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RESEARCH ARTICLE

Evaluation of the catalytic effect of potassium tungstate in green decontamination for detoxification of 2-chloroethyl phenylsulfide (2-CEPS)

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Abstract

Objectives. 2-Chloroethyl phenylsulfide (2-CEPS) is a relevant simulant of chemical warfare sulfur mustard gas (yperit) as part of an environmentally-friendly decontamination processes. This study presents the initial results of research the catalytic ability of tungstate in the conversion process of 2-CEPS.

Methods. The decontamination system employed in this study comprised hydrogen peroxide (H₂O₂), potassium tungstate acting as a metal transition salt catalyst, a surfactant, and organic solvents. The research investigated the impact of K₂WO₄ concentration on the conversion efficiency and rate of the target compound. As well as additionally exploring the influence of the substrate-to-catalyst ratio on the reaction pathway, the study evaluated the stability of the detoxifying mixture.

Results. Increasing the concentration of K₂WO₄ is shown to lead to an increase in the efficiency and conversion rate of 2-CEPS. As well as demonstrating stability and durability, the catalyst did not cause unwanted H₂O₂ breakdown. After 18 h of mixing, the conversion retained efficiency above 95% within 15 min of the reaction. The degradation kinetics follow a pseudo-first-order model, indicating that the reaction rate is directly influenced by the K₂WO₄ concentration. In addition to enhancing the oxidative capacity of the solution, increased tungstate concentration promotes the formation of undesirable sulfone byproducts.

Conclusions. The study investigated the catalytic activity of tungstate within an eco-friendly solution formulated to degrade 2-CEPS. Our findings demonstrate a strong correlation between the concentration of potassium tungstate (K₂WO₄) and the rate of 2-CEPS degradation. A key advantage of tungstate is its exceptional stability and durability as a catalyst. Efficient decontamination is ensured thanks to its minimal interference with the stability of hydrogen peroxide (H₂O₂).

Keywords

2-CEPS, yperit, chemical warfare, green decontamination solution, tungstate, catalysis

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НАУЧНАЯ СТАТЬЯ

Оценка каталитического эффекта вольфрамата калия в экологически безопасной детоксикации 2-хлорэтилфенилсульфида (2-CEPS)

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Аннотация

Цели. Представлены первые результаты изучения каталитической способности вольфрамата в процессе превращения 2-хлорэтилфенилсульфида (2-CEPS) — аналога иприта, сернистого иприта — в рамках экологически безопасных методов дезактивации.

Методы. Система дезактивации включала пероксид водорода (H_2O_2), вольфрамат калия (катализатор на основе соли переходного металла), поверхностно-активное вещество и органические растворители. Исследовалось влияние концентрации K_2WO_4 на эффективность и скорость превращения целевого соединения. Дополнительно изучалось соотношение субстрата и катализатора, а также стабильность детоксицирующей смеси.

Результаты. Показано, что увеличение концентрации K_2WO_4 приводит к росту эффективности и скорости превращения 2-CEPS. Катализатор продемонстрировал стабильность и долговечность, не вызывая нежелательного разложения H_2O_2 . После 18 ч перемешивания степень конверсии сохранялась выше 95% в течение 15 мин реакции. Кинетика деградации соответствует модели псевдопервого порядка, что указывает на прямую зависимость скорости реакции от концентрации K_2WO_4 , однако повышение концентрации вольфрамата способствует образованию нежелательных сульфоновых побочных продуктов.

Выводы. Исследована каталитическая активность вольфрамата в экологически безопасном растворе, разработанном для деградации 2-CEPS. Установлена четкая зависимость между концентрацией K_2WO_4 и скоростью разложения 2-CEPS. Ключевое преимущество вольфрамата — его исключительная стабильность и долговечность в качестве катализатора, а также минимальное влияние на стабильность H_2O_2 , что обеспечивает эффективную дезактивацию.

Ключевые слова

2-хлорэтилфенилсульфид, иприт, химическое оружие, экологичный раствор для обеззараживания, вольфрамат, катализ

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INTRODUCTION

Although chemical decontamination processes based on chlorine-containing substances such as hypochlorite and chloramine are effective and cost-efficient, there are still negative impacts on human health and the environment [1]. The potential use of such compounds is additionally limited due to their high corrosiveness, which can damage equipment, weapons, and storage facilities.

Green chemistry, also known as clean chemistry or sustainable chemistry, attracts significant attention from scientists, economists, and politicians. Green chemistry focuses on designing and producing environmentally

friendly products while minimizing the use and creation of hazardous substances [2]. The environmental friendliness of chemicals can be categorized into edible, contactable, and approved for use in agriculture and industry [3]. H_2O_2 is an ideal oxidizing agent, capable of oxidizing with an atomic efficiency of 47%, producing water as the only available theoretical product, and safe for storage, transportation, and use. H_2O_2 has high oxidizing properties, especially when combined with a suitable catalytic activator. Generally, there are four groups of commonly used catalytic activators: organic activators, metal ion activators, metal complexes (biomimetic), and metal salt activators. Metal salt activators are especially

interesting due to their diversity and strong catalytic abilities [4]. In 2003, Ryoji Noyori used tungstate as a catalyst for strengthening the oxidation reaction with H_2O_2 . Tungstate is physiologically harmless and does not cause the decomposition of H_2O_2 [5]. In 2010, George W. Wagner demonstrated the use of molybdate metal salt activator as a catalyst to create the peroxy anion (OOH) for accelerating the oxidizing ability of H_2O_2 [6]. Environmentally-friendly organic solvent components Triton X-100 (TX-100), solvent propylene carbonate (PC), and propylene glycol (PG), were used in respective volume ratios of 10%, 10%, and 20% [7].

In the present study, we used 30% H_2O_2 as an oxidizing agent to detoxify 2-CEPS along with potassium tungstate as the catalyst/activator and organic solvents containing TX-100, PC, and PG in respective volume ratios of 10%, 10%, and 20%. Several factors affecting efficiency, speed, and conversion direction were investigated. The catalyst/substrate ratio was shown to greatly influence the conversion process and product formation trends; the tungstate catalyst in the decontamination mixture demonstrated high stability, durability, and minimal unwanted decomposition of H_2O_2 .

MATERIALS AND METHODS

Chemical and equipment

Chemical

2-CEPS (98%) by *Sigma-Aldrich* (USA); H_2O_2 (30%), TX-100 (99%) by *Merck* (Germany); PC (99%), PG (99%), K_2WO_4 (99%), Na_2CO_3 (99%), Na_2SO_3 (99%) by *Macklin* (China); chloroform, methanol with suitable purity for high-performance liquid chromatography (HPLC) by *Fisher Scientific* (United Kingdom); distilled water.

Equipment

HPLC HP-1100 chromatograph (*Agilent Technologies*, USA), chromatographic column C8 (250 mm \times 4.6 mm \times 5 μ m), UV-VIS diode array (DAD) detector with scan range of 0–1100 nm; Agilent 5975 gas chromatography-mass spectrometer (GC–MS *Agilent Technologies*, USA), DB-5MS column (30 m \times 0.32 mm \times 0.25 mm); Pioneer™ precision balances *Ohaus*, USA) with 0.0001 g sensitivity; MS2 minishaker (USA).

Methods

2-CEPS degradation and analytical methods

The 2-CEPS concentration during the reaction was determined according to the HPLC method. Elution was isocratic with a flow rate of 1.0 mL/min using a mixture of methanol and diluted water (70/30, v/v). At an injection

volume of 10 μ L, the detection wavelength was 252 nm, and the retention time was 7.188 min.

The intermediates produced during the degradation of 2-CEPS were analyzed using GC–MS chromatography. Helium with a purity above 99.999% was applied as the carrier gas at a constant flow rate of 1.0 mL/min and pressure of 60 psi. The injection was implemented in splitless mode over 1.0 min, and the injection volume was 1.0 μ L. The shunt flow was set at 50.0 mL/min. The carrier gas saving time and flow rate were 2.0 min and 20.0 mL/min, respectively. The inlet temperature was held at 280°C. The optimized initial temperature of the oven was set at 40°C for 1 min, then increased at a rate of 10°C per minute to 280°C for 5 min. The substances were identified by comparing the mass spectra of the analytes with the NIST mass spectral library (*National Institute of Standards and Technology*, USA) and using fragment-matching methods [7].

Experimental setup

The reactions were carried out in 10-mL capped test tubes at room temperature (25°C). After taking organic solvents of 100 μ L of TX-100, 100 μ L of PC, and 200 μ L of PG [3, 5], decontamination solutions were added to the test tube to make up a total volume of 1 mL. Then 20- μ L 2-CEPS was added to the test tube (at a volume ratio between decontamination and reactant of 50 : 1) and the tube was agitated. At 1, 5, 10, 15, and 30-min time points, 59 μ L of the sample was added to a test tube containing 1 mL of a reaction quenching mixture containing 0.2 M Na_2SO_3 and 0.2 M Na_2CO_3 and agitated. Chloroform of 2 mL was then added to the mixture and agitated for 2 min [8]. The extracted solution was filtered through a 0.22 μ m filter and analyzed using HPLC to record the concentration of 2-CEPS. Intermediates were analyzed by GC–MS. After repeating each experiment 3 times, the average value was taken.

The removal efficiency (η , %) was calculated using the following formula:

$$\eta = \frac{C_0 - C_t}{C_0} \times 100\%,$$

where C_0 and C_t (ppm) are the concentrations of 2-CEPS in before and after treatment, respectively.

RESULTS AND DISCUSSION

Analyzing the effect of K_2WO_4 concentration on reaction time and conversion efficiency

Since decontaminants used in military contexts require rapid toxicity conversion, the reaction rate and conversion efficiency are the most important factors. To analyze the effect of K_2WO_4 concentration on the

degradation process of 2-CEPS, the concentration of H_2O_2 was fixed at 4.32 M, while K_2WO_4 concentrations were varied between 0 M, 0.0005 M, 0.00075 M, 0.001 M, and 0.0025 M. The results are shown in Figs. 1 and 2 and Table 1.

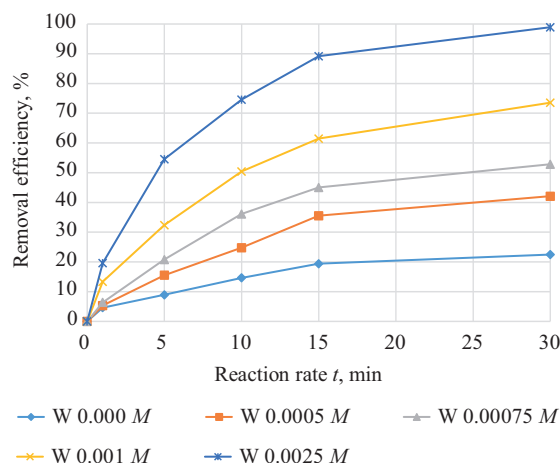


Fig. 1. Effect of K_2WO_4 concentration on removal efficiency of 2-CEPS

As shown in Fig. 1, at a fixed concentration of H_2O_2 , the removal rate of 2-CEPS depends on the concentration of K_2WO_4 . In the case without K_2WO_4 , the conversion reaction of 2-CEPS proceeds slowly, recording an efficiency of 22.47% after 30 min. In the cases with K_2WO_4 , the conversion rate increases significantly, reaching a maximum of 98.92% after 30 min at a concentration of 0.0025 M.

According to the analyzed K_2WO_4 concentration condition (Fig. 2), $\ln(C_t/C_0)$ decreases linearly over time (t), implying that the conversion reaction of 2-CEPS

Table 1. Relationship between K_2WO_4 concentration (C_{WO_4}) and the rate constant of removing 2-CEPS reaction (K)

C_{WO_4}	K	K/C_{WO_4}
0.00050	0.0281	56.2
0.00075	0.0400	53.3
0.00100	0.0620	62.0
0.00250	0.1429	57.2

is based on the pseudo-first-order kinetic reaction. At the same time, Table 1 shows that the ratio K/C_{WO_4} is relatively constant, around 53.3 to 62.0, implying that the reaction rate constant is first-order with respect to the concentration of K_2WO_4 . This can be explained in terms of the case with K_2WO_4 , where the oxidation sulfide compound process by H_2O_2 based on the metal W has two stages:

Stage 1. During the aqueous phase, the catalyst precursor K_2WO_4 is rapidly oxidized by H_2O_2 , forming a bis-peroxo wolfram complex [5, 9].



In this stage, the molar ratio of K_2WO_4/H_2O_2 was analyzed from 1/8640 to 1/1728. Therefore, the rate of forming complex depends on the concentration of K_2WO_4 , and it can be assumed that the total K_2WO_4 has been converted to the peroxo complex.

Stage 2. The peroxo complex approaches and oxidizes the sulfide. When adding 2-CEPS, because of the volume is equal to 1/50 of the decontaminant, it quickly dissolves into the solution, supporting the peroxo to contact and react easily.

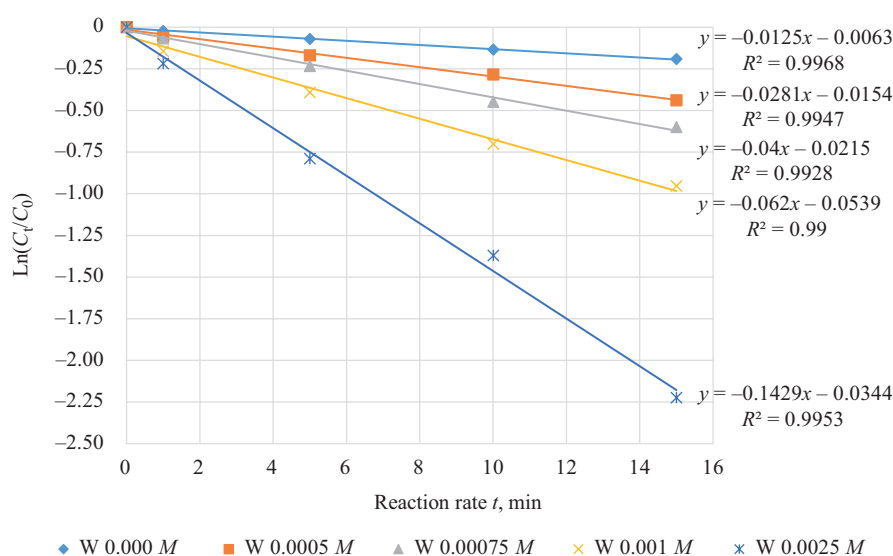


Fig. 2. Kinetic chart of $\ln(C_t/C_0)$ vs t

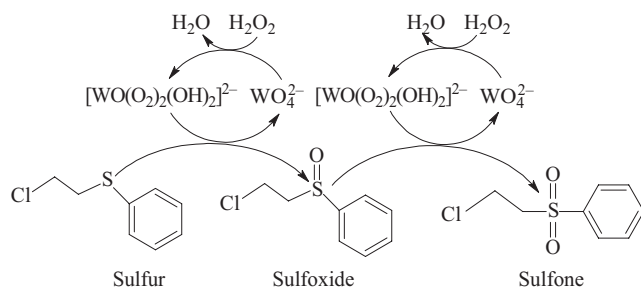


Fig. 3. 2-CEPS conversion process

In Stage 2, the molar ratio of peroxy/2-CEPS is from 1/267 to 1/54. Initially, the reaction rate records almost unchanged at this ratio due to its dependence on the peroxy concentration. However, when the concentration of 2-CEPS starts to decrease, the reaction rate becomes dependent on both the concentrations of peroxy and also 2-CEPS.

In addition to the effect of kinetic factors, some dynamic factors may also affect the conversion process, such as the velocity of movement of molecules in the solution, spatial effects, and the reaction ability between the peroxy complex and 2-CEPS in the solution.

Thus, the detoxification of 2-CEPS in solution highly depends on the concentration of K_2WO_4 . At low enough concentrations, the reaction equation follows pseudo-first-order kinetics, and the rate constant is first-order depending on the concentration of K_2WO_4 .

Analyzing the effect of the catalyst/substrate ratio on the detoxification process trend

The aim of the oxidation decontamination yperite process is to convert it into compounds with lower toxicity, especially sulfoxide, which has lower toxicity and does not cause blistering. At the same time, this process must avoid producing sulfone, a reaction intermediate that has high toxicity and can be formed over the oxidation process [10]. To determine the effect of the catalyst/substrate ratio leads to the trend of producing conversion product 2-CEPS, we performed the experiments as described in the Methods section with $[\text{H}_2\text{O}_2] = 4.32 \text{ M}$ and $[\text{K}_2\text{WO}_4]$ concentrations of 0.0025 M , 0.01 M , and 0.09 M . The conversion products 2-CEPS were identified using the GC–MS spectral library after 15 min. The results from the GC–MS identified 6 product peaks at retention times of 14.491, 15.591, 16.956, 18.321, 18.539, and 18.736 min. Based on the mass spectra identified in Fig. 4, the substances have ion fragments with corresponding unique m/z , peak 1: ethenylsulfinyl benzene (77, 104, 125, 152); peak 2: ethenylsulfonyl benzene (77, 104, 125, 168); peak 3: methyl phenyl sulfone (65, 77, 78, 141, 156); peak 4: methylsulfinyl benzene (77, 109, 125, 172); peak 5: 2-chloroethyl sulfonyl benzene (63, 77, 125, 141, 204); peak 6: 2-phenylsulfonyl ethanol (77, 109, 125, 141), where peaks 1 and 4 are the sulfoxide family, while peaks 2, 3, 5, and 6 are the sulfone family.

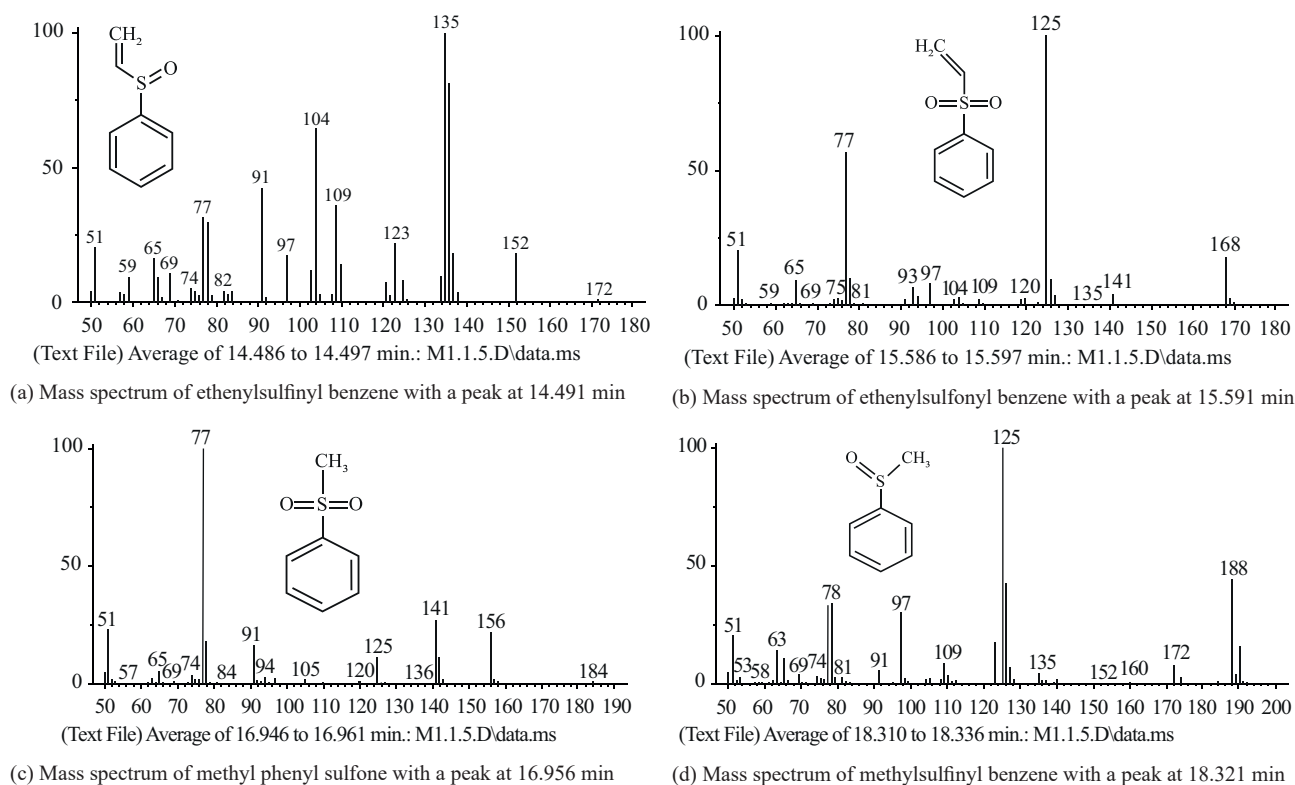


Fig. 3. 2-CEPS conversion process

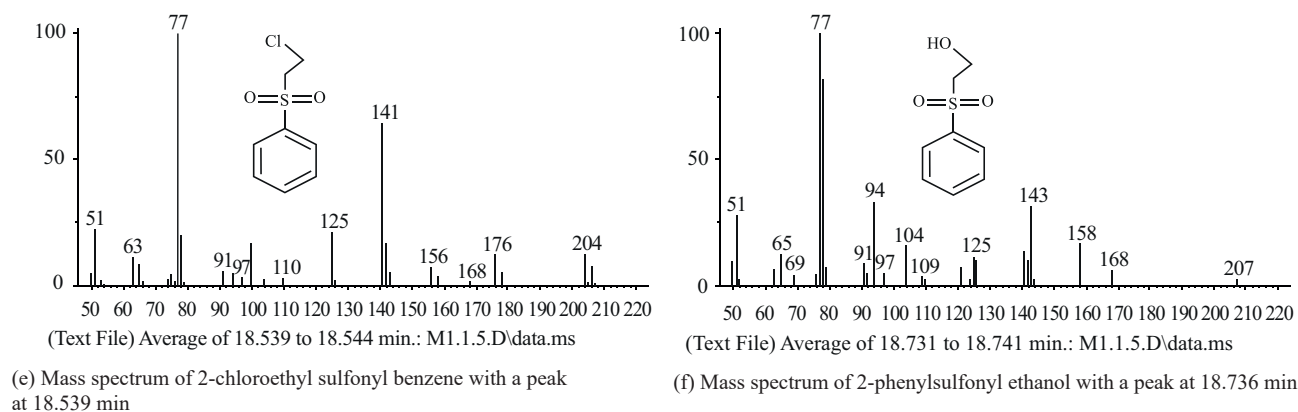


Fig. 3. (Continued). 2-CEPS conversion process

As well as showing the retention time, the GC-MS in Fig. 5 visually illustrates the effect of the catalyst/substrate ratio on the formation of products. Specific data on peak height and the relative percentage of sulfoxide and sulfone family products are presented in Table 2.

According to the results given in Fig. 5 and Table 2 when the concentration of K_2WO_4 increases, the sulfoxide family products decrease from 77.59% to 6.35%, while the sulfone product family increases from 22.41% to 93.65%. This can be explained in terms of the formed bisperoxotungstate compound (A)

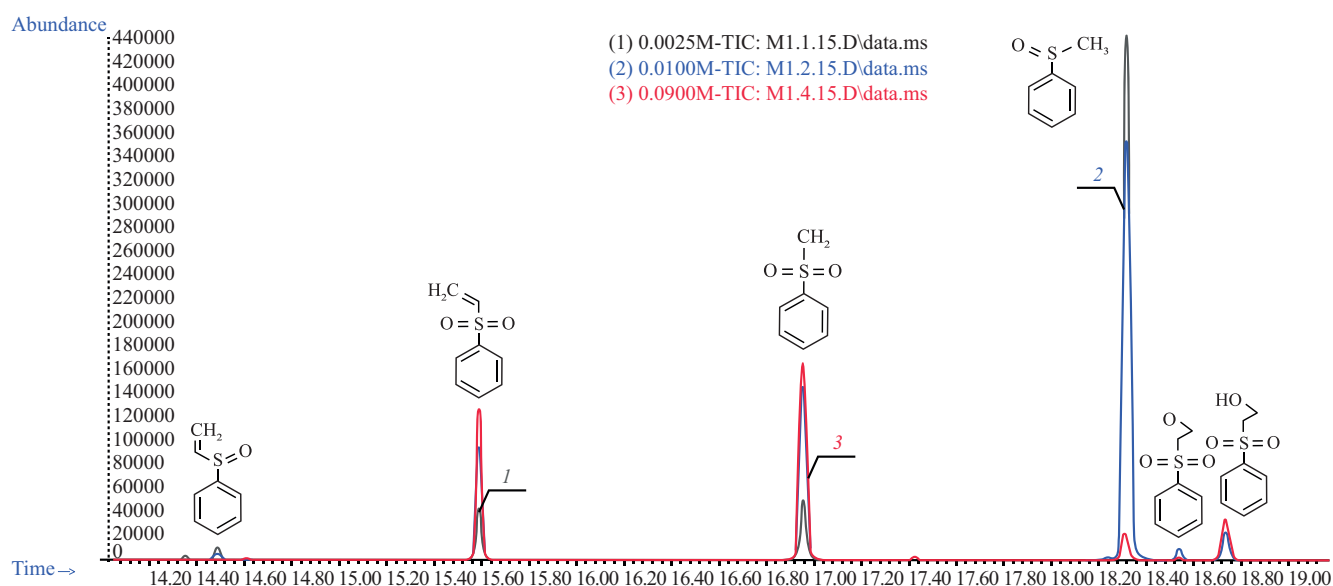


Fig. 5. GC-MS of mixture products after 15 min converting 2-CEPS

Table 2. Peak height and relative percentage of sulfoxide and sulfone products

K_2WO_4 concentration (C_M)	Peak height, h						% Sulfoxide	% Sulfone
	$h_{Peak\ 1}$	$h_{Peak\ 2}$	$h_{Peak\ 3}$	$h_{Peak\ 4}$	$h_{Peak\ 5}$	$h_{Peak\ 6}$		
0.0025	10^4	$45 \cdot 10^3$	$5 \cdot 10^4$	$44 \cdot 10^4$	10^4	$25 \cdot 10^3$	77.59	22.41
0.0100	$6 \cdot 10^3$	$95 \cdot 10^3$	$15 \cdot 10^4$	$36 \cdot 10^4$	10^4	$25 \cdot 10^3$	56.66	43.34
0.0900	0	$13 \cdot 10^4$	$168 \cdot 10^3$	$23 \cdot 10^3$	$3 \cdot 10^3$	$38 \cdot 10^3$	6.35	93.65

$$\begin{array}{c}
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 \begin{array}{c} \text{HO} \quad \text{O} \quad \text{O} \\ \diagdown \quad \diagup \quad \diagdown \\ \text{O} - \text{W} - \text{O} \\ \diagup \quad \diagdown \quad \diagup \\ \text{O} \quad \text{OH} \end{array} & \xrightleftharpoons[\text{-H}^+]{\text{H}^+} & \begin{array}{c} \text{HO} \quad \text{O} \quad \text{O} \\ \diagdown \quad \diagup \quad \diagdown \\ \text{O} - \text{W} - \text{O} \\ \diagup \quad \diagdown \quad \diagup \\ \text{O} \quad \text{O} - \text{H} \end{array} \\
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 \xrightleftharpoons[\text{-H}^+]{\text{H}^+}
 \begin{array}{c} \text{H} \quad \text{O} \quad \text{O} \\ \diagdown \quad \diagup \quad \diagdown \\ \text{H} - \text{O} - \text{W} - \text{O} \\ \diagup \quad \diagdown \quad \diagup \\ \text{O} \quad \text{O} - \text{H} \end{array}
 \end{array}
 \quad \text{K}^+ \quad \text{C}$$

Reaction time t (min)	Mo 0 h (%)	Mo 6 h (%)	Mo 12 h (%)	W 0 h (%)	W 18 h (%)	W 22 h (%)
0	0	0	0	0	0	0
1	44	19	-1	8	10	10
5	81	55	13	55	43	43
10	98	88	29	88	85	75
15	100	95	42	95	95	90
30	100	98	55	100	100	95

99% conversion efficiency when used after 15 min. Six hours after mixing, the conversion efficiency at 15 min of reaction remains around 95%; however, after 12 h of mixing, it drops to about 42%. In contrast, the oxidation system based on the K_2WO_4 catalyst shows excellent stability: 18 h after mixing at 15 min of reaction, the recorded conversion efficiency was still over 95%. After 22 h of mixing, the conversion efficiency significantly decreases: in this case, the reaction rate is slower than the oxidation system based on the K_2MoO_4 catalyst because K_2WO_4 is chemically stable and unconvertible in the decontamination environment. The tungstate complex is especially suitable for use as a pre-catalyst because it does not cause the inefficient decomposition of H_2O_2 [16].

CONCLUSIONS

The catalytic ability of tungstate in the green chemical decontamination solution for converting 2-CEPS has been investigated. The results of the study show that the conversion process of 2-CEPS in the solution is highly dependent on the concentration of K_2WO_4 . The degradation is based on a pseudo-first-order kinetic law; the reaction rate constant is first-order depending on the concentration of K_2WO_4 . Increasing the concentration of the tungstate catalyst implies raising the oxidative capacity of the decontaminant, but also leads to a trend of producing unwanted sulfone products. Additionally, tungstate is an effective, highly durable catalyst, which even more importantly does not cause inefficient decomposition of H_2O_2 .

Authors' contributions

Nguyen Thanh Hoa—conceptualization of the research paper, critical revision with the introduction of valuable intellectual content.

Do Ngoc Khue—idea of a new method, concept of the study, and planning consultations.

Nguyen Khanh Hung—experimental studies and participation in writing of the text of the article.

Dao Duy Hung—experimental studies and participation in writing of the text of the article.

All authors have read and approved the final version of the manuscript.
The authors declare no conflicts of interest.

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