

Characterization of Continuous Neutralization of a Chemical Warfare Agent and Its Simulants

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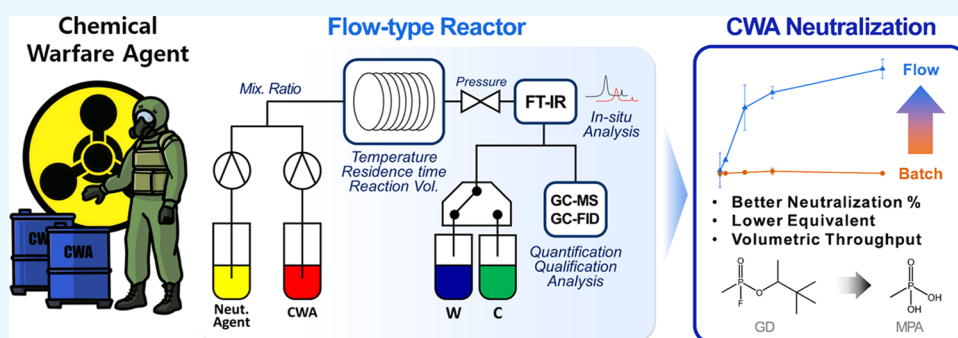
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ABSTRACT: The persistent threat posed by chemical warfare agents (CWAs) necessitates the development of efficient and safe methods for their neutralization. In this study, we investigated the continuous neutralization of CWAs and their simulants using flow chemistry, which combines the benefits of safety, precise control over reaction parameters, and scalability. We focused on the integration of continuous-flow reactors to achieve controlled and rapid neutralization, thus addressing challenges such as the need for rapid reaction kinetics and the establishment of robust pathways for neutralization. Because the flow-chemistry approach can contribute significantly to the development of neutralization technologies for CWAs, we performed a thorough characterization in terms of reaction kinetics and neutralized product identification. The results demonstrated that the proposed continuous-flow-type neutralization reaction was faster and more efficient than batch-type neutralization reactions. Furthermore, in the early stages of the neutralization reaction, flow-type neutralization not only required less neutralizing agent than batch-type neutralization but was also faster. Thus, the chemical neutralization process proposed in this study can be used as a pragmatic foundation for developing demilitarization methods for CWAs.

INTRODUCTION

Chemical warfare agents (CWAs) are among the most lethal weapons developed by humans. They are designed to attack the nervous system, respiratory system, and skin of humans. Even minimal exposure can cause severe injury or death, making CWAs a significant threat to public safety and national security. CWAs can be broadly classified into nerve and blister agents. Nerve agents, which include a series of G agents (for example, sarin (GA) and soman (GD)) and the V series (for example, VX), are odorless and colorless liquids that disrupt the normal functioning of the nervous system, leading to respiratory failure and death. Blister agents, such as sulfur mustard (HD) and lewisite, cause severe chemical burns and blistering of the skin, eyes, and respiratory system.^{1,2}

The danger posed by CWAs has led to extensive research on and development of methods to neutralize these agents.^{3,4} Metal–organic frameworks (MOFs) are efficient materials that can capture and degrade CWAs, such as GA, VX, or HD.^{5–8} Although MOFs are likely to show high neutralization efficiency in a short time on a laboratory scale, they have the disadvantage of being expensive in terms of synthesizing the

catalyst and using it for neutralization on an actual field scale. Chemical neutralization—a highly effective method for rendering CWAs nontoxic—involves the use of reactive chemicals to convert toxic agents into nontoxic compounds.^{9–11}

The choice of a neutralizing agent depends on the target CWA type. A series of hydroxide solutions, such as sodium hydroxide (NaOH) and potassium hydroxide (KOH), are effective chemical neutralizers for CWAs because of their high reactivity toward the functional groups of CWAs.¹² However, if the neutralization is achieved by only using water, which can be easily found even on the frontline of a battlefield, neutralization without such an inorganic neutralization

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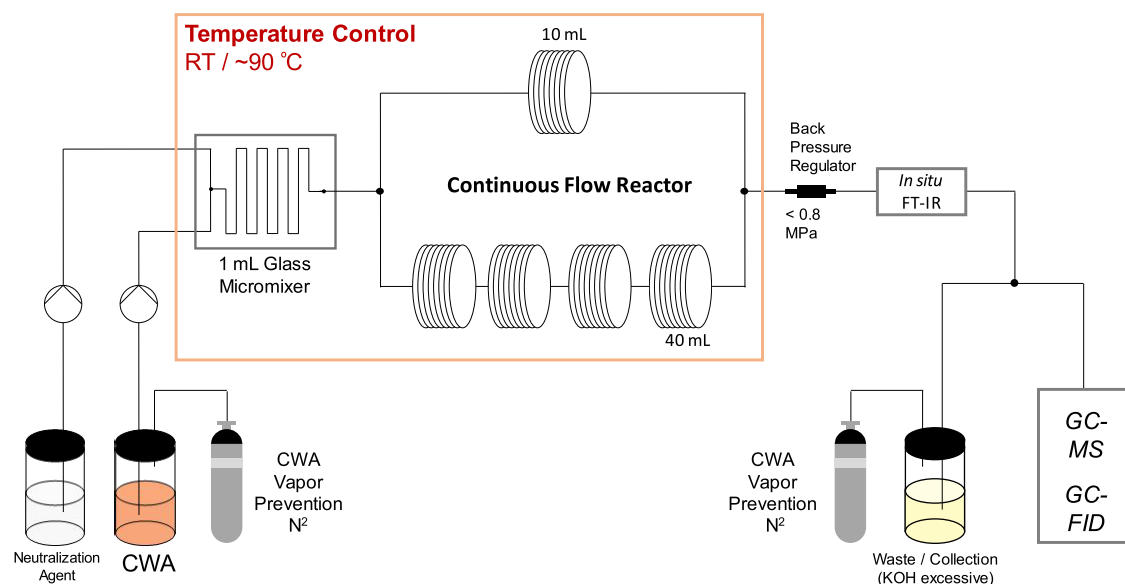


Figure 1. Schematic of the flow reactor system.

solution is worthy of investigation. Regarding blister agents, because of the immiscibility of HD toward water-based neutralization solutions, a specialized neutralizing agent such as monoethanolamine (MEA) is required.¹³ An MEA solution can be used as a selective neutralizer for blister agents such as HD because MEA exhibits the combined properties of amines and alcohols.¹⁴ MEA is notably nontoxic, noncorrosive relatively nonflammable, and inexpensive. Additionally, its high boiling point (171 °C) makes it suitable for exothermic reactions. The reaction between MEA and HD is relatively fast and is accompanied by moderate heat release. Furthermore, the miscibility of MEA and HD ensures the establishment of homogeneous reaction conditions, which is a crucial for achieving optimal neutralization.^{15,16}

Chemical neutralization is a complex process that requires precise control of the mixing process, temperature, pressure, and reaction time to achieve a high degree of neutralization. Batch-type neutralization reactions are widely used for chemical neutralization. However, batch-type reactions can be problematic because of issues with pressure and temperature control—particularly during scale-up.¹⁷ When batch-type neutralization involves a target CWA instead of a simulant, the batch-type reactor poses safety and health risks because the highly pressurized reactor is vulnerable to explosion.¹⁸ Moreover, achieving a neutralization rate of >99% is necessary to prevent the reformation of the CWA.¹⁹ Therefore, chemical neutralization requires a sophisticated process that enables control of the mixing ratio, temperature, pressure, and byproducts.

Compared with batch-type reactions, flow-type neutralization reactions offer several advantages, including precise control of temperature and pressure, scalability, and the ability to handle a wide range of reaction conditions.^{20–22} Flow-type neutralization is achieved by continuously pumping the CWA and neutralizing agent into a reactor. The use of flow reactors enables the precise control of the reaction conditions, as the flow rate, residence time, and temperature can be accurately controlled using a programmable computer. The flow-type reactor also offers improved mixing and heat transfer compared with batch-type reactors, leading to safer and more efficient

neutralization of CWAs.^{23,24} While batch-type reactions are limited by physical mixing, flow-type reactions have an advantage under the limitation of immiscibility between the target CWA and neutralizer. Furthermore, high-pressure and high-temperature environments confer better physical properties to the solution, such as lower viscosity and better heat transfer.¹⁷ Although several research groups have demonstrated the feasibility of flow-type continuous neutralization toward CWA simulants,^{25–27} the neutralization of actual CWAs has not yet been thoroughly explored in continuous-flow reaction systems.

In this study, we evaluated the efficiency of a flow-type continuous neutralization method using various analytical approaches, including gas chromatography with flame ionization detection (GC-FID) and *in situ* Fourier transform infrared spectroscopy (FT-IR). Based on the analytical results, we compared the efficiency of flow-type neutralization with that of batch-type neutralization. For accurate comparison, the residence time in the flow neutralization was normalized with respect to the reaction time of batch-type neutralization. In conjunction with the neutralization results of two representative simulants, we characterized the neutralization of GD under several conditions within a continuous-flow reactor.

EXPERIMENTAL SECTION

Chemicals and Reagents. Diethyl chlorophosphate (DECP), bis(2-chloro ethyl)ether (BCEE), MEA, dodecane, and *N,O*-bis(trimethylsilyl)trifluoroacetamide were purchased from Aldrich. The nerve agent GD (pinacolyl methylphosphonofluoridate) was synthesized by the Agency for Defense Development. The purity of the synthesized GD was verified to be >97% using ¹H-NMR spectroscopy and gas chromatography–mass spectrometry (GC-MS). **Caution:** GD is an extremely toxic chemical and should be handled with the appropriate precautions. Ethyl acetate (EA) and KOH were purchased from Samchun Chemicals (Seoul, South Korea). All chemicals were used as received, without further purification.

Instrumentation. The experimental setup for flow-type continuous neutralization of a customized flow reaction system (Figure 1). A commercially available flow

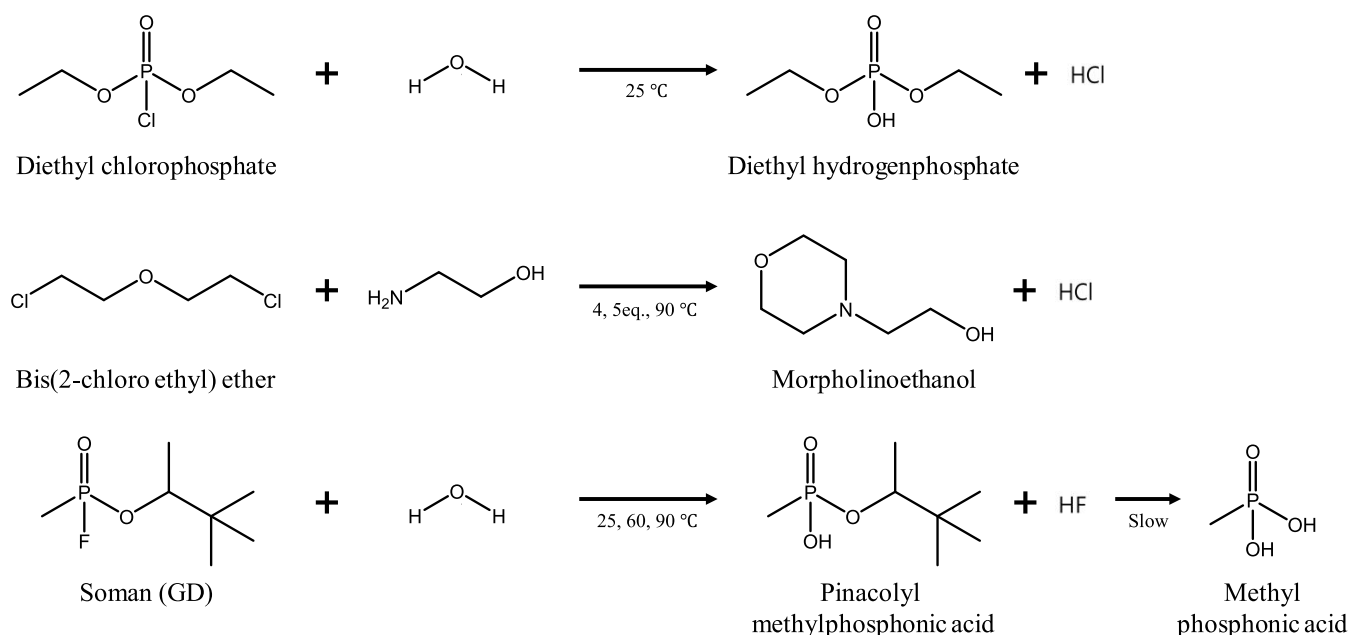


Figure 2. Neutralization mechanisms of DECP, BCEE, and GD.

reactor (R4+/R2, Vapourtec, U.K.) consisting of R2 flow delivery units with two high-performance liquid chromatograph pumps and four R4 flow reactor units integrated with 1 mL residence mixing units was used to neutralize the CWA simulants.

A flow was delivered to merge and mix the CWA and neutralizer within a T-connector connected to a 1 mL residence micromixer chip. The micromixer, which constituted the flow-type reaction system, provided a high surface area for the neutralization reaction of the neutralization target substances, such as DECP, BCEE, and GD, used in this study. The increase in surface area is effective in removing the heat generated during the exothermic reaction; which can improve the reaction rate. Additionally, a flow system with a constant speed can provide efficient mixing of the reaction solution.²³ The resulting mixture was continuously flowed into four consecutive flow reactor units each consisting of a 1/16" perfluoroalkoxy (PFA) tube unit (outer diameter of 1.58 mm and internal diameter of 750 μm). A single 10 mL flow reactor was used for neutralization times of 10 min, whereas four consecutive flow reactors (with a total of 40 mL) were used for neutralization reaction times of >20 min. The pressure throughout the flow reactors was controlled by an adjustable backpressure regulator, which was set to <0.8 MPa for safety. Each flow reactor was equipped with an air-heated temperature control system that could reach 140 $^\circ\text{C}$. The temperature and pressure throughout the flow reactors were monitored in real time.

Because some experiments involved actual CWAs, which can be lethal even for highly trained experts, the vapor prevention system was applied to bottles containing a CWA for safety reasons. To prevent the leakage of vaporized CWA, N₂ inert gas was continuously introduced into the sealed CWA bottle at a certain pressure (<0.1 MPa) that could not affect the internal pressure of the flow and pump systems. The prevention apparatus was also installed in the collection bottle at the end of the flow system.

In this research, it was particularly emphasized and noted that when the real CWA GD is neutralized by water, a small

amount of HF is generated as an intermediate (Figure 2); this can not only be highly toxic to humans but also affect the experimental equipment, such as glass-based apparatuses. Therefore, the entire flow reactor was preliminarily replaced with a Teflon-based material to avoid potential corrosion from the HF intermediate.

Because of the mere possibility of chemical substances remaining even after neutralization, to pre-establish a safe experimental environment, the waste bottle at the end of the flow reactor was preliminarily filled with premade KOH solution at a sufficient concentration (>4.0 M) to completely neutralize any remaining chemical substances.

Gas Chromatography Analysis. A gas chromatograph (Agilent, GC 8850) equipped with a flame ionization detector instrument was used. GC-FID was used for the quantitative analysis of the neutralization efficiency of the simulants and GD. Calibration solutions were prepared using EA. For the inlet, the temperature was set to 250 $^\circ\text{C}$, the pressure to 133.84 kPa, and the carrier-gas flow rate to 3 mL/min. The FID heater temperature was set to 300 $^\circ\text{C}$, air was injected at 400 mL/min, and H₂ was injected at 30 mL/min. The oven temperature was set to 40 $^\circ\text{C}$ for 1 min and then increased to 280 $^\circ\text{C}$ at a rate of 10 $^\circ\text{C}/\text{min}$.

Internal Standard Calibration. The GC-FID was calibrated using an internal standard for each sample. Twenty microliters of DECP, BCEE, and GD were added to 20 mL vials and weighed. EA (20 mL) was added to each vial to prepare reference solutions. An internal standard solution was prepared by adding 3.2 mL of dodecane to a 100 mL volumetric flask and filling the flask with EA. Calibration solutions were prepared by mixing different concentrations of the reference solution with a constant concentration of the internal standard solution and analyzed using GC-FID to obtain calibration curves for each chemical. All the calibration curves exhibited excellent linearity, with correlation coefficients (R^2) of >0.9999 (Figure S1).

Batch-type Neutralization. One hundred microliters of the simulants (DECP and BCEE) and GD were placed in a 10 mL vial and weighed accurately. The neutralizing agent (100 or

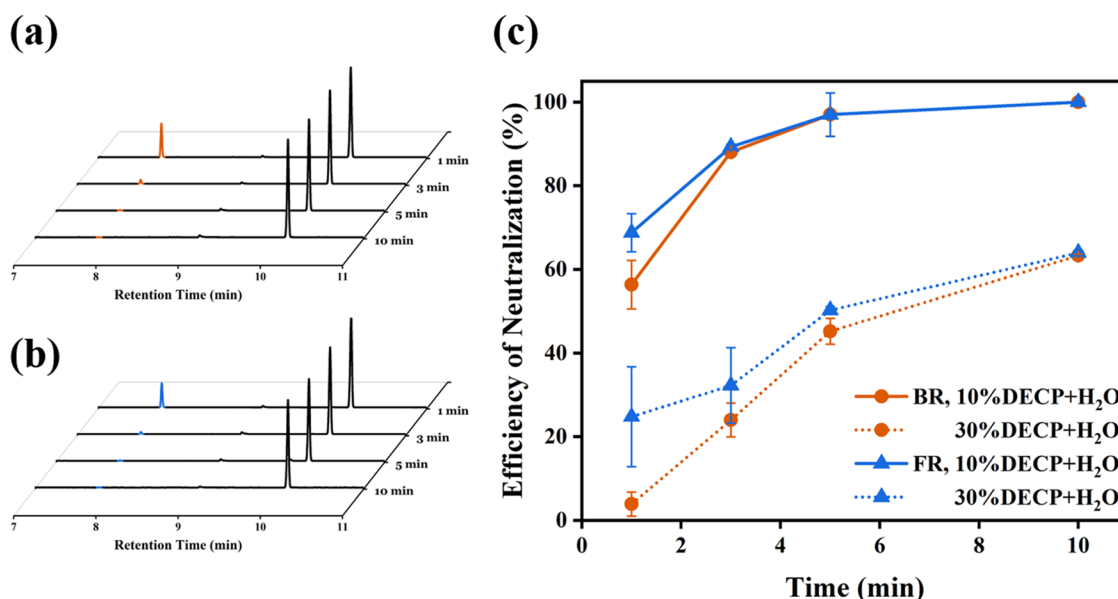


Figure 3. Representative GC-FID monitoring of DECP (7.78 min) of 10% DECP + H₂O in (a) batch- and (b) flow-type reactors. (c) Efficiency of the neutralization of DECP in batch- and flow-type reactors.

300 μ L of DECP and 100 μ L of BCEE and GD) was added to the vial along with a 200 mm magnetic stirrer. After a specified reaction time, 6 mL of EA and 1 mL of the internal standard solution were added to the vial, which was shaken. A 100 μ L aliquot of the mixed solution, 900 μ L of EA, and approximately 50 mg of MgSO₄ were transferred to a 1.5 mL Eppendorf tube and centrifuged at 13,500 rpm for 3 min. A 40 μ L aliquot of the supernatant was transferred to an analytical vial and diluted with 960 μ L of EA. The amounts of the simulant and GD remaining after the neutralization reaction were measured using GC-FID. The reactions were conducted in triplicate under all conditions.

Flow-type Neutralization. Using a Vapourtec R-series flow reaction system, target neutralization substances (GD or CWA simulants DECP and BCEE) and neutralizing agents (for example, MEA for BCEE and distilled water for DECP and GD) were directed into a wide-bore T-connector (inner diameter of 1.5 mm), which was directly connected to a 1 mL glass micromixer. The flow rates of the target neutralization substances and neutralizing agents are presented in Table S2. After mixing, the heterogeneous mixture quickly became homogeneous and then flowed into a 10 mL flow reactor. After passing through the chosen number of consecutive flow reactors, depending on the experimental conditions, the reactant mixture was sequentially conveyed to an *in situ* FT-IR instrument and collection bottle. *In situ* FT-IR analysis (Bruker, alpha2) was performed to track changes in the peak positions during the neutralization process. The neutralized sample was analyzed using FT-IR spectroscopy at the end of the flow-type equipment with a platinum ATR cell, and it then flowed into the collection bottle.

A 100 μ L aliquot of the mixed solution was obtained from the collection bottle, transferred to a 1.5 mL Eppendorf tube containing 900 μ L of EA and approximately 50 mg of MgSO₄ and centrifuged at 13,500 rpm for 3 min. A 100 μ L aliquot of the supernatant was transferred to an analytical vial and diluted with 900 μ L of EA. The amounts of the simulant and GD remaining after the neutralization reaction were measured

using GC-FID. The reactions were conducted in triplicate under all conditions.

RESULTS AND DISCUSSION

Neutralization of DECP with H₂O. DECP is commonly utilized as a simulant for nerve agents.²⁸ In this experiment, the neutralization efficiency of nerve agents in two reactor systems—batch-type and flow-type—was studied using DECP as a simulant to analyze the characteristics of the neutralization reaction. Water was used as the neutralizing agent because it is economical and readily available, even in a war field.²⁹ DECP is neutralized through a nucleophilic substitution reaction induced by water, where the P–Cl bond of DECP is hydrolyzed by water to form a P–OH bond (Figure 2).³⁰ This was confirmed by the GC-MS results for the neutralized products (Figure S2). The neutralization efficiencies were compared according to the volume percent (% v/v) of the simulant, which was set at 10 and 30% v/v. During the neutralization of DECP, the neutralization efficiency of the reaction was monitored using GC-FID. A reduction in the DECP peak at 7.78 min was observed, as shown in Figure 3. Subsequently, the neutralization efficiency was quantitatively assessed using the DECP calibration curve presented in Figure S1a. The results for the batch and flow systems from 1 to 10 min at volume concentrations of 10 and 30% were compared.

Under the condition of 10% v/v DECP, the batch-type reaction exhibited a neutralization efficiency of 56.4% in 1 min and reached 100% within 10 min, whereas the flow-type reaction exhibited a neutralization efficiency of 68.8% in 1 min and reached 100% within 10 min. In the initial stages of the reaction, the flow neutralization occurred relatively quickly for the flow type.

For the neutralization of 30% v/v DECP, both reactor systems exhibited lower neutralization efficiency compared with 10% v/v. For the batch-type reaction, the neutralization efficiency was 3.9% in 1 min and 63.3% in 10 min, and for the flow type, it was 24.8% in 1 min and 64.0% in 10 min. In the 30% v/v DECP condition, the flow-type reaction tended to have a higher efficiency than the batch-type reaction.

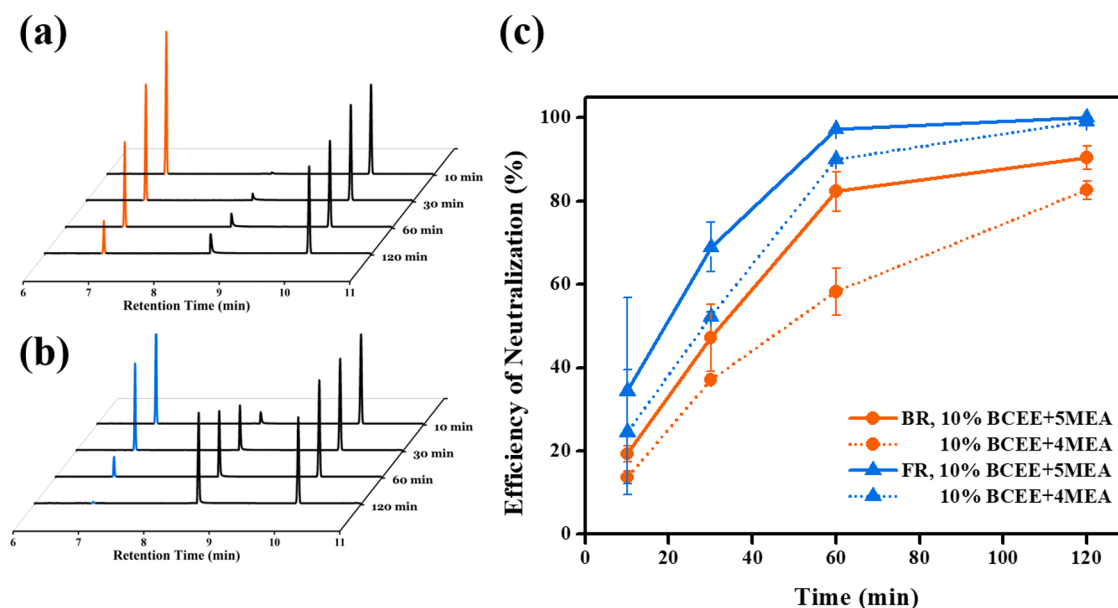


Figure 4. Representative GC-FID monitoring of BCEE (6.93 min) of 10% BCEE + 4 equiv MEA in (a) batch- and (b) flow-type reactors. (c) Efficiency of the neutralization of BCEE in batch- and flow-type reactors.

Moreover, for all the volume concentrations tested, the flow-type exhibited a higher neutralization efficiency—particularly in the initial stages of the reaction.

To compare the two reactors, the rate constants were calculated using a first-order reaction rate equation according to the linear relationship between $\ln[\text{DECP}]$ and the reaction time. During the initial period from 0 to 3 min, the concentration of quantitatively analyzed DECP present in the solution was substituted into eq 1 to derive the function describing the relationship between time and concentration. Subsequently, eqs 2 and 3 were utilized to compute the rate constant k and half-life (Table S3).

$$\ln [A]_t = -kt + \ln [A]_0 \quad (1)$$

$$\text{rate} = k[A]_0 \quad (2)$$

$$t_{1/2} = \frac{1}{k} \ln \frac{[A]_0}{[A]_0/2} = \frac{1}{k} \ln 2 = \frac{0.693}{k} \quad (3)$$

The rate constants of the batch-type reaction were calculated to be 6.96×10^{-1} and 0.95×10^{-1} at 10 and 30% v/v DECP ($t_{1/2} = 1.00$ and 7.33 min), respectively, whereas the rate constants of the flow-type were calculated to be 7.12×10^{-1} and 1.16×10^{-1} at 10 and 30% v/v DECP ($t_{1/2} = 0.97$ and 5.95 min), respectively. Neutralization occurred more rapidly in the flow-type reaction than in the batch-type reaction during the initial stages of DECP neutralization at both 10 and 30% volume concentrations of DECP. At the 30% volume concentration, the reaction rate in the flow-type reactor was confirmed to be 1.23 times that in the batch-type reactor.

Increasing the volume concentration of DECP in the neutralization reaction offers the advantage of directly increasing the neutralization throughput while reducing the quantity of waste generated. However, it also has the disadvantage of increasing the half-life. While the half-lives for both the batch- and flow-type reactors decrease with an increasing DECP concentration, the half-life of deceleration is significantly shorter for the flow type.

To facilitate comparison with batch reactions, the neutralization efficiency was evaluated under the same experimental conditions (Figure 1 and Table S2). Additionally, *in situ* FT-IR spectra of the flow-type reactor served as an indirect real-time indicator for tracking changes in peaks when distinct variations in bonding occurred during the reaction (Figures S5 and S6).

Under all conditions, the neutralization reaction was completed faster in the flow-type reactor than in the batch-type reactor. The higher reaction rates observed in the flow system suggest potential for more cost-effective applications in the future. Additionally, when necessary, the provision of IR data can contribute to the development of a safer reaction system.

Neutralization of BCEE with MEA. BCEE, which is also known as oxygen mustard, was selected as a simulant for HD. BCEE is more toxic than half-sulfur mustard, which is generally used as a simulant for HD. However, BCEE is highly stable and only chemically reacts in the presence of strong nucleophiles at high temperatures.¹⁹ MEA was selected as the neutralizing agent for BCEE because of several key advantages. It interacts swiftly with HD, resulting in controlled heat emission. Additionally, the solubility of MEA in HD contributes to a uniform reaction environment, which is essential for the thorough neutralization of the agent.^{15,16}

The conditions and mechanism for neutralizing BCEE are depicted in Figure 2. BCEE is neutralized through nucleophilic substitution reactions mediated by water or MEA. However, analysis of the neutralized products using GC-MS revealed primarily the presence of decomposition products formed by MEA (Figure S3). This indicates that neutralization by MEA dominates when an excess of MEA is added.

The neutralization efficiency and kinetics of BCEE were studied by monitoring the reaction progress with time for various molar ratios of the neutralizing agent. Figure 4 shows the neutralization efficiencies of BCEE from 10 min to 2 h at 4 or 5 equiv of MEA, which were compared between the batch- and flow-type reactions. Using GC-FID, we quantitatively assessed how well the neutralization reaction worked. We observed a reduction in the intensity of the BCEE peak at 6.93

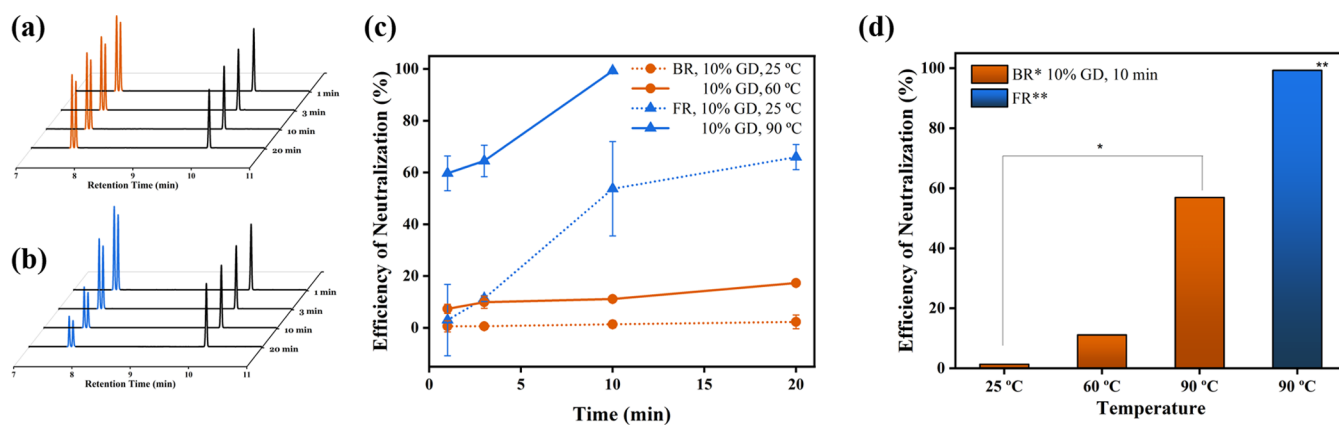


Figure 5. Representative GC-FID monitoring of GD (7.70 and 7.77 min) of 10% GD + H₂O in (a) batch- and (b) flow-type reactors. (c) Efficiency of the neutralization of GD in batch- and flow-type reactors. (d) Comparison of GD neutralization efficiency for different temperature conditions at 10 min in batch- and flow-type reactors.

min, as shown in Figure 4a. Next, we measured the effectiveness of the neutralization by using the BCEE calibration curve shown in Figure S1b.

The efficiency of the batch- and flow-type neutralization reactions was assessed over a period from 10 min to 2 h, beginning with conditions involving 4 equiv of MEA. Initially, in the batch-type reaction, the neutralization efficiency after 10 min was 13.7% with 4 equiv of MEA. Within 2 h, this efficiency increased to 82.6% but did not result in complete neutralization of BCEE. In the flow-type setup, the use of 4 equiv of MEA yielded a higher efficiency: starting from 24.5% at 10 min, it increased significantly over time, reaching 52.3% at 30 min and 90.0% at 1 h; after 2 h, the neutralization was almost complete, with an efficiency of 99.2%.

Following the analysis of reactions with 4 equiv of MEA, the experiments were extended to those with 5 equiv of MEA. In the batch-type reaction, a slightly higher initial neutralization efficiency of 19.3% was observed after 10 min, which increased to 90.4% after 2 h, indicating a slight improvement with the additional equivalent of MEA. Conversely, the flow-type reactions with 5 equiv of MEA exhibited a higher efficiency than those with 4 equiv, starting at 34.4% neutralization at 10 min and reaching full neutralization (100%) within 2 h.

The rate constants were calculated as described in the previous section. For the batch-type reaction, the rate constants were 1.45×10^{-2} and 2.05×10^{-2} with 4 and 5 equiv of MEA ($t_{1/2} = 47.66$ and 33.74 min), respectively. For the flow-type reaction, the rates constants were 4.09×10^{-2} and 5.99×10^{-2} with 4 and 5 equiv of MEA ($t_{1/2} = 16.97$ and 11.57 min), respectively. Thus, with 4 and 5 equiv of MEA, the flow-type reaction was 2.81 and 2.92 times, respectively, faster than the batch-type reaction.

Notably, the results indicated that the flow-type reaction with 4 equiv of MEA significantly outperformed the batch-type reaction with 5 equiv of MEA. This finding highlights the efficiency of flow-type systems, even when operating with small amounts of the neutralizing agent. The flow-type setup with only 4 equiv of MEA achieved nearly complete neutralization at 99.2% after 2 h, whereas the efficiency of the batch-type setup with 5 equiv of MEA was 90.4%. This performance disparity indicates the capability of the flow-type system to attain high levels of neutralization more efficiently, even with a lower MEA concentration. Such a result emphasizes the intrinsic advantages of the flow-type reaction mechanism,

suggesting that even with less neutralizing agent, the system can deliver superior outcomes compared with batch-type reactions that utilize higher concentrations. This not only shows the effectiveness of flow-type setups in chemical neutralization but also implies potential for significant resource optimization in neutralization processes.

However, the viscosity of the neutralization products increased with the MEA equivalents (Table S1). Therefore, the optimal equivalent conditions of MEA are deemed necessary when the neutralization is conducted in the flow-type reaction because of the sensitivity of the flow reactor to viscosity.

Neutralization of GD with H₂O. The efficiency of the neutralization of GD was compared between batch and flow-type reactors. Similarly to the previous experiment with DECP, water was used as the neutralizing agent. The experiments were conducted under both ambient and elevated temperature conditions in their respective reactors.

Moreover, GD underwent a neutralization mechanism where the P–F bond was cleaved by the nucleophilic attack of water, forming a P–OH bond (Figure 2). Furthermore, the neutralized product was derivatized and identified using GC-MS to confirm the results presented in Figure S4. The neutralization reaction of GD was monitored over time, and decreases in the GC-FID peak of GD were observed at 7.70 and 7.77 min. After the neutralization was complete, the neutralized product was confirmed by the mass spectrum at 11.909 min, as shown in Figure S4b.

In contrast to DECP, GD was neutralized with a mere 1% efficiency at room temperature after 10 and 20 min when water was employed as the neutralizing agent, indicating negligible progress in the neutralization of GD in batch-type reactions (Figure 5c). This underscores the need for strategies to enhance the neutralization reaction, which is hampered by the low solubility of GD in water. Although increasing the reaction temperature to 60 °C led to slight improvements in the neutralization efficiency in batch-type reactions—11.1% after 10 min and 17.3% after 20 min—these levels of efficiency were still insufficient, necessitating higher temperature conditions. Conversely, the flow-type reactor consistently demonstrated higher neutralization efficiencies at room temperature without heating, with 53.7% efficiency at 10 min and 66.0% at 20 min. The efficiency after 20 min was more than 3-fold higher, and

this improvement was also reflected in the comparison of rate constants.

The rate constants for the batch-type reaction at room temperature and at 60 °C were calculated to be 0.10×10^{-2} and 0.90×10^{-2} , with half-lives of 679.05 and 77.33 min, respectively. For the flow-type GD neutralization reaction, the rate constant was determined to be 5.85×10^{-2} at room temperature, with a half-life of 11.85 min. Consequently, the flow-type reaction was 57 times faster than the batch-type reaction at room temperature. This confirms the efficiency of the flow-type reactor in achieving higher GD neutralization efficiency without the heating conditions required for batch-type reactions.

However, unlike CWA simulants, actual CWAs require neutralization efficiencies greater than 99%. In the flow-type reactor, conditions were identified that achieved more than 99% efficiency at 90 °C within a reaction time of 10 min, as shown in Figure 5c, whereas the batch reactor only achieved 57% efficiency under the same conditions in Figure 5d. Thus, although the neutralization efficiency of the batch-type reactor increases with temperature, it remains lower than that observed in the flow-type reactor under optimal conditions. High temperatures in batch-type reactions can lead to instantaneous temperature increases due to the exothermic nature of the neutralization reaction, thus posing a risk of generating side reactions. In contrast, the flow-type reactor mitigates this issue via mechanical control, preventing temperature spikes and ensuring stable high-temperature reactions with greater efficiency.

CONCLUSIONS

CWAs are extremely dangerous and should be completely neutralized through a rapid and safe process. This study demonstrated that the proposed continuous-flow-type neutralization reaction is faster and more efficient than the batch-type neutralization reaction. In particular, in the early stages of the neutralization reaction, flow-type neutralization is more efficient than batch-type neutralization, and a smaller amount of neutralization agent is required. With this advantage, CWA risk reduction, cost reduction, and a higher throughput per unit time can be achieved at scales larger than the laboratory scale.

The chemical neutralization results obtained in this study can be used as a pragmatic foundation for the investigation and development of neutralization methods for CWAs. Although flow-type neutralization is currently in the specialization stage at the laboratory scale, further research is needed in various aspects so that our findings can be leveraged to develop a technology for the rapid neutralization of CWAs.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.4c04000>.

Viscosity analysis, gas chromatography calibration, GC-MS analysis, FT-IR analysis, neutralization of DECP with KOH (PDF)

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†J.K. and N.Y.K. contributed equally to this work.

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Notes

The authors declare no competing financial interest.

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