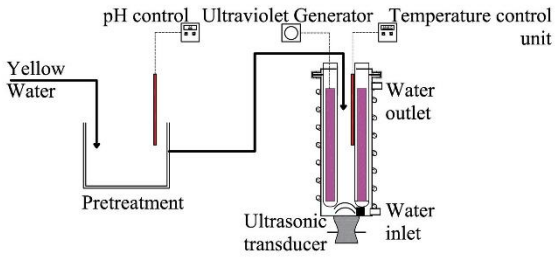

Fig. 1 Experimental diagram of the Sono-Photo-Fenton Method.

The experimental diagram is presented in Fig. 1. The experimental diagram of the Sono-Photo-Fenton process was developed with reference to the study by Nguyen Van Hoang et al. [20]. The TNT yellow water was adjusted to pH=3.0 in a pretreatment vessel. Then, the nZVI particles were added and stirred for 30 minutes. In the following, the yellow water was filled into Sono-Photo reactor. The reactor, with a total volume of 1.2 liters, was made of stainless steel. The ultrasonic transmitter was attached to the bottom of the reactor and connected to an ultrasonic generator with a frequency of 40 kHz and a variable intensity of 0-100W (Ultrasonic Generator). Four UV lamps (each with a power of 10 W and an emission wavelength of 254 nm) were installed inside the reactor to provide the required UV-C irradiation for

the photo-Fenton process. The temperature inside the reactor was monitored using a temperature sensor and controlled by a cooling water system. During the entire experiment, temperature and pH data were controlled, measured and collected.

3.3 Experimental Procedure

The removal efficiency of TNT, DNT, and MNT compounds using the Sono-Photo-Fenton-like system combined with zero-valent iron nanoparticles (nZVI) was investigated and compared through experiments with synthetic wastewater containing each compound separately. Each wastewater sample was prepared with an initial concentration of 50 mg/L, pH = 3.0, and H_2O_2 concentration of 40 mM. The operating conditions included an nZVI concentration of 2 mM, UV lamp power of 10 W, ultrasound frequency of 40 kHz, and ultrasonic power of 60 W.

The influence of light power (0–40 W), ultrasonic power (0–80 W), nano zero-valent iron (nZVI) dosage (1–5 mM), and H_2O_2 concentration (20–100 mM) on the treatment efficiency of actual yellow water were investigated using an experimental design in Design Expert 13 software. The volume of water used in each experiment is 1.0 liter. The response surface methodology (RSM) was employed using a central composite design (CCD) containing five center points to evaluate the interactive effects of the operating variables. The model and regression equation were evaluated for their fit to the experimental data using ANOVA (P-value = 0.05). A total of 30 experiments were conducted, including six replicates (Table 1).

Table 1. Elements for experimental design.

Variables	Unit	Symbol	Level				
			-2	-1	0	1	2
A: Light power (UV)	W	X_1	0	10	20	30	40
B: nZVI amount	mM	X_2	1	2	3	4	5
C: H_2O_2 amount	mM	X_3	20	40	60	80	100
D: Ultrasonic power (US)	W	X_4	0	20	40	60	80

3.4 Analytical Method

TNT, DNT and MNT concentration in the solution were analyzed by high performance liquid chromatography HPLC (Agilent, USA, 1100 Series) with Hypersil C18 column (200 x 4 mm), mobile phase of methanol and water 65/35, pH=7.

Treatment efficiency was evaluated through the concentration of nitrotoluene compounds. The treatment efficiency was calculated as follows:

$$H\% = \frac{C_0 - C_t}{C_0} x\ 100\ (\%) \quad (1)$$

Where: H was the treatment efficiency, C_o and C_t were the concentration of TNT, DNT or MNT at the initial time and after treatment in t time

4. RESULTS AND DISCUSSION

4.1 Removal Efficiency Of TNT, DNT, MNT In The Sono-Photo-Fenton-like System Process Combined With nZVI

The removal efficiency of TNT, DNT, and MNT in the Sono-Photo-Fenton-like process combined with nZVI was given in Fig. 2. The degradation rate of nitrotoluene compounds was compared under the same experimental conditions: an initial pollutant concentration of 50 mg/L, pH = 3.0, an nZVI dosage of 2 mM, an H_2O_2 concentration of 40 mM, a light power of 10 W, and an ultrasonic power of 60 W. The results showed that MNT degraded the fastest, achieving 100% removal efficiency within 20 minutes of treatment, whereas DNT required 30 minutes to reach a 100 % removal efficiency. The removal efficiency of TNT after 30 minutes of the Sono-Photo-Fenton-like system process combined with nZVI was only 90.1%. All pollutants were completely removed after 40 minutes of reaction.

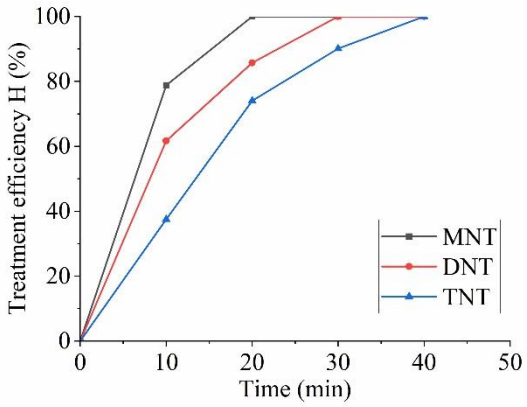

Fig. 2 Degradation efficiency of nitrotoluene compounds

The different degradation rates of MNT, DNT, and TNT under the same conditions depended on their chemical structures and the number of nitro groups (Fig 3). The degradation rate of nitroaromatic compounds by hydroxyl radicals decreased as the number of nitro groups increased, due to the electron-withdrawing effected [25]. Consequently, TNT exhibited lower degradation efficiency compared with DNT and MNT. Furthermore, the presence of three nitro groups in the TNT structure rendered it more chemically stable and more resistant to degradation.

The degradation mechanism of nitrotoluene compounds may involve nZVI particles generating electrons in water that reduced nitro groups in nitrotoluene compounds to amines. The degradation of explosive pollutants could occur via reductive

reactions, where nitro groups were reduced to amino groups [26]. The •OH radicals generated in the Sono-Photo-Fenton process abstract hydrogen atoms from the methyl group to form a methyl radical, which was subsequently oxidized into intermediate compounds. This was followed by the decarboxylation of aromatic acids, similar to the mechanism reported by Ming-Jer et al. [27]. Finally, hydrolysis and mineralization occurred, producing CO_2, H_2O, and other byproducts. MNT contained only one nitro group, while DNT and TNT contained two and three nitro groups, respectively. As a result, MNT required fewer reduction steps in the initial phase, allowing for a faster degradation process. In contrast, DNT and TNT, due to the presence of multiple nitro groups, undergo more reduction reactions to convert nitro groups into amino groups, thereby slowing down the overall degradation rate. Since TNT degraded more slowly than DNT and MNT, it provided favorable conditions for observing the degradation process when conducting optimization experiments. Hence, if TNT was completely degraded, then MNT and DNT would also be fully degraded.

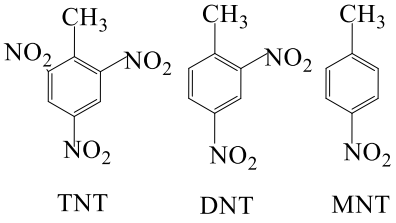

Fig. 3 The structure of 2,4,6-TNT, 2,4-DNT and p-MNT

4.2 Removal Efficiency Of TNT In Yellow Water By The Sono-Photo-Fenton-like Process Combined With nZVI

The experimental results were analyzed using regression analysis based on the collected data, and a second-order polynomial equation was proposed by the software for modeling. The reduced quadratic model and the ANOVA results were presented in Eq. (2).

$$H = -58.832 + 2.432\,X_1 + 35.547\,X_2 + 0.375\,X_3 + 1.331\,X_4 - 0.242\,X_1X_2 + 0.025\,X_1X_3 - 0.0008\,X_1X_4 + 0.096\,X_2X_3 - 0.117\,X_2X_4 + 0.003\,X_3X_4 - 0.051\,X_1^2 - 5.069\,X_2^2 - 0.008\,X_3^2 - 0.012\,X_4^2; \quad (2)$$

Where X_1, X_2, X_3, and X_4 represent the variables as defined in Table 1.

The F-value of 62.3 implies that the model is significant, while a P-value of less than 0.0001 indicated model terms are significant. Additionally, the predicted R^2, adjusted R^2, and overall R^2 values were 0.9052, 0.9673, and 0.9831 respectively, indicating that the regression equation was statistically significant and capable of predicting the effects of the factors on treatment efficiency.

4.2.1 Effect of ultrasonic (US) power on treatment efficiency

The effect of ultrasonic power on treatment efficiency is presented in Fig. 4c. It could be observed that under the conditions of pH = 3.0, initial TNT concentration of 80.8 mg/L, nZVI concentration of 3 mM, H_2O_2 concentration of 60 mM, UV power of 20 W, and without ultrasonic, the treatment efficiency was only 53.95%. When the ultrasonic power was increased to 20 W, the efficiency rose to 74.02%. The highest efficiency of the Sono-Photo-Fenton-like process was achieved at an ultrasonic power of 40 W, reaching 82.2%. At 60 W, the efficiency slightly decreased to 80.7%, and at 80 W, the efficiency dropped further to 70.57%.

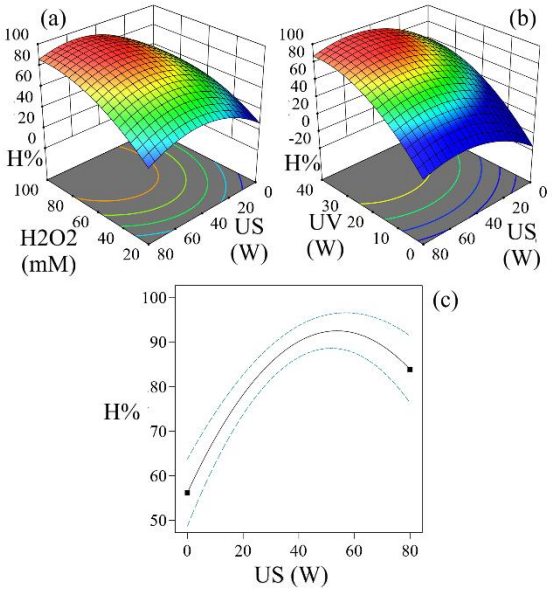

Fig. 4 Combined effect of US power and H_2O_2 concentration (a), combined effect of UV – US power (b), effect of ultrasonic (US) power (c**)** on treatment efficiency

As ultrasonic power increased, the catalyst surface was continuously cleaned by ultrasound, leading to an increased active surface area, which enhanced the generation of hydroxyl radicals (●OH) [28]. Higher power levels also result in the formation of more cavitation bubbles, shortening their lifespan and causing rapid collapse, thereby producing more ●H and ●OH radicals, which enhanced reactions with pollutant molecules [29, 30]. Additionally, the increase in ultrasonic power facilitated better mass transfer and more uniform catalyst dispersion within the Sono–Photo–Fenton-like system [31, 32]. However, excessive ultrasonic power could raise the solution temperature, leading to the decomposition of hydrogen peroxide, and therefore, negatively

impacting treatment efficiency [20].

Fig. 4b illustrated that an increase in both UV and US power led to improving TNT degradation efficiency. The optimal treatment efficiency was achieved when ultrasonic power ranged from 40 to 60 W and UV power ranged from 30 to 40 W. Meanwhile, Fig. 4a showed that, an increase in H_2O_2 concentration led to an improvement in treatment efficiency at the optimal US power of 40-60 W.

4.2.2. Effect of UV power on treatment efficiency

Fig. 5c demonstrated the effect of UV power on the efficiency of TNT in yellow water by Sono-Photo-Fenton-like process. When the UV power increased from 10W to 20W, the treatment efficiency rose from 66.2% to 82.2%. In the following, when the UV power continued to increase to 30W and 40W, the treatment efficiency reached 88.4% and 87.9%, respectively.

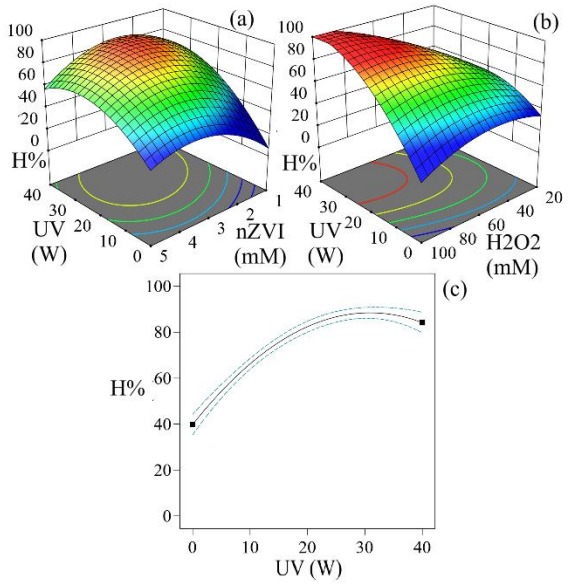

Fig. 5 Combined effect of UV power and nZVI concentration (a), combined effect of UV power and H_2O_2 concentration (b), effect of UV (c) on treatment efficiency.

Increasing light intensity promoted the formation of hydroxyl radicals on the catalyst surface, thereby improving treatment efficiency [20]. Fig. 5a illustrated the combination effect of UV power and nZVI concentration on treatment efficiency. The results indicated that when both factors were increased simultaneously, the treatment efficiency also increased. Under the effect of UV irradiation, Fe^{3+} ions were continuously reduced to Fe^{2+}, which enhanced the treatment efficiency of the Fenton process.

$$Fe^{3+} + H_2O + UV \rightarrow Fe^{2+} + \bullet OH + H^+ \quad (3)$$

Fig. 5b showed the change in treatment efficiency under the combined effect of UV power and H_2O_2 concentration. When UV power was increased in combination with higher H_2O_2 concentration, the efficiency of organic matter treatment increased due to the photolysis of H_2O_2 generating ●OH radicals (Eq. (4)).

$$H_2O_2 + UV \rightarrow \bullet OH + \bullet OH \quad (4)$$

4.2.3. The effect of the initial nZVI amount on treatment efficiency

Fig. 6a depicted the treatment efficiency of acidic yellow water at different nZVI concentrations from 1 mM to 5 mM. The results indicated that increasing the dosage of nano-material from 1 mM to 3 mM improved TNT removal efficiency from 59.3% to 82.2%. However, the treatment efficiency gradually decreased to 64.93% as the nZVI concentration increased to 5 mM. It can be seen that iron played an important role in promoting the reaction through its role in the formation of hydroxyl radicals, thereby affecting the efficiency of organic matter degradation by Fenton-like process [33]. The effectiveness of zero-valent iron in conventional Fenton systems is often hindered by its poor dispersion and limited catalytic efficiency, which restrict large-scale application [18]. Therefore, higher doses of zero-valent iron were required, leading to increased treatment costs. During the Sono-Photo-Fenton-like process, under the effect of ultrasound, iron nanoparticles were well dispersed in the environment, thereby increasing treatment efficiency [34]. At the same time, increasing the material dosage enhanced the formation of cavitation bubbles on the material surface, thereby improving the generation of free hydroxyl radicals [32]. However, treatment efficiency may decrease when using higher material concentrations as it can lead to the reaction of iron ions and hydroxyl radicals formed on the material surface, thereby reducing reaction efficiency [35].

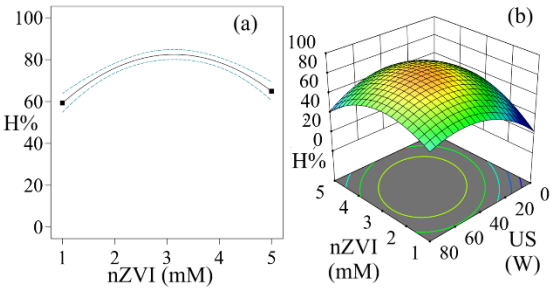

Fig. 6 Effect of nZVI concentration (a), combined effect of Ultrasonic power and nZVI concentration (b)

Fig. 6b demonstrated the variation in treatment efficiency under the combined influence of ultrasonic power and nZVI concentration. The results indicated that the highest efficiency were reached by the nZVI

concentration of 2 mM to 4 mM and the ultrasonic power between 40 W and 80W

4.2.4. The effect of the H_2O_2 dosage on treatment efficiency

Fig. 7a showed the effect of H_2O_2 concentration from 20 mM to 100 mM on the treatment efficiency. It was observed that TNT treatment efficiency increased with higher H_2O_2 concentration. When the initial hydrogen peroxide dosage ranged from 20 to 80 mM, TNT treatment efficiency increased from 56.43% to 85.2%. However, when the concentration reached 100 mM, the pollutant removal efficiency decreased to 82.10%.

Adding higher amount of H_2O_2 led to a higher rate of ●OH radical generation, thereby increasing the TNT degradation rate. In aqueous environments, H_2O_2 could decompose to form •OH radicals via a thermochemical mechanism, UV absorbed, Fenton process … However, excessive H_2O_2 may reduce TNT treatment efficiency due to side reactions. The reduction in treatment efficiency at higher H_2O_2 concentrations was due to reaction of hydrogen peroxide as a free radical scavenger (Eq. (5)) [31].

$$^{\bullet}OH + H_2O_2 \rightarrow HOO^{\bullet} + H_2O \quad (5)$$

Fig. 7b illustrates the effect of nZVI concentration at different H_2O_2 concentration. The degradation efficiency of TNT increased with higher nZVI concentrations, reaching maximum efficiency when the H_2O_2 concentration was also optimized. For instance, at an nZVI concentration of 1 mM, the optimal H_2O_2 concentration was approximately 60 mM, while at the nZVI concentration of 2-3 mM the optimal H_2O_2 concentration ranged from 80 to 100 mM.

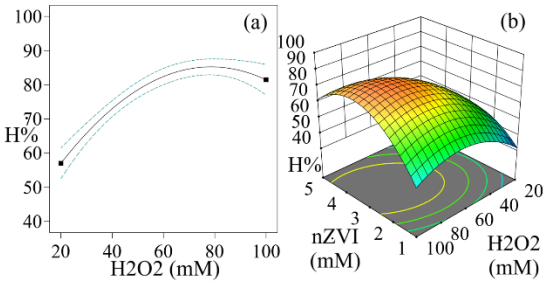

Fig. 7 Effect of H_2O_2 concentration (a), combined effect of H_2O_2 concentration and nZVI concentration (b)

4.3 Optimization Of TNT Removal In Yellow Water Treatment

In this study, the optimal conditions were determined using Design-Expert 13 software, aiming to balance economic feasibility due to high TNT removal efficiency in yellow water treatment. Fig. 8 presented one of the 100 solutions suggested by the software, with a desirability value of 1.0 with an ultrasonic power of 60 W, UV power of 30 W, nZVI concentration of 2 mM, and an initial H_2O_2 concentration of 80 mM.

By evaluating the treatment efficiency of yellow water under optimal conditions, it was observed that after 60 minutes the TNT removal efficiency reached 88.19%, while DNT and MNT were completely degraded. Moreover, the chemical oxygen demand (COD) of yellow water decreased by 60% after treatment.

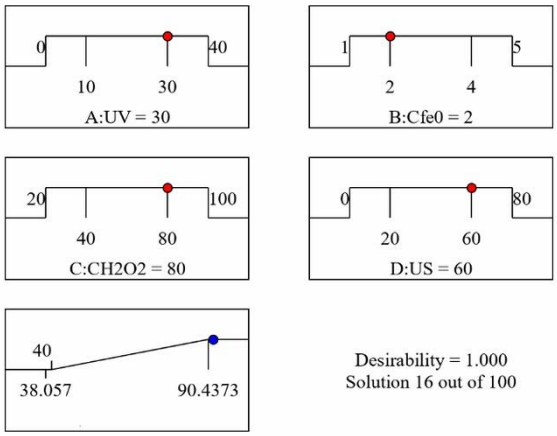

Fig. 8. Optimal setting for the numerical optimization of factors affecting the yellow water treatment by Sono-Photo-Fenton-like process, generated by the software

The application of the homogeneous Photo-Fenton process for treating acidic wastewater generated from the TNT production line achieved about 71% efficiency at pH=3, C_{Fe2+} = 7.19 mM, C_{H2O2} = 140 mM, and after 60 minutes [14]. Meanwhile, using the Fenton process combined with nZVI concentration of 1 gram/liter and pH=3, achieved about 89% treatment efficiency for TNT in acidic yellow water [13]. In this study, at the same time with 2 mM nZVI, 80 mM H_2O_2, the treatment efficiency reached 88.19%. Thus, the Sono-Photo-Fenton-like process combined with nZVI not only provided comparable or superior treatment efficiency to previous studies but also used fewer chemicals.

5. CONCLUSION

The study evaluated the treatment efficiency of acidic yellow water from the TNT production line using the Sono-Photo-Fenton-like process combined with nZVI. In particular, the study assessed the effects of parameters such as light power, ultrasonic power, nZVI concentration, and H_2O_2 concentration on treatment efficiency of TNT in yellow water by using the Response Surface Methodology. From these results, the optimal operating conditions for

TNT removal was determined. Under the optimal conditions at pH 3.0, nZVI concentration 2 mM, hydrogen peroxide concentration 80 mM, light power 30W, and ultrasonic power 60W, the TNT treatment efficiency in this study reached 88.19%, which is comparable to the results of previous studies. Moreover, Sono-Photo-Fenton-like process combined with nZVI can reduce chemical consumption, reduce reaction time, resulting in less sludge production. These initial research results may provide a basis for proposing a potentially feasible Sono-Photo-Fenton-like process for practical application.

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