

Journal of Integrated SCIENCE & TECHNOLOGY

A nucleophilic non aqueous decontaminant for degradation of chemical warfare agents

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Received on: 26-Nov-2020, Accepted and Published on: 21-Dec-2020

ABSTRACT



An improved non-aqueous decontaminant has been explored against chemical warfare agents sulfur mustard (HD) and soman (GB). This decontaminant comprises of 2-aminoethanol (30% w/v), dimethylamino ethanol (42% w/v), sodium hydroxide (2% w/v), benzotriazole (1% w/v), and dimethyl sulfoxide (25 %) and it chemically degraded more than 99 % of sulfur mustard and soman within a time of 45 min at -35°C. It was found to be effective over a broad range of temperatures from -35 to +55°C without losing its efficacy even at sub zero temperatures. This decontaminant exhibited decontamination ratio of $V_{[Detoxicant]}/V_{[HD, GD]}$ 50 or 5 while a recently reported one exhibited a ratio of 100 or 2 against HD or GD respectively. Although, this ratio decreased slightly in the case of GD, it is sufficient enough for complete decontamination. This improved decontaminant meets all the military requirements and promise its field application in near future.

Keywords: Chemical warfare agents, Non aqueous decontaminant, Nucleophile, Chemical detoxification, DS-2 solution

INTRODUCTION

Quick and complete removal of chemical warfare agents¹⁻⁵ (CWAs) from dirtied surfaces is essential immediately after chemical attack.⁶⁻⁹ Prevailing and workable approaches for decontamination¹⁰⁻¹⁵ use aqueous and non-aqueous chemical preparations.¹⁶⁻²¹ Different countries are exploring different decontamination formulatios for effective and efficient detoxification of CWAs, In this context, a non aqueous

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Cite as: J. Integr. Sci. Technol., 2020, 8(2), 51-56.

©ScienceIN ISSN: 2321-4635 http://pubs.iscience.in/jist

decontaminant comprised of aminoalcohol, diethylenetriamine and NaOH having operational effectiveness in the range of -30 to +49°C temperatures²²⁻²³ has been explored by Karcher Germany (GDS-2000)²⁴ and has substituted decontamination solution-2 (DS-2)¹⁶ as battle field decontaminant by many nations in the world. Subsequently, decontaminants comprised of aminoethanol, alcohol, dimethyl sulfoxide, and potassium hydroxide were introduced as the next generation variants, which are operative, eco-friendly and do not comprise chemicals like diethylenetriamine that causes human health risks.²⁵⁻²⁶ However, it freeze below -20°C hindering its use at subzero temperatures, hence there is necessity for development of a non-aqueous decontaminant comprised of eco-friendly components and sub zero operability up to -35°C, and meet all the primary military decontamination requirements. Inspired by these, an effective decontamination methodology based on a nucleophilic non aqueous decontaminant composed of 2aminoethanol, potassium hydroxide, and *N*-methyl-2pyrrolidone has been developed recently by our group.²⁷

It exhibited a decontamination ratio $V_{[Formulation]}/V_{[HD or GD]}$ of 100 or 2 against sulphur mustard (HD) or soman (GD) and completely degraded them within 15 min at room temperature with a corrosion rate of 0.0032 mm/y. It met all other decontamination requirements, however, it needs to be further improved in its decontamination ratio, and corrosiveness towards metal surfaces, hence, efforts continued in that direction to improve this formulation further.

Herein, we reported an improved battle field operable and effective nucleophilic decontaminant against CWAs HD and GD based on non-aqueous, eco-friendly components. Effect of volume ratio of CWAs to detoxicant, reaction temperature, and decontamination time on decontamination efficacy against CWAs were optimized to suit combat scenario. Chemical degradation products were characterized by gas chromatograph fitted with mass selective detector (GC-MSD). The present decontamination formulation was also examined for degree of corrosion and its capability to decontaminate various surfaces alike tainted metal, painted metal, and impermeable NBC suit to assess its field operability.

2. EXPERIMENTAL SECTION

2.1. Materials: 2-Aminoethanol (AE), dimethylethanolamine (DMEA), dimethyl sulfoxide (DMSO), sodium hydroxide (NaOH), benzotriazole (BZ), isopropanol (IP), and calcium chloride lumps were procured from E. Merck India Ltd, Mumbai (India). HD and GD of > 99 % assay were synthesized in schedule 1 infrastructure of our establishment (designated by Organization for Prohibition of Chemical Weapons). Chemical degradation studies were also carried out in the same facility. Caution: These agents are highly toxic, hence to be handled by trained personnel wearing individual protective equipment only.

2.2. Preparation of Non Aqueous Formulations: Firstly, a fixed amount of 2-Aminoethanol AE was added to round bottom flask equipped with calcium chloride guard tube in moisture free environment. Later, NaOH flakes equivalent to 2 % wt of the total non aqueous formulation was mixed to AE and stirred for around 10 h at 60°C to obtain 2-Aminoethanol solution of NaOH. Afterwards, remaining amounts of DMEA, benzotriazole BZ, DMSO were added sequentially to above solution to obtain the final decontamination formulation and stored in stoppered flasks in moisture free environment.

2.3. Study of Degradation Reactions of HD and GD: Chemical degradation reactions were carried out at different temperatures viz. 27°C (room temperature), +55°C, and -35°C by treating 20 μ L of either HD or GD with respective volume ratio of decontaminant. These experimental temperatures were selected based on battle field requirements in Indian terrain. The decontaminant was varied from 100 -4000 μ l for HD and 20-300 μ l in the case of GD. After periodic intervals of time, 10 mL of IP was added to the reaction mixture and shaken for 2 minutes to extract the remaining CWAs into the solvent to study kinetics of decontamination. Concentrations of the agent residues in the above solutions were determined by GC-FID fitted with BP-5 GC capillary column. This GC column had a length of 30 m, internal diameter of 0.5 mm, and a film coating of 0.5 μ thickness. Decontamination reaction products were symbolized using GC-MS system of Agilent, USA make. EI source was used for ionization of compounds eluting from HP 5MS column (30 m length, 0.25 mm id, and 0.25 μ m film thickness). Deep freezer of M/s. AR Enterprises, New Delhi make was used for studying decontamination reactions at -35°C and an oven with constant temperature chamber of M/s Narang scientific company, New Delhi was utilized for studying decontamination reactions at 55°C.

2.4. Decontamination studies of HD and GD on various tainted surfaces: Impermeable NBC suit, metal panel, and painted metal panel specimens of 2 cm X 2 cm dimensions were used for studying surface decontamination efficiency against the CWAs. Initially, 20 μ L of CWAs was uniformly distributed over the specimen and kept at room temperature for 1 h in vertical or horizontal position. After 1 hr, 1:150 V/V ratio of decontaminant was spread over the tainted specimen and allowed for 30 min. Subsequently, the decontaminated specimens were washed with 10 mL IP and the extracted solutions were analyzed by GC ²⁸.

2.5. Corrosion studies on the decontaminant: 5 cm X 5 cm metal panels of SS 316 grade were weighed and immersed in prepared non aqueous decontaminant or DS-2 solution in 500 mL beakers for 72 h at room temperature $(27 \pm 2^{\circ} \text{ C})$ for corrosiveness of the prepared decontaminant.²⁹ After 72 h of treatment, treated panels were cleaned thoroughly and dried at room temperature. Samples were dried, weights were taken, change in weight was recorded, and compared to fresh samples. Corrosion rate of SS specimen was calculated using an equation as reported somewhere else ²⁹ and the results was compared against DS-2 solution.

3. RESULT AND DISCUSSION

3.1. Screening of Various Ingredients for Making Non Aqueous Decontaminant: To evolve a battle field decontaminant for degradation of CWAs HD and GD, a variety of formulations based on different compositions of AE, DMEA, NaOH, DMSO, and BZ have been explored and screened for their reactivity against CWAs.30 Every constituent of the decontamination formulation performs a vital role in the complete and efficient degradation of CWAs. AE has dual functional groups amine and alcohol groups and also works as a good solvent for alkalis. Due to it's self associating properties, it forms cages close to Na⁺ ions leading to sequestration of Na⁺. Additionally, AE is commercially accessible, cost-effective, less noxious, bio degradable, and have better solvency for CWAs. In this decontaminant, AE acts as nucleophile source as well as sequestering agent for Na⁺ cation from the alkali. The nucleophilicity of aminoalkoxide ion is the utmost vital factor for degradation of CWAs. In order to produce the nucleophile, strong base NaOH was selected as it easily produces alkoxide ion due to its reaction with AE. Furthermore, Na⁺ has sturdier clatherating effect with amine group of AE, consequently; it freed alkoxide ion (NH2RO) for nucleophilic attack on the CWAs.

In spite of these mentioned promising properties, decontaminant comprising exclusively of AE and NaOH, could not completely degrade HD. Nevertheless, its decontamination efficiency is plausible to be significantly augmented if suitable solvent system is added which enables the generation of more amount of Nu⁻ and it should also maintain the fluidity at subzero temperature like -35°C. Solvent system utilizing the combination of both DMSO, DMEA in an optimized combination meets both the above requirements. As an added advantage it could efficiently dissolve the CWAs, components of formulation AE, NaOH, and have strong affinity towards Na⁺ ion²²⁻²⁹. It might be also possible that DMEA, DMSO combination efficiently solvate Cl⁻ or F⁻ with small ionic radius that were produced during the reaction of non aqueous formulation with HD or GD. Consequently, equilibrium could have been shifted to right side of the reaction and led to completion of the detoxification reaction. Formulation composed of AE, NaOH along with DMEA had shown excellent fluidity at -40°C and had not frozen at that temperature, however, it could not completely degrade HD within 20 min at room temperature. Whereas, AE, NaOH along with DMSO had completely degraded HD within 20 min at ambient conditions, however, it had frozen at -40°C impeding its operability at that temperature. Nevertheless, AE, NaOH along with the combination of DMSO and DMEA as discussed above had shown operability at -40°C as well as significant chemical reactivity against HD. Our previous synthesized formulation based on AE, NMP and KOH had fluidity at -35°C and could completely degrade HD in 15 min at ambient temperature.²⁷

The amount of NaOH plays a very important part for the production of adequate quantity of nucleophile. NaOH was varied from 1.0 to 2. 5 % with 0.5 % increments against HD inferred that, 2.0 and 2.5 % NaOH showed same decontamination efficacy, however, aimed at preparation of non-aqueous decontaminant with field application, 2.0 % NaOH was selected as optimal weight in order to lessen the corrosiveness and enhance the low temperature operability. BZ was added to avoid corrosive action of the decontaminant on metal surfaces. It is well known that, BZ forms a passive layer consisting of complex between metal surface and BZ thus preventing corrosion.²³

Table 1 Comparison of non-aqueous formulation for chemicaldegradation of CWAs.

Formulation	Decon. Efficiency HD(%)	Ratio of decontaminant/agent
AE (60% w/v), NMP (38% w/v) and KOH (2% w/v) ²⁷	99.9%	100 (v/v)
AE (30% w/v), DMEA (42% w/v), NaOH (2% w/v), BZ (1% w/v), DMSO (25% w/v)	99.9%	50 (v/v)

Subsequently after adjusting all the active components and taking care of all operational field requirements in mind, AE, NaOH, DMEA, DMSO, BZ were shortlisted for making the decontaminant. With the aim of arriving at the finest composition for degradation of CWAs, AE was varied from 20-40 percent, NaOH was varied in between 1-2.5 %, DMEA was varied in between 20 to 42 %, DMSO was varied between 20-40%, and BZ was varied in between 0.5 to 1 %. It can be inferred from Table 1, the best results against HD were found to be obtained for nonaqueous decontaminant consisted of 30 % of AE, 42 % DMEA, 2 % NaOH, 1 % BZ, and 25 % DMSO, hence, studied further.

3.2. Effect of volume ratio of decontaminant and CWAs on efficiency of decontamination: Amount of the decontaminant plays a very important role for the complete degradation of HD or GD and it was optimized by varying volume ratios of CWA and decontaminant. As depicted in Figure.1, decontamination efficiency increases with increase in ratio of $V_{[detoxicant]}/V_{[Agent]}$. Total degradation could be accomplished within 20 min by using $V_{[detoxicant]}/V_{[Agent]}$ ratio of minimum 50 for HD and 5 for GD, respectively.



Figure 1. Effect of Volume volume ratio of decontaminant on efficacy of degradation of HD and GD.

3.3 Effect of temperature on the chemical degradation of HD and GD: Chemical degradation of HD or GD with non-aqueous decontaminant was carried out at 27 °C, 55°C, and - 35° C temperatures. Performance data of the decontaminant towards HD at these temperatures are shown in Figure 2. Non aqueous decontaminant in the ratio of 1:50 V/V of HD and decontaminant, totally destroyed HD in 20 min at room temperature (27°C) and in 6 min at 55°C while the efficiency was decreased to 96 % at -35°C even in 45 min. Nevertheless, when volume ratio increased to 1:200 V/V, the decontaminant fully degraded HD within 45 min at -35°C.

Figure 3 depicts the graph drawn between 1/(conc. change) and time, shows linear plots indicating the second order kinetics of HD degradation by non aqueous decontaminant.³¹

Rate constant and half life values were also computed and the data is incorporated in Table 2. Rate of chemical degradation reaction raised from 4 x 10^{-4} to 0.009 mg⁻¹ml.sec⁻¹ when temperature was raised from -35 to 55°C. Activation energy of this degradation reaction was computed to be 10.57 kCal/mol as per the Arrhenius equation.²⁹



Figure 2. Effect of temperature on degradation of HD using non aqueous decontaminant.



Figure 3. Kinetics of degradation of HD using non aqueous decontaminant at various temperatures.

Non aqueous decontaminant entirely degraded GD in 7 min at room temperature (27°C) and in 5 min at 55°C when used at a volume ratio of 1:5 V/V % of GD and decontaminant (Figure 4). However, it degraded only 62 % of GD in 15 minutes at -35°C. Nevertheless, by increasing the decontaminant ratio to 1:15 V/V, GD could be totally degraded chemically in 10 min at -35°C.

 Table 2. Kinetic data of degradation reaction of HD with nonaqueous decontaminant.

Temp. (°C)	Volume ratio of HD: Decontaminant	Rate constant, k (mg ⁻¹ ml sec ⁻¹)	Half life, t _{1/2} (sec)
27	1:50	0.0020	19.68
55	1:50	0.0090	4.37
-35	1:50	4x10 ⁻⁴	98.42
-35	1:200	0.0024	16.4

Chemical degradation reactions of GD with non aqueous decontaminant followed second order kinetics. Rate constant values and half life values were computed and incorporated in Table 3.



Figure 4. Effect of temperature on degradation of soman.

 Table 3. Kinetic data of degradation reaction of GD with nonaqueous decontaminant.

Temp. (°C)	Volume ratio of GD: Decontaminant	Rate constant, k (mg ⁻¹ ml sec ⁻¹)	Half life, $t_{1/2}$ (sec)
27	1:5	0.0020	24.5
55	1:5	0.0030	16.34
-35	1:5	1x10 ⁻⁴	490
-35	1:15	0.0013	37.7



Figure 5. Kinetics of degradation of soman using non aqueous decontaminant at various temperatures.

Rate of detoxification reaction rose from 1×10^{-4} to 0.002 mg⁻¹ ml sec⁻¹ with the raise in temperature from -35 to 55°C (Figure 5).

Energy of activation for this decontamination reaction of GD was calculated to be 2.85 kCal/mol.³¹

3.4 Surface decontamination efficiency of non aqueous composition towards surfaces tainted with HD or GD in vertical and horizontal conditions

Metal, painted metal, and impermeable NBC suit specimens were treated with HD and decontaminated using the non aqueous formulation. This decontaminant exhibited 99, 99, 97 % of surface decontamination efficiency values in 30 min respectively on metal, painted metal, impermeable suit surfaces when spray applied with 1:150 V/V % of agent to decontaminant in vertical conditions. Whereas, it demonstrated >99.9 % of surface decontamination efficiency in horizontal condition for all surfaces. Under similar conditions, GD contaminated specimens were completely decontaminated within 30 min both in horizontal as well as vertical positions. Differences in decontamination efficiency values obtained in horizontal and vertical conditions could be due to the difference in the amount of agent and decontaminant available for reaction and different contact time. It could be understood that both the agent and decontaminant drifts down from the object in vertical conditions unlike in horizontal conditions leading to different contact times between the agent and decontaminant.

In the case of painted surfaces, slight discoloration was observed in the case of present formulation, whereas, DS-2 completely removed paint from surfaces in 30-45 min of treatment time & decontamination time. However, both the decontaminants exhibited similar decontamination efficiency values against studied chemical warfare agents on painted surfaces.

3.5 GC-MS data of degradation reactions of HD and GD

GC-MS data illustrated the chemical degradation of HD into divinyl sulfide and 2-chloro ethyl vinyl sulfide by non aqueous decontaminant. It proposes that the detoxification of HD encompasses E2 double elimination mechanism.³¹ Whereas, GD chemically degraded to *O*-pinacolyl *O*-(2amino)ethylmethylphosphate as per GC-MS data. It infers that the degradation of GD includes cleavage of P-F bond and replacement with Nu⁻ (NH₂CH₂CH₂O⁻) as reported elsewhere.^{27,33-} ^{34.} GC-MS data and the reaction pathways of detoxification of HD and GD are not being shown here as similar results were already published.³⁴

3.6 Degree of corrosion of non-aqueous decontaminant

Degree of corrosion induced by the developed decontaminant in SS 316 specimens was examined and the data was compared with that of DS-2 to investigate its suitability for use in battle field. Results designated that degree of corrosion in the SS 316 materials was negligible when compared to DS-2 (Table 4) and show that prepared non aqueous decontaminant is significantly better than it. This decontaminant also was found to be equal in corrosive nature than a recently reported one comprised of amino ethanol, KOH, and NMP which showed a corrosion rate of 0.0032 $\rm mm/y.^{27}$

Not only in the case of degree of corrosion, the fluidity of the present formulation is retained even at -35°C promising its extensive application at sub-zero temperatures down to -35°C for decontamination of chemical warfare agents whereas DS-2 solution freezes thus impeding its application at this temperature and below it.

 Table 4. Corrosion rate and degree of corrosion of non-aqueous decontaminant and DS-2 towards SS 316 panels.

Decontaminant	Corrosion rate (mm/y)	Corrosion degree
Prepared non-aqueous decontaminant	Negligible	Non corrosive
DS-2	0.0300	Slightly corrosive

Additionally, present formulation consists of bio-degradable³⁵ components like, amino ethanol, dimethyl amino ethanol, dimethyl sulfoxide, etc, making it viable for easy disposal after neutralization unlike DS-2 in which harmful chemicals like 2 methoxy ethanol and diethylene triamine are present which are relatively less bio-degradable making it difficult for easy disposal.¹⁷

CONCLUSIONS

A decontaminant with enhanced efficiency has been developed for complete degradation removal of HD and GD from tainted surfaces and this decontaminant completely degraded HD or GD within 20 minutes at room temperature. Decontamination kinetics were analyzed and found that it follow second order behaviour. This formulation demonstrated excellent surface decontamination efficiency values in the range of 97-99 % over vide variety of surfaces such as metal, painted metal, impermeable NBC suit samples dirtied with HD or GD in vertical as well as horizontal conditions. The present formulation exhibited negligible degree of corrosion towards SS metal surfaces and was found to be significantly better than DS-2. It is anticipated that present formulation finds application as field decontaminant in near future.

ACKNOWLEDGEMENT

The authors are grateful to Dr D K Dubey, Director, DRDE for his constant encouragement and providing the necessary facility for this work. This manuscript is assigned DRDE accession number DRDE/PD/30/2019. Authors are thankful to Dr. Manisha Sathe for her invaluable suggestions.

Conflict of Interest: Authors declare no conflict of interest.

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