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Parameters controlling the advanced oxidation degradation kinetics of nitroglycerin and pentaerythritol tetranitrate

DOI 10.1515/gps-2016-0210
Received November 25, 2016; accepted February 13, 2017; previously published online April 1, 2017

Abstract: Several advanced oxidation processes have been performed for the decomposition of ester nitrates (ENs), such as nitroglycerine (NG) and pentaerythritol tetranitrate (PETN). The reaction kinetics for removing NG and PETN by some of the advanced oxidation processes (e.g. UV-H_2O_2, Fenton, UV-Fenton) followed the pseudo-first-order model. The reaction rates in different systems followed the sequence ENs/UV < ENs/H_2O_2 < ENs/UV-H_2O_2 < ENs/Fenton < ENs/UV-Fenton. The effect of various parameters, such as pH, concentration of hydrogen peroxide, and temperature, on the degradation of NG and PETN were studied.

Keywords: advanced oxidation process (AOP); Fenton; nitroglycerin (NG); pentaerythritol tetranitrate (PETN); UV-Fenton; UV-H_2O_2.

1 Introduction

Nitroglycerin (NG) and pentaerythritol tetranitrate (PETN) are chemical compounds found in the family of ester nitrates (ENs). They have important explosive properties and, therefore, are applied in the field of explosives and propellants [1]. NG is used as the main ingredient in explosives and propellants, whereas PETN is a commonly used explosive in a mixture with hexogen (RDX) to ignite explosive reactions. A high level of NG is highly toxic to microorganisms, fishes, mice, and humans [1]. With long-term exposure to NG, humans can experience chronic headaches, rapid heartbeat, nausea, or vomiting. Similar to NG, PETN is very harmful to the environment. The toxicity of PETN is higher than that of RDX and it is classified as toxic to fishes and other organisms [2]. The United States Department of Defense has classified NG and PETN as highly dangerous components because of their widespread use and their impacts on the environment. Hence, finding ways to safely decompose EN compounds (including NG) in the environment is an urgent issue. However, the solubility of PETN is much less than that of NG, so water pollution by NG is a more urgent problem that must be prioritized.

At present, common methods for decomposing NG and other ENs compounds into less harmful or nontoxic substances have been proposed, such as hydrolysis in alkaline media [3, 4], decomposition by using zero-valence iron $Fe^{(0)}$ or nano FeO [1, 5], biological degradation [6, 7], or using pyrite and magnetite [8]. Although advanced oxidation processes (AOPs) have been widely studied and used to treat persistent organic pollutants, including different explosive compounds like nitrotoluenes (NTs) [9–13], nitramines (NAs) [14, 15], and nitrophenols (NPs) [15, 16], the application of AOPs for the degradation of NG and PETN is very limited. In addition, the oxidation decomposition of NG and PETN by AOPs in the presence of radiation, such as UV-H_2O_2 or photo-Fenton (UV-Fenton) have recently attracted the interest of researchers in the field of energetics and kinetics of reactions. The effects of key parameters on the degradation of NG and PETN in different systems may serve as bases upon which favorable conditions for the treatment of such explosives can be chosen.

The goal of this study is to examine the effectiveness and kinetic characteristics of the degradation of NG and PETN by several AOPs, such as UV-H_2O_2, Fenton's reagent, and UV-Fenton's reagent. For comparison, the control reactions of the decomposition of these two ENs using UV or H_2O_2 were identified.

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2 Materials and methods

2.1 Materials

Liquid NG, crystalline PETN, $FeSO_4 \cdot 7H_2O$, and hydrogen peroxide (30% w/w) were of analytical grade. All solvents, including acetonitrile (ACN), ethanol, methanol, and hexane (Merck KGaA, Germany) were at HPLC grade. De-ionized water was used to prepare the aqueous solutions. All chemicals were used as received without any further purification.

2.2 Instruments

All photochemical reactions were performed in a home-built photoreactor system, which was built following the procedure described elsewhere [15]. The concentrations of NG (liquid) and PETN were determined by using Agilent HPLC HP 1100 (Waldbronn, Germany) equipped with a diode array detector (DAD). The conditions of the HPLC measurements are listed in Table 1. An OAKTON pH meter, series 510 (Cole-Parmer Instrument Co., USA) was used to adjust the pH of the prepared solutions.

2.3 Methodology

In the oxidation of organic compounds (RH) by OH radical generated from Fenton's reagent, the degradation kinetic study followed the pseudo-first-order model with an assumption that the concentration of hydroxyl radical was much higher than that of RH [17]. The equation is given by

$$\ln \frac{C_{\mathrm{RH}(t)}}{C_{\mathrm{RH}(0)}} = -k'_{\mathrm{RH}} t, \qquad (1)$$

where $C_{\mathrm{RH}\,(t)}$ and $C_{\mathrm{RH}\,(0)}$ are the concentrations of RH at any time t and at initial $t = 0$ of the reactions, respectively, and k'_{RH} is the pseudo-first-order reaction rate constant. This model has also been applied in previous works for determination of k'_{RH} [11–13, 15, 16, 18].

3 Results and discussion

3.1 Kinetics of NG and PETN degradation in various advanced oxidation systems

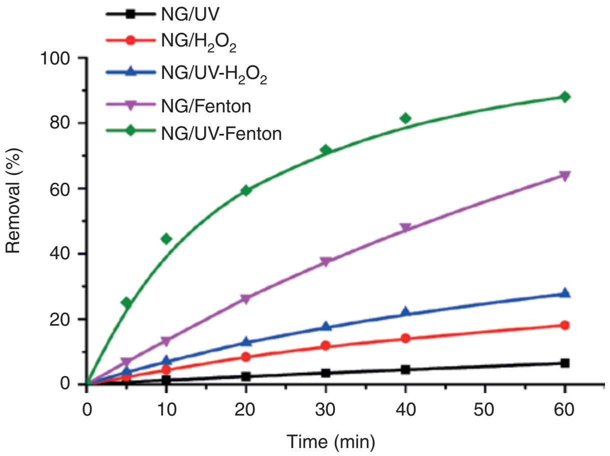


Figure 1: Efficiency of NG removal vs. time in different advanced oxidation systems.
$[NG]_o = 0.228$ mM; $[Fe^{2+}]_o = 0.4$ mM; $[H_2O_2]_o = 10$ mM; pH = 3; $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

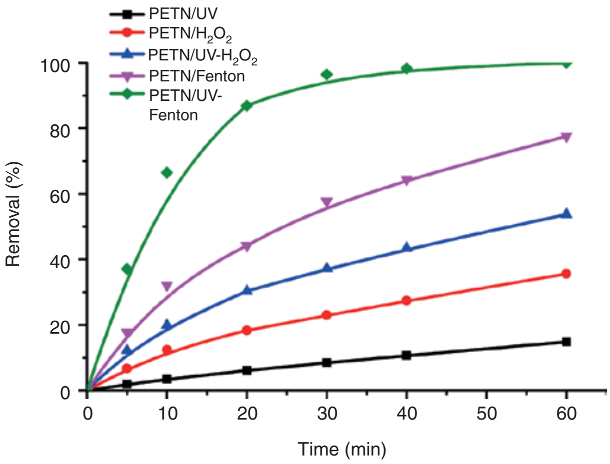


Figure 2: Efficiency of PETN removal vs. time in different advanced oxidation systems.
$[PETN]_o = 0.056$ mM; $[Fe^{2+}]_o = 0.4$ mM; $[H_2O_2]_o = 10$ mM; pH = 3; $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

Figures 1 and 2 present the respective efficiencies of the degradation of NG and PETN in various oxidation systems (ENs/UV, ENs/H_2O_2; ENs/UV-H_2O_2, ENs/Fenton, and ENs/UV-Fenton). Clearly, the combination of the oxidative agents (i.e. H_2O_2 or Fenton) with UV radiation increased the efficiency of NG degradation (Figure 1). Therefore, the conversion effectiveness of NG in different systems followed the sequence (at 40 min) given by NG/UV (~6%) < NG/H_2O_2 (~20%) < NG/UV-H_2O_2 (~30%) < NG/Fenton (~60%) < NG/UV-Fenton (~100%). A similar trend for PETN degradation processes was also observed: PETN/UV (15%) < PETN/H_2O_2 (38.5%) < PETN/UV-H_2O_2 (50.3%) < PETN/Fenton (72.8%) <PETN/UV-Fenton (100%).

Table 1: Experimental conditions for detecting NG and PETN by HPLC.

Conditions	NG	PETN
Mixture ratio (ACN/H_2O)	70/30 (v/v)	70/30 (v/v)
Flow rate (ml · min^{-1})	1	1
Retention time, t_R (min)	3.86	4.56
Detection wavelength, λ (nm)	215	204

In the case of UV alone, the light radiation interacted directly with EN molecules to dissociate into species thanks to the photon energy. However, the yield of the reaction was very low [19] due to the low quantum yield of the direct photodecomposition of ENs. In the case of oxidative reagent H_2O_2, the conversion efficiency was higher, because it was a strong oxidative agent. Its oxidation potential ($E_{ox}=1.78$ V) was only lower than those of ozone ($E_{ox}=2.07$ V) and $^{\bullet}OH$ ($E_{ox}=2.80$ V) [20]. However, EN-saturated compounds contained a nitrogen atom at the highest oxidation state (+5), so their ability to react with H_2O_2 proved to be limited. The combination of UV and H_2O_2 generated hydroxyl radical $^{\bullet}OH$ due to the photolysis reaction of H_2O_2 [21], consequently increasing the effectiveness of the degradation reaction, as given by

$$H_2O_2 + h\nu \rightarrow 2^{\bullet}OH. \tag{2}$$

In the presence of ferrous ions, hydrogen peroxide molecules were decomposed rapidly as a result of a catalytic activity [17, 22], as presented below.

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO^- + {}^{\bullet}OH \quad (k=63\ \text{M}^{-1}s^{-1}) \tag{3}$$

$$Fe^{3+} + H_2O_2 \rightarrow Fe^{2+} + H^+ + {}^{\bullet}O_2H \quad (k=3.1\times10^{-3}\text{M}^{-1}s^{-1}) \tag{4}$$

Under UV exposure, there exist three simultaneous reactions: direct photolysis of ENs by UV radiation, direct oxidation of ENs by H_2O_2, and EN decomposition reaction by $^{\bullet}OH$ radical generated from the photocatalytic activity.

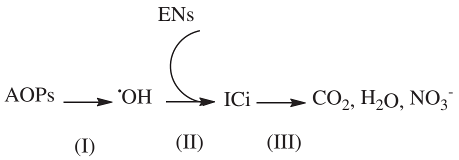
$$Fe^{3+} + H_2O + h\nu \rightarrow Fe^{2+} + H^+ + {}^{\bullet}OH \tag{5}$$

or

$$[Fe^{3+}(OH^-)]^{2+} + h\nu \rightarrow Fe^{2+} + {}^{\bullet}OH \tag{6}$$

It should be noted that $[Fe^{3+}(OH)^-]^{2+}$ absorbs photons significantly stronger than H_2O_2 at the wavelength of 254 nm (molecular absorption coefficient of $[Fe^{3+}(OH)^-]^{2+}$ is in the range of 1500–3500 ($l\cdot mol^{-1}\cdot cm^{-1}$) and that of H_2O_2 is 18.6 ($l\cdot mol^{-1}\cdot cm^{-1}$) [21]. Therefore, UV-Fenton is the most effective system for destroying ENs among the investigated AOPs.

The kinetics of NG and PETN decomposition in various advanced oxidation systems (ENs/AOPs) can be described by using the pseudo-first-order law, which is a general model for the oxidation of organic compounds, including some highly explosive compounds by $^{\bullet}OH$ radical [12, 16, 17, 23]. The $^{\bullet}OH$ radical is a strong and non-selective oxidative species that can react with many organic substances (the rate constant of 10^8-10^{11} $\text{M}^{-1}s^{-1}$) [24]. The oxidation process of ENs by $^{\bullet}OH$ radical also includes three main stages as follows:



where AOPs means advanced oxidation processes that can create hydroxyl radicals (e.g. UV-H_2O_2, Fenton, and UV-Fenton); ICi are the intermediates generated from the reaction of EN with $^{\bullet}OH$ radical; and CO_2, H_2O, NO_3^- are the final products of the EN mineralization process. Stage (II) determines the rate of EN degradation. The reaction rate constants can be determined using Equation (1).

Figures 3 and 4 present the plots of $-\ln(C/C_o)$ against time (t) of the decomposition of NG and PETN. The

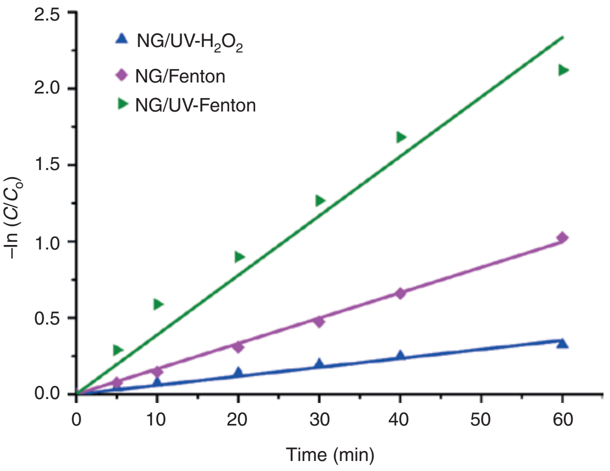


Figure 3: The plot of $-\ln(C/C_o)$ vs. time in different systems. NG/UV-H_2O_2; NG/Fenton; NG/UV-Fenton; $[NG]_o=0.228$ mM; $[Fe^{2+}]_o=0.4$ mM; $[H_2O_2]_o=10$ mM; pH = 3; $\lambda=245$ nm; $I_{UV}=1100$ LUX, $T=298$ K.

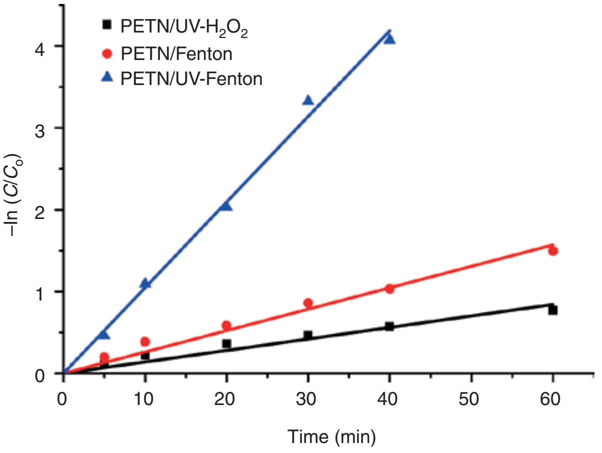


Figure 4: The $-\ln(C/C_o)$ vs. time in different systems. PETN/UV-H_2O_2; PETN/Fenton; PETN/UV-Fenton; $[PETN]_o=0.056$ mM; $[Fe^{2+}]_o=0.4$ mM, $[H_2O_2]_o=10$ mM; pH = 3; $\lambda=245$ nm; $I_{UV}=1100$ LUX, $T=298$ K.

Table 2: Influence of H_2O_2 concentration on the degradation of NG and PETN in different AOPs.

$[H_2O_2]_o$ (mM)	k'_{NG} (min^{-1})			k'_{PETN} (min^{-1})		
	UV-H_2O_2	Fenton	UV-Fenton	UV-H_2O_2	Fenton	UV-Fenton
10	0.0059	0.0166	0.0389	0.0140	0.0262	0.1047
40	0.0063	0.0140	0.0655	0.0155	0.0229	0.1137
160	0.0072	0.0123	0.0481	0.0160	0.0204	0.1113

$[NG]_o = 0.228$ mM; $[PETN]_o = 0.056$ mM; $[Fe^{2+}]_o = 0.4$ mM; pH = 3, $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

pseudo-first-order reaction rate constant k' was determined. The results are listed in Table 2.

Many parameters affect the efficiency and kinetics of the advanced oxidation degradation of ENs, such as concentrations, pH, and temperature. Understanding how they influence the decomposition of EN explosives enables us to choose favorable conditions for the treatment of ENs.

3.2 Influence of H_2O_2 concentration

The influence of hydrogen peroxide concentration on the rate of NG degradation reaction is shown in Figure 5. Pseudo-first-order rate constants of NG and PETN decomposition were determined and collated in Table 2.

We noticed that when H_2O_2 was added into the NG solutions (systems NG/H_2O_2 and NG/UV-H_2O_2), the rate of NG degradation slightly increased with the increase of H_2O_2 concentration (Figure 5 and Table 2). However, in Fenton and photo-Fenton systems, the reaction rate first increased with the addition of H_2O_2, and then reduced when the hydrogen peroxide concentration was 160 mm.

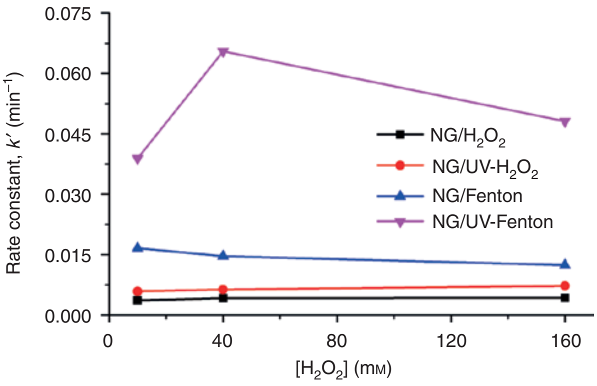


Figure 5: Influence of initial H_2O_2 concentration on NG decomposition by different AOPs.
$[NG]_o = 0.228$ mM; $[Fe^{2+}]_o = 0.4$ mM; pH = 3; $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

A similar effect of hydrogen peroxide on the rate of PETN destruction was observed (Figure 5 and Table 2).

Higher initial hydrogen peroxide concentration enhanced reactions (2) and (3), after which it increased the amount of hydroxyl radical and increased the speed of reaction. However, when the concentration of hydrogen peroxide was in excess, it turned into a scavenger that consumed $^\bullet OH$ radicals, resulting in the decrease of the ENs degradation rate [25] given by

$$^\bullet OH + H_2O_2 \rightarrow H_2O + HO_2^\bullet. \qquad (7)$$

Based on obtained results, H_2O_2 concentration of 10 mM was selected for further studies.

3.3 Influence of pH

The effect of pH on the oxidation of organic compounds by radiation (UV) or hydrogen peroxide is often insignificant. Hence, we considered the degradation efficiency varying with the change of pH from 2 to 5 in the EN/Fenton and EN/UV-Fenton systems (Figure 6).

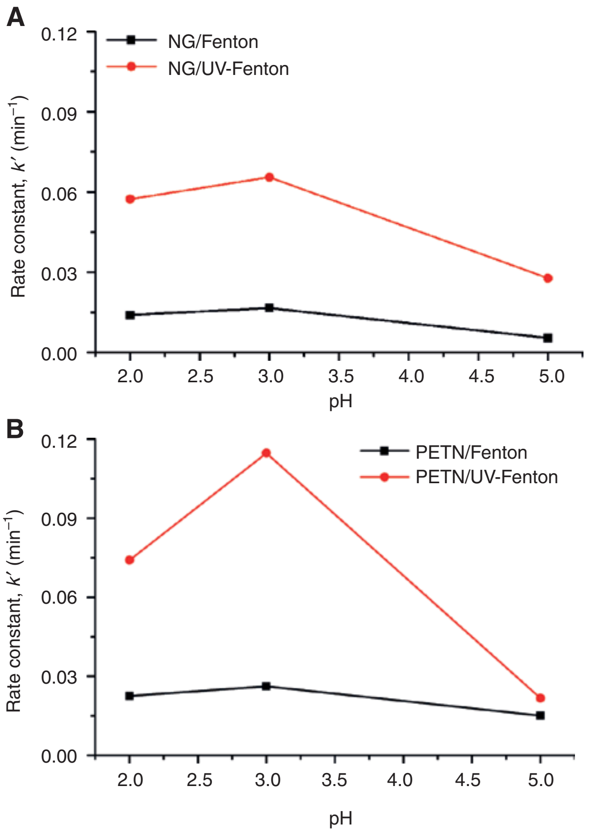


Figure 6: Effects of pH on the degradation of (A) NG and (B) PETN.
$[NG]_o = 0.228$ mM; $[PETN]_o = 0.056$ mM; $[Fe^{2+}]_o = 0.4$ mM; $[H_2O_2] = 10$ mM; $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

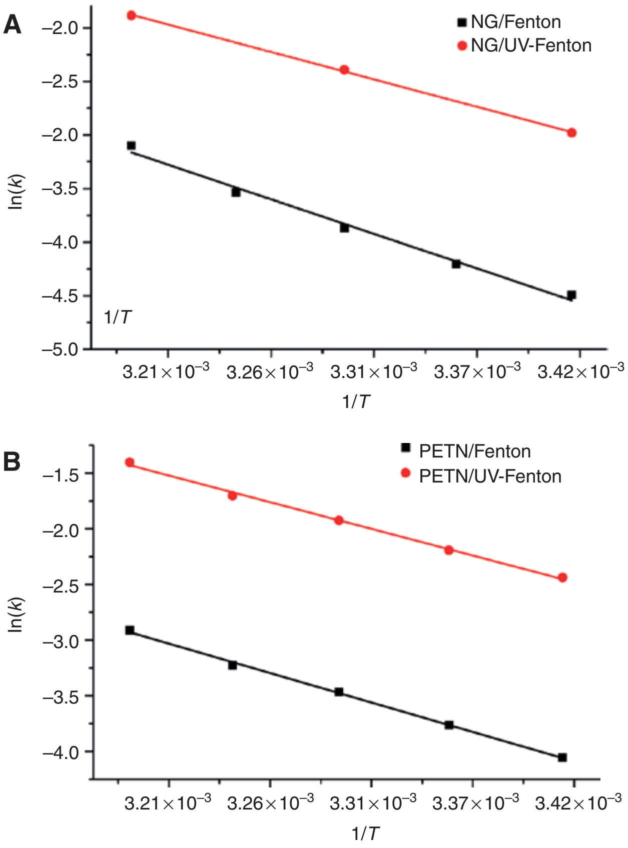


Figure 7: Effects of temperature on the degradation of (A) NG and (B) PETN using Fenton and UV-Fenton. $[NG]_o = 0.228$ mм; $[PETN]_o = 0.056$ mм $[Fe^{2+}] = 0.4$ mм; $[H_2O_2] = 10$ mм; pH = 3; $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

The reaction rates gained the maximum value at pH 3 for decomposing both NG and PETN. The explanation can be given as follows: ferrous ion in Fenton (or UV-Fenton) served as a catalyst for the decomposition of H_2O_2 to generate hydroxyl radical, as shown in reactions (3) and (4). Due to the hydrolysis of ferrous ions, the oxidation resulting from the use of Fenton systems was more favorable in an acidic medium. However, pH < 2.5 favors the formation of $[Fe(H_2O)]^{2+}$ complexes [22, 25, 26], resulting in the decrease of free ferrous ions taking part in (3), which in turn, can reduce the production of hydroxyl radicals. Moreover, at low pH, the radical $^{\bullet}OH$ can be easily scavenged by hydrogen peroxide (6), reducing the concentration of this active species that is mainly in charge of the oxidation reaction. The optimal range of pH for most Fenton reactions is around the value of 3. At higher pH, ferrous ion is not very stable, and can be easily oxidized to form ferric ion and then ferrous hydroxyl complexes [25] due to hydrolysis. These complexes cannot react with H_2O_2 to recycle initial ferrous ions, resulting in the decrease of $^{\bullet}OH$ formation, which is then followed by the reduction of oxidation rate. The higher the pH level, the easier the occurrence of the hydrolysis process. At pH 5, most ferric ions are either hydroxyl complexes or even precipitates to form $Fe(OH)_3$, so the reaction rate sharply decreases. For studying other effects, experiments were carried out at pH 3.

3.4 Influence of temperature

Figure 7 presents the influence of temperature on EN decomposition by the Fenton and UV-Fenton systems. As can be seen, the increasing temperature in the range of 293–313 K had a promotional effect on all investigated advanced oxidation systems. The linear plots of ln(*k*) against 1/*T* indicated that the processes followed an Arrhenius-type behavior in the considered temperature range.

The activation energies and energetic parameters were calculated from Figure 7 and Eyring equation. The results are shown in Table 3. Obviously, among these advanced oxidation processes, UV-Fenton is the most effective system for the treatment of wastewater polluted by NG and other EN compounds, such as PETN [27].

4 Conclusion

The primary goal of this paper was to study the degradation of NG and PETN by such advanced oxidation

Table 3: Activation energies and energetic parameters for the degradation of NG or PETN using different AOPs.

Energetic parameters	Nitroglycerin (NG)			Pentaerythritol tetranitrate (PETN)		
	UV-H_2O_2	Fenton	UV-Fenton	UV-H_2O_2	Fenton	UV-Fenton
E_a (kJ/mol)	74.86	52.59	41.71	68.56	43.10	39.02
$\Delta H^{\neq}$ (kJ/mol)	72.39	50.11	39.23	66.09	70.57	36.50
$\Delta S^{\neq}$ (J/mol · K)	−44.7	−111.5	−135.6	36.1	−140.0	−140.6
$\Delta G^{\neq}$ (kJ/mol)	85.71	83.34	79.64	55.32	82.33	78.42

$[NG]_o = 0.228$ mм; $[PETN]_o = 0.056$ mм; $[Fe^{2+}]_o = 0.4$ mм; $[H_2O_2]_o = 10$ mм; pH = 3, $\lambda = 245$ nm; $I_{UV} = 1100$ LUX, $T = 298$ K.

processes as UV, H_2O_2, UV-H_2O_2, Fenton, and photo-Fenton reactions. The conversion yields and the rates of NG and PETN decomposition depend on the reactivity of the advanced oxidation activity (i.e. the capability to generate hydroxyl radical) of the oxidative systems. The efficiency of EN degradation increases with the sequence given by UV < H_2O_2 < UV-H_2O_2 < Fenton system < UV-Fenton system. The controlling parameters of degradation kinetics were also investigated.

The kinetics for the degradation reactions followed the pseudo-first-order rule, which is consistent with the general model for oxidation organic compounds, such as nitramine [15] or nitrotoluen [13] using AOPs systems.

Acknowledgments: The financial support of this work by the Vietnam National Foundation for Science and Technology (NAFOSTED 104.04-2014.25) is gratefully acknowledged by the authors.

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