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Clean-up of chemical warfare agents on soils using simple washing or chemical treatment processes

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Abstract

Several simple processes have been studied for the destruction of chemical agents, Soman and Mustard, on soils. A double wash or an extended single wash with water was effective in removing Mustard and Soman from soil; addition of either anionic or cationic surfactant did not improve removal efficiency. Soils with higher organic carbon content were more difficult to decontaminate. The most effective chemical process for the removal of Mustard was treatment with hypochlorite; treatment with Na₂CO₃ or NaOH were almost as effective as hypochlorite in cleaning Mustard contaminated soil. Soman was removed most effectively by treatment with Na₂CO₃. Overall the most efficient process for the destruction of both Mustard and Soman was treatment with Na₂CO₃ solution.

1. Introduction

Chemical weapons have been reported to have been used recently in several conflicts despite being prohibited by the 1925 Geneva Protocol. The most recently verified use has been during the Iran–Iraq war [1]. Iraq possessed large stockpiles of chemical weapons and bulk chemical agents including vesicants and the most toxic nerve agents. These are being destroyed under the supervision of the United Nations by a variety of procedures including low- and high-temperature incineration and chemical degradation. At weapons storage and disposal sites, spills and leaking munitions contaminate the environment in general, and the soil in particular, and will pose a clean-up problem.

Chemical weapons were not used during World War 2 although several nations possessed agents, mainly vesicants. The majority of these agents were destroyed after World War 2 but isolated cases of the discovery of old munitions occur. Often these munitions or storage containers are leaking and require decontamination. The soil in

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the immediate vicinity of leaking canisters may also require decontamination. Of necessity, such procedures must be simple and be capable of being undertaken in situ using simple techniques and processes. The alternative is removal of contaminated material to a disposal site with all the concomitant hazards. For these reasons, a study was initiated with the primary objective being the development of a decontamination process for soils contaminated with chemical agents.

Four different soils, ranging from loamy sand to sandy clay loam, were spiked at the 5 and 20 mg/g levels with Mustard [bis(2-chloroethyl) sulphide] or Soman (pinacolyl methylphosphonofluoridate). After allowing the spiked soils to equilibrate, they were subject to a variety of washing processes. Residual chemical agent was extracted from the wet soils by a modified procedure of D'Agostino and Provost [2] in which acetone replaced hexane as the primary extraction medium.

2. Experimental

2.1. Soil samples and sample handling

Samples of dry soil (5 g) were weighed into 100 ml glass conical flasks fitted with ground glass stoppers and were spiked with either 25 mg or 100 mg Soman or Mustard applied as 5 μ L droplets from a micro-syringe. Four replicates were prepared for each agent level. The agent and soil were mixed intimately by vigorous shaking and allowed to stand for 90 min at 20 °C prior to treatment. The treatment solution (25 ml) was added and the mixture shaken for 30 min. After centrifuging (10 min at 700g) supernatant solution was removed by decantation. The soil samples were extracted with 2×10 ml acetone with ultrasonic vibration (10 min) and then centrifuged (10 min at 700g). The acetone extracts were combined and made up to 25 ml for analysis. The soil was re-extracted using the above procedure with dichloromethane. Solvent extracts were filtered prior to analysis by gas chromatography. For samples which were washed twice, the initial wash solution was removed by decantation after centrifuging as above.

2.2. Instrumental

Gas chromatography was performed with a Varian 3400 gas chromatograph equipped with on column injection and a flame photometric detector. A $12 \text{ m} \times 0.53 \text{ mm}$ ID BP-10 SGE capillary column was used isothermally in capillary mode at $130 \,^{\circ}$ C for Soman and at $150 \,^{\circ}$ C for Mustard analyses. Agent concentration data was acquired in triplicate for each replicate sample at both spiking levels and percentage residuals calculated by external standard calibration.

3. Results and discussion

Four different soil types ranging from sand to sandy clay loam with varied organic content and pH were investigated during the study. Spiking levels of 5 and 20 mg/g

Treatment	Level (mg/g) Recovery after extraction (%) ^a		extraction (%) ^a	Total residual (%)	
		Acetone	CH ₂ Cl ₂		
Water wash	5 20	16.1 ± 0.5 24.6 ± 1.2	$\begin{array}{c} 0.74 \pm 0.02 \\ 1.12 \pm 0.03 \end{array}$	17 26	
10% ethanol	5 20	12.48 ± 0.74 31.15 ± 1.60	- 0.67 \pm 0.0 1.46 \pm 0.97	13 33	
0.1% CTAC	5 20	6.40 ± 0.30 11.79 ± 0.69	$\begin{array}{c} \textbf{0.49} \pm \textbf{0.03} \\ \textbf{0.66} \pm \textbf{0.02} \end{array}$	7 12	
0.1% DB S	5 20	7.91 ± 0.17 13.19 ± 0.90	$\begin{array}{c} 0.43 \pm 0.06 \\ 0.67 \pm 0.02 \end{array}$	8 14	
2 × water wash	5 20	1.96 ± 0.07 3.32 ± 0.24	$\begin{array}{c} 1.41 \pm 0.04 \\ \textbf{0.49} \pm 0.01 \end{array}$	3 4	
2 × 10% ethanol	5 20	1.82 ± 0.08 6.13 ± 0.37	$\begin{array}{c} 0.49 \pm 0.0 \\ 0.49 \pm 0.02 \end{array}$	2 7	
2×0.1% CTAC	5 20	2.10 ± 0.19 5.32 ± 0.16	Trace 0.51 ± 0.08	2 6	
2×0.1% DBS	5 20	3.84 ± 0.13 8.08 ± 0.30	Trace 0.71 ± 0.03	4 9	

Table 1 Recovery of Mustard from Soil F contaminated at 5 and 20 mg/g after various physical washing processes

^a Mean \pm SD (n = 4).

Mustard and Soman were selected as being well above typical battlefield contamination density levels that have been estimated [2] to be $100-1000 \mu g/g$ based on a contamination density of $1-10 g/m^2$. The spiking levels were typical of leaks which may occur from aging munitions or spillage during destruction of chemical weapons.

Preliminary experiments were carried out on Soil F, a loamy sand, to survey the effectiveness of several simple clean-up processes. Table 1 details the recovery and residual percentages of Mustard from Soil F for a range of washing solutions ranging from plain water to cationic surfactant, cetyltrimethylammoniumchloride (CTAC) and anionic surfactant, sodium dodecylbenzenesulphonate (DBS). Treatment with either detergent solution significantly increases the removal of Mustard from the soil. At the upper contamination level of 20 mg/g, 26% Mustard remains on the soil after a single water wash, whereas treatment with either detergent reduces this level to 12-14%. Inclusion of 10% ethanol in the water wash does not significantly improve the washing process. Washing twice reduces the residual Mustard levels further to 4% for water. A simple water wash for 60 min would appear to be adequate for removal of Mustard from contaminated soil. With a double wash treatment, differences between water and surfactant treatments are less marked.

Mustard has been shown to be very persistent as discrete globules when undisturbed in still water; the rate of hydrolysis to thiodiglycol is dependent on the rate of

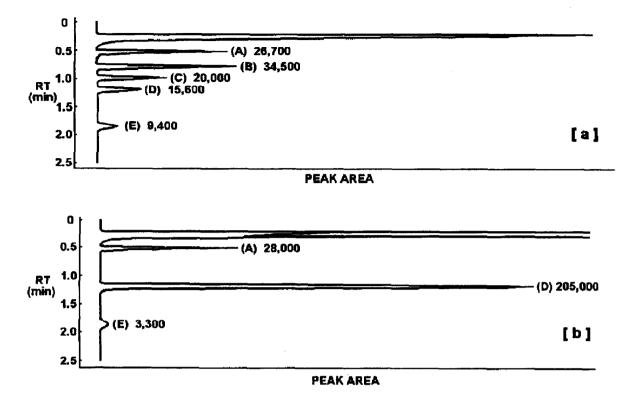


Fig. 1. Gas chromatograms of methylene chloride extract of wash water: (a) immediately after treatment, (b) 24 h after treatment. A = dithiane, B = mustard, C = half-mustard ClCH₂CH₂SCH₂CH₂OH, D = thiodiglycol, E = mustard disulphide ClCH₂CH₂SSCH₂CH₂Cl.

solution from the bulk liquid into the aqueous phase which is slow [3]. Once in solution Mustard is hydrolysed rapidly to thiodiglycol with a half life of approximately 8 min. When absorbed on to soil particles, Mustard presents a large surface area in the form of a thin film and the results indicate that this is readily hydrolysed to thiodiglycol [4]. Fig. 1 illustrates a typical capillary column gas chromatograph of a dichloromethane extract of the wash water immediately after soil treatment. In Fig. 1(a), residual Mustard and the hydrolysis products thiodiglycol and half-Mustard as well as the impurities dithiane and disulphide, which were originally present in the Mustard, are identifiable. Fig. 1b shows the gas chromatograph of a dichloromethane extract of the wash water 24 h after treatment; only thiodiglycol remains as a breakdown product. The original impurities in the Mustard, dithiane and disulphide also are evident and identified by GC/MS. The treatment water would not contain any residual Mustard after 24 h and could be disposed of safely.

Table 2 details the recovery and residual percentages of Soman from Soil F for a similar range of washing solutions to those for Mustard. There is little significant difference between the various aqueous solutions. At the upper contamination level of 20 mg/g, 16–19% Soman remains on the soil after a single treatment and inclusion of 10% ethanol in the water wash does not greatly improve the washing process.

Treatment	Level (mg/g) Extraction after treatment (%) ^a			Total residual (%)
		Acetone	CH ₂ Cl ₂	-
Water wash	5	15.54 ± 0.48	0.25 ± 0.02	16
	20	16.78 ± 0.50	0.41 ± 0.03	17
10% ethanol	5	14.91 ± 0.29	0.39 ± 0.01	15
	20	15.37 ± 0.52	0.34 ± 0.02	16
0.1% CTAC	5	17.72 ± 0.94	0.60 ± 0.10	18
	20	18.44 ± 0.36	0.53 ± 0.04	19
0.1% DBS	5	15.68 ± 0.40	0.35 ± 0.08	16
	20	16.34 ± 0.46	0.41 ± 0.02	17
$2 \times$ water wash	5	3.69 ± 0.13	0.08 ± 0.01	4
	20	3.46 ± 0.18	0.08 ± 0.01	4
$2 \times 10\%$ ethanol	5	3.50 ± 0.12	0.09 + 0.01	4
	20	3.66 ± 0.16	0.12 ± 0.01	4
$2 \times 0.1\%$ DBS	5	3.57 ± 0.18	ND	4
	20	3.13 ± 0.22	0.06 ± 0.00	3
2 × 0.1% CTAC	5	4.59 + 0.14	0.08 ± 0.01	5
	20	4.32 ± 0.11	0.11 ± 0.01	4

Table 2 Recovery of Soman from soil F contaminated at 5 and 20 mg/g after various physical washing processes

* Mean \pm SD (n = 4); ND = not detected.

Tab	le 3
Soil	properties

Soil	Texture	pH	Organic carbon	Clay (%)
F	Loamy sand	5.1	4.6	1
W	Sandy loam	7.6	0.7	8
E	Clay loam (heavy)	5.5	7.3	49
Y	Sandy clay loam	6.2	13.0	35

However, as with Mustard, washing twice reduces the residual agent levels further to 3-4%.

The three washing processes involving plain water, CTAC and DBS were studied for a range of soils of varied properties contaminated with Mustard and Soman. Essential soil properties are detailed in Table 3 and range in pH, organic carbon content and clay content.

In Table 4 we detail the recovery of Mustard and Soman from soils after treatment with water only. Soils containing higher contents of clay and organic carbon have been shown to exhibit greater sorption effects by agents [5, 6]. Soils with high clay

Agent	Soil	Level (mg/g)	Extraction after treatment (%) ^a		Total residual (%)
			Acetone	CH ₂ Cl ₂	
Mustard	Y	5	11.47 ± 0.34	1.19 ± 0.03	13
		20	11.12 ± 0.19	0.86 ± 0.02	12
Mustard	Ε	5	8.55 ± 0.07	2.57 ± 0.14	11
		20	11.19 ± 0.69	1.85 ± 0.06	13
Mustard	W	5	7.65 ± 0.15	1.89 ± 0.15	10
		20	12.53 ± 1.31	1.35 ± 0.08	14
Soman	Y	5	13.64 ± 0.35	0.41 ± 0.02	14
		20	15.22 ± 0.79	0.52 ± 0.07	16
Soman	Е	5	17.06 ± 0.63	0.77 ± 0.02	18
		20	17.16 ± 0.73	0.84 ± 0.03	18
Soman	w	5	7.78 ± 1.06	0.14 ± 0.00	8
		20	10.08 ± 0.64	0.08 ± 0.01	10

 Table 4

 Recovery of Mustard and Soman from soils contaminated at 5 and 20 mg/g after washing with water

* Mean \pm SD (n = 4).

content or with high organic carbon content will adsorb agent and recovery may be lower than in soils with low clay and low organic carbon content. Agent that is not adsorbed by clay or absorbed by organic matter is potentially removable by physical treatment processes such as washing. At both contamination levels on the three soils there is little difference in the residual level of Mustard after treatment. For Soman, Soils Y and E with higher levels of organic carbon exhibit higher residuals than Soil W.

Tables 5 and 6 detail the recovery of agents from soils after treatment with solutions of surfactants CTAC and DBS. Neither surfactant improves the removal of Mustard from the soils; with CTAC, soils with high organic carbon show greater residual Mustard levels than those with lower. For example, at the high spiking level Soil Y has residual Mustard of 31% and 34% after treatment with CTAC and DBS, respectively. This compares with 12% for treatment with water alone. For Soil W with low organic carbon there is little difference between treatment with water or DBS; CTAC treatment results in a higher residual of 18% Mustard.

Soman exhibits a similar behaviour to Mustard. With water and DBS treatments, Soil W retains less Soman than Soils E and Y with higher organic carbon. CTAC treatment is also less effective than water or DBS treatment although the differences are not so marked as with Mustard; this may be attributable to the more polar nature of Soman leading to lower solubilities in soil carbon and a more effective removal by washing.

Militarily, the preferred process for the destruction of chemical agent contamination on soil is the application of a concentrated slurry of chlorinated lime in water [7]. Table 5

Agent	nt Soil Level (mg/g)	Level (mg/g)	Extraction after treatment (%) ^a		Total residual (%)
		Acetone	CH ₂ Cl ₂		
Mustard	Y	5	15.74 ± 1.14	1.23 ± 0.04	17
		20	29.14 ± 2.03	1.78 ± 0.19	31
Mustard	Ε	5	13.19 ± 0.54	3.15 ± 0.22	16
		20	24.28 ± 1.12	3.42 ± 0.14	28
Mustard	W	5	8.26 ± 0.62	2.06 ± 0.17	10
		20	16.87 ± 0.43	1.06 ± 0.08	18
Soman	Y	5	18.07 ± 0.27	0.90 ± 0.02	19
		20	17.62 ± 1.58	0.85 ± 0.08	18
Soman	Е	5	19.03 ± 0.65	1.35 ± 0.12	20
		20	19.03 ± 0.59	1.34 ± 0.06	20
Soman	W	5	18.75 ± 0.42	0.19 ± 0.03	1 9
		20	15.96 ± 0.81	0.16 ± 0.01	16

Recovery of Mustard and Soman from soils contaminated at 5 and 20 mg/g after washing with CTAC(0.1%)

^a Mean \pm SD (n = 4); ND = not detected.

 Table 6

 Recovery of Mustard and Soman from soils contaminated at 5 and 20 mg/g after washing with DBS (0.1%)

Agent	Soil	Level (mg/g)	Extraction after treatment (%) ^a		Total residual (%)
			Acetone	CH ₂ Cl ₂	
Mustard	Y	5	21.62 ± 1.02	1.30 ± 0.07	23
		20	31.71 ± 1.18	2.12 ± 0.20	34
Mustard	E	5	11.41 ± 0.72	1.72 ± 0.01	13
		20	12.00 ± 1.91	1.53 ± 0.17	14
Mustard	w	5	12.21 ± 0.22	1.13 ± 0.11	13
		20	12.28 ± 0.74	1.10 ± 0.06	13
Soman	Y	5	15.72 ± 1.11	0.53 ± 0.02	16
		20	18.00 ± 0.53	0.59 ± 0.05	19
Soman	Ε	5	17.42 ± 0.48	1.04 ± 0.04	18
		20	17.72 ± 0.38	1.15 ± 0.02	19
Soman	w	5	10.31 ± 0.97	0.06 + 0.01	10
		20	12.03 ± 0.98	0.13 ± 0.01	12

* Mean \pm SD (n = 4).

Such a mixture is highly corrosive and deleterious to the soil environment. While washing with water is one of the least damaging procedures that can be envisaged for soil treatment, some low levels of residual agents remain. It is probable than such residual levels are not harmful and would naturally degrade through chemical and

Treatment	Level (mg/g)	Extraction after treatment (%) ^a		Total residual (%)
		Acetone	CH ₂ Cl ₂	
0.1 M NaHCO3	5	7.84 ± 0.88	ND	8
	20	20.83 <u>+</u> 1.44	1.10 ± 0.06	22
$0.1 M \operatorname{Na_2CO_3}$	5	3.02 ± 0.13	ND	3
	20	4.87 ± 0.27	0.36 ± 0.03	5
$0.2 M Na_2CO_3$	5	2.88 ± 0.21	ND	3
- •	20	6.09 ± 1.05	0.60 ± 0.02	7
0.1 M NaOH	5	2.85 ± 0.36	ND	3
	20	5.50 ± 0.31	0.44 ± 0.02	6
0.2 M NaOH	5	3.50 ± 0.63	ND	4
	20	28.30 ± 0.67	1.10 ± 0.26	29
0.25% NaOCl	5	2.05 ± 0.03	ND	2
	20	11.18 ± 0.40	0.64 ± 0.02	11
1% NaOCi	5	1.51 ± 0.09	ND	2
	20	2.89 ± 0.17	0.13 ± 0.02	3

 Table 7

 Recovery of Mustard from soils contaminated at 5 and 20 mg/g after chemical treatment

* Mean \pm SD (n = 4); ND = not detected.

biological processes. In an attempt to eliminate completely residual agent we have investigated a number of simple chemical treatment processes. These range from $NaHCO_3$ to NaOCl solutions.

In Table 7 we present the recovery of Mustard from Soil F after a range of chemical treatments. The military terrain decontamination process essentially involves stoichiometric oxidation and may be represented by sodium hypochlorite (NaOCl) solutions and these are the most effective treatments. Treatment with 0.25% NaOCl is effective for the lower spiking level but insufficient for the upper. Treatment with 1% NaOCl is effective at both spiking levels. Of interest is the treatment with Na₂CO₃ and NaOH which traditionally have not been recognised as decontaminants for Mustard. Both of these treatments are almost as effective as hypochlorite in cleaning Mustard-contaminated soil and are more effective than a single wash with water.

Table 8 shows the residual Soman on Soil F after the chemical treatments. Since the alkaline hydrolysis of nerve agent is catalytic it would be expected that Na_2CO_3 and NaOH treatments would be effective processes for Soman. This is confirmed with residual levels of Soman <1% for both treatments; 1% NaOCI is also an effective treatment for Soman but less so than alkali. To minimise environmental degradation the least aggressive treatment should be employed; for Soman this would be 0.1 M Na₂CO₃; the lower pH in comparison to 0.1 M NaOH and the absence of chlorine would be less damaging to soil ecology. If low Soman levels of 4% are tolerable, then a double wash with water would be the least damaging process. In the event, traces of

Treatment	Level (mg/g)	Extraction after treatment (%) ^a		Total residual (%)
		Acetone	CH ₂ Cl ₂	
0.1 M NaHCO ₃	5	10.42 ± 0.22	0.36 ± 0.02	11
	20	11.96 ± 1.20	0.45 ± 0.06	12
$0.1 M \operatorname{Na_2CO_3}$	5	0.14 ± 0.01	0.06 ± 0.01	< 1
	20	0.27 ± 0.02	0.04 ± 0.01	< 1
$0.2 M Na_2CO_3$	5	0.09 ± 0.02	0.01 ± 0.01	< 1
	20	0.12 ± 0.01	0.04 ± 0.01	< 1
0.1 M NaOH	5	ND	ND	ND
	20	0.07 ± 0.01	0.03 ± 0.01	< 1
0.2 M NaOH	5	ND	ND	ND
	20	ND	ND	ND
0.25% NaOCI	5	6.96 ± 0.33	0.41 ± 0.01	7
	20	10.42 ± 0.44	0.51 ± 0.08	11
1% NaOCl	5	0.81 ± 0.07	0.03 ± 0.01	< 1
	20	1.72 ± 0.16	0.09 ± 0.01	2

 Table 8

 Recovery of Soman from soil F contaminated at 5 and 20 mg/g after chemical treatment

^a Mean \pm SD (n = 4); ND = not detected.

Soman would degrade naturally through hydrolysis and microbial action although the latter would be slow.

While washing twice with water reduces Mustard and Soman levels to 4%, chemical treatment with 1% NaOCl will reduce Mustard levels to 2–3% and with $0.1 M \text{ Na}_2\text{CO}_3$ to 3–5%. The optimum choice for maximum destruction of Mustard remains hypochlorite; however a more benign treatment is a double water wash. In this event residual Mustard levels would be expected to decline further in the wet soil on standing.

Table 9 shows that similar results can be obtained for a range of soils with differing characteristics when treated with Na_2CO_3 or NaOCI. The use of NaOC1 for the optimum destruction of Mustard is confirmed; similarly Na_2CO_3 is effective for Soman. For a universal clean-up reagent, 0.1 *M* or 0.2 *M* Na_2CO_3 would be effective against Soman, and only marginally less so against Mustard.

The present results indicate that Mustard and Soman, when finely dispersed, may not persist in soils subject to significant rainfall as the agents may be washed through the surface soil into the water table and decomposed. In covered areas which are dry, such as sheds, hangars, etc., such natural washing obviously will not occur and can be induced by the processes shown in this study. Since Soman is moderately soluble in water (solubility 2.1 at 20 °C), large deposits of this agent in soil may be dispersed gradually. However, larger deposits of Mustard will not be dispersed and would need to be identified and specially treated. This accords with our experience of disposal

Agent	Soil	Treatment	Total residual ^a (%)
Mustard	Y	0.2 M Na ₂ CO ₃	6.9 ± 0.6
Soman	Y	$0.1 M Na_2 CO_3$	1.3 ± 0.1
Mustard	Y	1% NaOCI	2.4 ± 0.6
Soman	Y	1% NaOCl	3.2 ± 0.2
Mustard	Ε	$0.2 M \operatorname{Na_2CO_3}$	3.2 ± 0.7
Soman	Ε	$0.1 M Na_2 CO_3$	1.0 ± 0.1
Mustard	Ε	1% NaOCl	2.5 ± 0.1
Soman	Е	1% NaOCl	4.6 ± 0.4
Mustard	w	$0.2 M \operatorname{Na_2CO_3}$	4.9 ± 0.2
Soman	W	$0.1 M \operatorname{Na_2CO_3}$	0.2 ± 0.1
Mustard	W	1% NaOCI	2.2 ± 0.1
Soman	W	1% NaOCI	0.5 ± 0.1

Table 9 Recovery of Mustard and Soman from soils contaminated 20 mg/g after treatment with Na₂CO₃ or NaOCl

^a Mean \pm SD (n = 4).

sites; Mustard persists where it is in bulk or absorbed into other phases such as partially combusted organic matter. The current study demonstrates how dispersed Mustard and Soman in soils not subject to major wetting can be removed.

Bulk aqueous washings from soils contaminated with Mustard or Soman and treated with sodium carbonate or sodium hydroxide solutions will contain the breakdown products thiodiglycol or sodium methylphosphonate and pinnacolyl alcohol, respectively. While these products are not considered to be toxic from the military perspective, caution should be taken during ultimate disposal. Disposal through incineration or by plasma torch would be the most rigorous solution; in practice landfill disposal may be entirely adequate, dependant upon local environmental regulations.

4. Conclusions

The removal of chemical agents Soman and Mustard from a variety of soils has been studied using simple washing processes and chemical treatment. A double wash with plain water is shown to be effective in removing Mustard from soil and the wash water is free from Mustard after 24 h. Soils with a high content of organic carbon are more difficult to decontaminate due to partitioning of agent into the organic phase. Solutions of hypochlorite are the most effective for the destruction of Mustard but sodium carbonate solution is almost as effective. As with Mustard, a double wash using water is shown to remove 96% of Soman contamination from soil. Sodium hydroxide solution is the most effective decontaminant for Soman; however, sodium carbonate is almost as effective and may be less harmful to soils. Solutions of hypochlorite are less effective against Soman than alkali. Overall, the most efficient process for the destruction of both Mustard and Soman on soils is treatment with sodium carbonate solution which is also less damaging than hypochlorite or sodium hydroxide.

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