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Nanomaterials based Decontamination Formulation for use in Personal Decontamination Kit Against Chemical Warfare Agents

G.K. Prasad*, Anshoo Gautam, G.M. Kannan, J. Acharya, A.K. Gupta, V.B. Thakare, and B. Singh

DRDO-Defence Research and Development Establishment, Gwalior - 474 002, India
**E-mail: gkprasad2001@gmail.com*

ABSTRACT

Present paper relates to nanomaterials based decontamination formulation made up of TiO_2, MgO, and ZnO nanoparticles for the use against chemical warfare agents. This decontamination formulation was prepared by mixing 90 per cent of TiO_2 nanoparticles of 5-15 nm size range, 8 per cent of MgO nanoparticles of 5-15 nm size range, and 2 per cent of ZnO nanoparticles of 20-30 nm size range. Prepared formulation exhibited 98-99 per cent of physical removal efficiency against contaminated glass, rubber, painted metal, metal surfaces. It efficiently removed chemical warfare agents from contaminated skin thus ameliorating chances of fatality which is expected due to skin penetrated chemical warfare agents. It also chemically degraded 97 per cent of sulphur mustard in 24 h, 99.9 per cent of sarin in 2 h, respectively unlike Fuller's earth which chemically degraded only 63 per cent of sulphur mustard and 59 per cent of sarin in 24 h .

Keywords: Sorbent; Nanomaterials; Decontamination; Sulphur mustard; Sarin, Physical removal efficiency; Chemical reactivity

1. INTRODUCTION

Sorbent decontaminants (SD's) are commonly used for physically removing the chemical warfare agents (CWA's) from contaminated surfaces. They remove CWA's in liquid form from contaminated surfaces by physically adsorbing them. After adsorptive removal, the sorbent decontaminant is removed by wiping it off to obtain clean surfaces. Fuller's earth (FE) clay with high surface area and large capacity to adsorb toxic chemicals has been used as SD in personal decontamination kits (PDK) against CWA contaminated surfaces. This clay has been used to aid the decontamination of contaminated skin, NBC suit, respiratory mask, boots, gloves, and small arms at battle field and laboratory sites where CWA contamination takes place. Nonetheless, removal of CWA from surfaces by FE was found to be curtailed posing threat of contact with left over CWA; hence its use is constrained[1-2].

On the other side, MgO, CaO, MnO_2, ZnO, CuO, TiO_2, CuO, ZnO nanoparticles exhibited excellent chemical reactivity against CWA[3-10]. Among these metal oxide nanoparticles, TiO_2, MgO, and ZnO nanoparticles displayed better decontamination properties against contaminated surfaces[11-12]. When these nanoparticles are mixed in suitable proportion, reactive SD[13-15] with high surface area and enhanced chemical reactivity against CWA's could be obtained. Obtained nanomaterials based decontamination formulation (NDF) is expected to show striking personal decontamination applications against contaminated skin. Moreover, there are no reports in literature on the use of NDF for personal decontamination applications against chemical warfare agents. Inspired by the above, we have prepared NDF based on TiO_2, MgO, ZnO nanomaterials and characterised it by X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), and nitrogen adsorption techniques. The physical removal efficiency (PRE) values, chemical degradation efficiency (CDE) values of SD's like NDF, individual TiO_2, MgO, ZnO nanoparticles against 2, 2'-dichlorodiethyl sulfide (Sulfur mustard, HD) and isopropyl methyl phosphonofluoridate (Sarin,GB) were studied using gas chromatography (GC). Additionally, it was evaluated for its decontamination efficiency on animal model to assess its suitability for use in PDK against CWA contaminated skin.

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2. MATERIALS AND METHODS

2.1 Materials

Magnesium acetate tetrahydrate, Zinc acetate dihydrate, oxalic acid dihydrate, acetonitrile, dichloromethane (DCM), titanium tetrachloride, isopropanol, ethanol were obtained from E. Merck India Ltd. HD and GB of 99 per cent purity were synthesised in Schedule I facility of our establishment and decontamination experiments were also carried out at the same facility (These agent are toxic, should be handled and studied by trained personnel only).

2.2 Characterisation

SEM data of NDF was recorded on Quanta 400, environmental scanning electron microscope of M/s.Field Electron and Ion Company, The Netherlands. Sample was placed on copper stubs with adhesive tape and coated with gold

prior to observations by SEM. TEM data were recorded using a field emission transmission electron microscope, Tecnai TEM of M/s.Field Electron and Ion Company, The Netherlands. The sample was dispersed in acetone and sonicated for 10 min prior to the fabrication of grids for TEM analysis. XRD patterns were recorded by the instrument of M/s. Panalytical Inc, The Netherlands using Cu Kα radiation. Nitrogen adsorption data were collected using surface area analyser of M/s. Micromeritics, USA. Sample was degassed at 200 °C for overnight at 10^{-3} mmHg and analysed for nitrogen adsorption at 77°K. Moisture content of the samples was determined by measuring the decrease in weight after heating them to constant weight at 120 °C.

2.3 Preparation of Nanomaterials based Decontamination Formulation

Initially, TiO_2, ZnO, and MgO nanoparticles were prepared individually as per the procedures given in the following sections. NDF was prepared by thoroughly mixing 90 per cent TiO_2 nanoparticles of 5-15 nm size range, 8 per cent MgO nanoparticles of 5-15 nm size range, and 2 per cent ZnO nanoparticles of 20-30 nm size range with help of planetary ball mill.

2.3.1 Synthesis of TiO_2 Nanoparticles

TiO_2 nanoparticles were prepared using a modified sol-gel method with $TiCl_4$ and ethanol. 50 g of $TiCl_4$ was slowly added to 500 mL of ethanol at 0 °C in an ice bath and the produced reaction mixture was stirred for 12 h. Obtained sol was stirred further for 2 h at ambient conditions. Subsequently, it was put in an oven and heat treated at 87 °C for 3 days for producing TiO_2 nanoparticles[16].

2.3.2 Synthesis of MgO Nanoparticles.

Initially, a solution was prepared by adding 12.6 g oxalic acid to 200 mL of isopropanol and heated to 70°C. To this suspension, a hot solution (70 °C) containing 21.28 g of magnesium acetate in 50 mL of isopropanol was added in drop wise manner. After complete addition and stirring for 1 h at 80 °C, suspension was filtered and dried at 100°C for 4 h. Obtained solid was calcined at 500 °C for 2 h to obtain MgO nanoparticles[17].

2.3.3 Synthesis of ZnO Nanoparticles.

ZnO nanoparticles were prepared by sol–gel method. Isopropanol solutions of oxalic acid and zinc acetate were added in a reaction flask with a constant heating and stirring. Temperature was maintained at 60°C throughout the reaction. Obtained materials were filtered, dried at 80°C and calcined[18] at 500 °C.

2.4 Procedure to Examine Physical Removal Efficiency and Chemical Degradation Efficiency of SD's NDF, TiO_2, MgO, ZnO nanoparticles, and Fuller's Earth

PRE studies were performed by spreading HD or GB (20 µL) on the surface of substrates (2 cm^2 each of glass, rubber, metal , and painted metal pieces) with a thickness of 2-4 mm. Stainless steel was used for making metal surfaces. Rubber sheets were obatained by cutting face mask and over boots of individual protective equipment. They were made up of bromobutyl rubber. After spreading the agent with glass rod, SD at a ratio of 1:50 per cent W/W (Agent: sorbent materials) was palced on contaminated surface. After 1 min, powder was wiped off and substrate was rinsed thoroughly with 5 mL of acetonitrile for five times (each time 1 mL) to extract remaining HD or GB. Concentration of HD or GB was monitored by GC equipped with FID detector.

Reaction of HD or GB with SD was studied by treating 20 µL of agent with it at a ratio of 1: 50 per cent W/W (Agent: sorbent materials). The residual or unreacted HD or GB was extracted from treated SD by using 5 mL of acetonitrile (1st time 3mL, 2nd time 1mL, 3rd time 1mL) to make sure the complete extraction. Extracted solution was looked at for GB by GC equipped with flame ionisation detector and BP5 column (30 m length, 0.5 mm i.d, and 0.5 µm film thickness) under a temperature programme from 40 to 210 °C at a rate of 5 °C/ min. In the case of HD, extracted solutions were quantitatively analysed by GC-FID under isothermal conditions at 110 °C. The concentrations of extracted solutions were calibrated with standard solutions for accurate quantification. The injection port was kept at 240 °C and the detection port at 250 °C.

2.5 Procedure to Evaluate Decontamination Efficiency using Animal Model

Swiss albino male mice (~25 g) were weighed and grouped. Each group contains 5 animals. All the animals were clipped on dorso-caudal surface, a day before experiment. After 16 h, 20 µL of > 98 per cent pure HD was applied percutaneously on 1 cm^2 (approx) area of the hair clipped skin on each mouse.

- In Positive control group all the animals were exposed with HD and kept for observation for next 14 days.
- In treated groups all the animals were decontaminated by test formulation after 30 s of percutaneous application of neat HD. For decontamination, test formulation was quickly sprinkled over the affected area and cotton swab was used to apply the formulation firmly before removing from skin. Thereafter, the treated animals were maintained for 14 days to observe gross body weight changes and any ensuing mortality.
- In negative control group all the animals were applied with test formulation without HD exposure by same procedure as mentioned in treated group. These animals were also kept for 14 days observation.
- Institutional animal's ethical committee approval number is TOX-61/56/GMK.

HD was only used for testing decontamination efficiency on animal model as GB has relatively low absorption by skin[19].

3. RESULTS AND DISCUSSIONS

3.1 Characterisation of Nanomaterials based Decontamination Formulation

The TEM image of NDF is shown in Fig. 1 TEM image depicts nanoparticles of spherical shape and size ranging from 5 to 30 nm to major extent. These nanoparticles are found to

have been aggregated due to van der Waals attractive forces. SEM image (Fig. 2) also illustrates the aggregates of spherically shaped nanoparticles of size less than 100 nm.

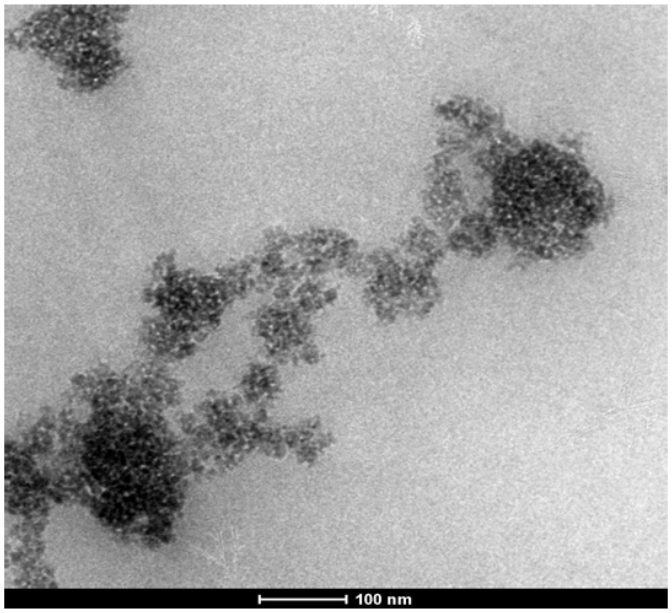

Figure 1. Transmission electron microscopy image of nanomaterials based decontamination formulation.

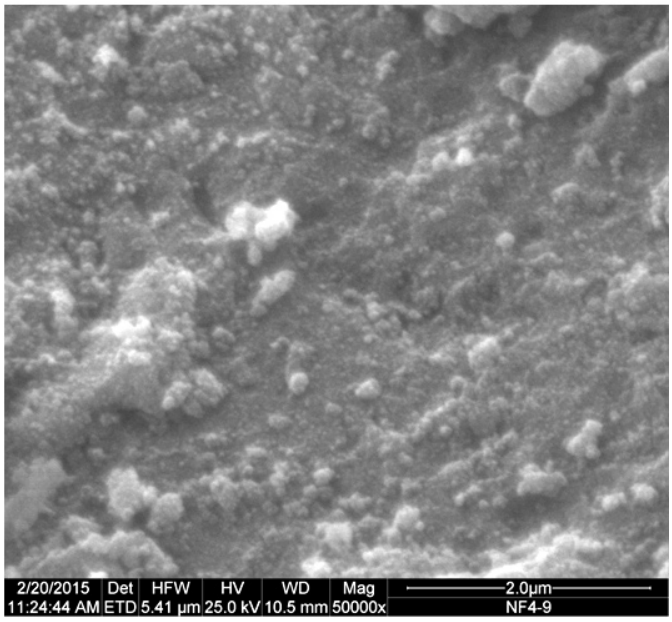

Figure 2. Scanning electron microscopy images of nanomaterials based decontamination formulation.

The XRD pattern of NDF is shown in Fig. 3 XRD pattern shows peaks at 25.36 (100), 37.92 (004), 47.82 (200), 54.62 (106), and 62.88 (215) and indicate the presence of TiO_2 nanoparticles of anatase phase. It also depict the peaks at 31.65 (100), 34.25 (002), 36.09 (101) indicating the presence of ZnO nanoparticles of zincite phase and shows peaks at 42.867(200), 62.184(220) indicating the presence of MgO nanoparticles of periclase phase. The crystallite sizes of TiO_2, MgO, and ZnO were found to be 5.6 nm, 11 nm, and 30 nm as per Scherrer analysis of the data and the same is consistent with TEM observations[12].

Nitrogen adsorption-desorption isotherms of NDF is shown in Fig. 4. Isotherm shows Type IV isotherms with H3 type hysteresis typical of mesoporous materials with slit type of pores according to IUPAC classification[20]. The formulation exhibited surface area of 225 m^2/g and pore volume 0.32 mL/g. The pore size distribution data indicated pore maxima of 4.6 nm indicating the presence of mesopores which might have been formed due to aggregation of nanoparticles. Surface area data of NDF, individual TiO_2, MgO, ZnO nanoparticles is shown in Table 1.TEM, XRD, nitrogen adsorption data of individual TiO_2, MgO, ZnO nanoparticles was already reported hence are not described here[21].

3.2 Physical Removal Efficiency of Nanomaterials based Decontamination Formulation, TiO_2, MgO, ZnO Nanoparticles, and FE

NDF, individual TiO_2, ZnO, MgO nanoparticles, and FE were investigated for their PRE's against HD or GB when used on contaminated surfaces like glass, metal, painted metal , and rubber. These surfaces were opted as most of the military equipments, vehicles, arms, and individual protective equipments are made up of above materials.

FE, NDF, TiO_2 nanoparticles, MgO nanoparticles exhibited 99 to 99.9 per cent of PRE's against HD or GB contaminated glass, metal, painted metal, rubber surfaces, whereas, ZnO nanoparticles demonstrated 90 to 96 per cent of PRE's against them. Amongst all, ZnO nanoparticles demonstrated lower PRE and the same can be attributed to lower surface area. Due to low surface area, adsorption capacity of ZnO nanoparticles towards HD or GB was low, hence exhibited lower PRE (Table 1).

3.3 Chemical Degradation Efficiency of HD and GB with Nanomaterials based Decontamination Formulation, TiO_2, MgO, ZnO Nanoparticles, and FE.

After physical decontamination of tainted surfaces, they were rendered safe. However, danger still exists as the SD holds the contaminant within itself. Used SD's are to be contained carefully to avoid cross contamination. They are to be disposed off properly by burying it under earth or they are to be decontaminated to avoid health hazards. This situation can be ameliorated if we use reactive SD which have high

Table 1. Surface area and physical removal efficiency values of of NDF, TiO_2 nanoparticles, MgO nanoparticles, ZnO nanoparticles and Fuller's earth against HD and GB.

Sorbent decontaminants	Surface area m^2/g	Moisture content (%)	Physical removal efficiency against contaminated surfaces (%)
NDF	225	3	>99
TiO_2 nanoparticles	222	3	>99
MgO nanoparticles	146	0.5	>99
ZnO nanoparticles	30	0.5	~96
Fullers earth	90	4	>99

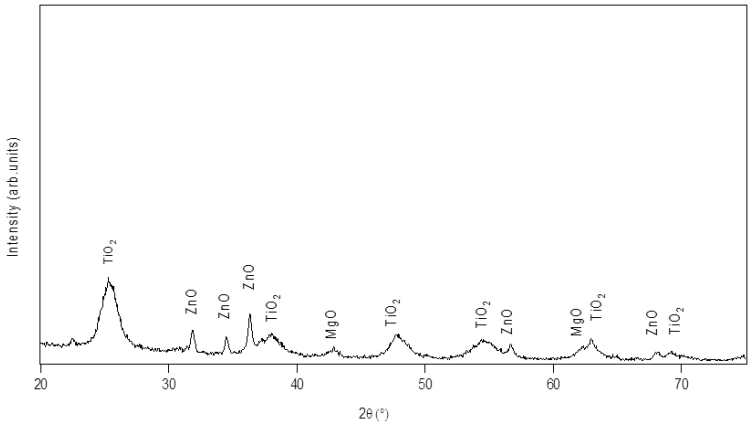

Figure 3. X-ray diffraction data of nanomaterials based decontamination formulation.

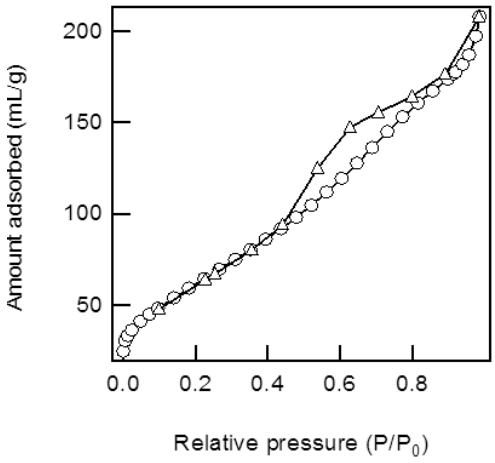

Figure 4. Nitrogen isotherms of nanomaterials based decontamination formulation.

adsorption capacity as well as adequate reactivity[21]. In order to assess NDF, FE, TiO_2, MgO, and ZnO nanoparticles of their suitability as reactive SD, they were tested for their chemical degradation efficiency against HD or GB.

Chemical degradation efficiency data of NDF, TiO_2, MgO, ZnO, and FE against HD and GB are presented in Table 2. NDF, TiO_2, MgO, ZnO nanoparticles, and FE chemically degraded 98 per cent, 98per cent, 98 per cent, 86.5 , and 63 per cent of HD in 24 hr and the SD's exhibited the following trend; NDF=TiO_2=MgO>ZnO > FE. Whereas, NDF, TiO_2, MgO, ZnO nanoparticles, and FE chemically degraded 99.9 per cent, 96per cent, 99.9 , 53 per cent, and 6 per cent of GB in 20 min and the SD's exhibited the following trend; NDF=MgO>TiO_2>ZnO > FE.

Table 2. Chemical degradation efficiency of NDF, TiO_2 nanoparticles, MgO nanoparticles, ZnO nanoparticles and Fuller's earth

Name of CWA	Chemical degradation efficiency (per cent)					Ratio of, agent: SD	Reaction time
	NDF	ZnO	TiO_2	MgO	Fuller's earth		
HD	98	86.5	98	98	63	1:50	24 h
GB	99.9	53	96	99.9	6	1:15	20 min

Chemical reactivity of the above SD's can be attributed to the presence of moisture, surface hydroxyl groups, Lewis acid sites, basic sites, defect sites, unusual lattice planes, etc. Mechanism of chemical degradation, products formed as per GC-MS, FT-IR data were already reported hence we are not discussing here[20]. Amongst the tested SD's, NDF and MgO nanoparticles demonstrated superior CDE values against HD and GB based on related studies and were found to be suitable for use in PDK.

3.4 Decontamination of Efficiency of Nanomaterials based Decontamination Formulation, TiO_2, MgO, ZnO Nanoparticles, and FE Against HD on Animal Model

Of all CWA's, HD has relatively high dermal toxicity, hence, it was tested as representative CWA for assessing decontamination efficiency of SD's against contaminated animal skin. It has already been reported that HD showed differential route specific toxicity with maximum toxic effect through percutaneous route[22]. Study could indicate the suitability of SD's for use in PDK[18].

It was observed that, mice of positive control group which were not decontaminated using test formulation died within 16 h of application. The mortality in this group of animals was due to the highly toxic nature of sulphur mustard. In addition to this, in treated groups where decontamination was done by MgO nanoparticles or ZnO nanoparticles individually also died within 7 days after decontamination.

Whereas in the groups where mice were treated with NDF, TiO_2 nanoparticles and FE showed maximum survival with marginal body weight loss compared to other treated groups. This weight loss might be due to the toxic effect of sulphur mustard that had penetrated through skin during 30 seconds after the application but prior to decontamination. It was grossly observed that in 48 hours post exposure animals showed reduced body movements and lethargy. There were no other noticeable changes observed in either group of animals after 4 days as compared to normal group of mice. It could be concluded from this study that decontamination efficiencies of NDF, TiO_2, FE are sufficient enough towards minimizing the injurious effects of liquid toxicant. Whereas, ZnO nanoparticles and MgO nanoparticles could not reduce the injurious effects of liquid toxicant hence all the mice were died within 7 days. Though it was observed that these nanoparticles are able to delay mortality in animals after HD exposure as compare to positive control. Animal decontamination study concluded that NDF, TiO_2 and FE can be used in personal decontamination kit (PDK) after preclinical toxicity and safety evaluation.

In addition to these observations, SD's with different compositions of TiO_2, MgO and ZnO nanoparticles were also made and tested for their PRE, CDE, and decontamination efficiency on animal model. Studies have indicated that NDF with 90 per cent TiO_2 nanoparticles, 8 per cent MgO nanoparticles and 2 per cent ZnO nanoparticles exhibited better decontamination properties than others. If we observe the above data, NDF and TiO_2 nanoparticles (alone) have exhibited similar physical removal efficiency, decontamination efficiency

on animal model, and chemical degradation efficiency against HD. However, NDF exhibited better reactivity against GB than TiO_2 hence it was considered further for use in PDK. This observation could be attributed to the added reactivity due to addition of MgO, ZnO nanoparticles to TiO_2 in NDF. Both MgO , ZnO nanoparticles were reported to be having excellent reactivity against GB[8,11].

Moreover, recently reported sorbent decontaminants such as CuO–Al_2O_3 mixed oxide aerogel, activated alumina exhibited 99 per cent of PRE, however, they could chemically degrade only 70–80 per cent of HD in 24 h. Fuller's earth exhibited > 98 per cent PRE, however, it lacks optimal chemical degradation efficiency against CWA like HD and GB. XE-555, resin mixture also lack CDE and agents remain intact within this SD even for days. FAST ACT, a recently reported decontaminant composed of nanomaterials exhibited > 98 per cent of PRE against contaminated surfaces and demonstrated excellent chemical reactivity against CWA , however, it could not minimize the injurious effects of liquid toxicant on contaminated skin. However, NDF exhibited > 99 per cent of PRE against contaminated surfaces and it demonstrated relatively better CDE against HD and GB. It also minimised injurious effects of liquid toxicant on contaminated animal skin and was found to be better amongst the above studied and reported SDs as reactive sorbent for use in PDK for decontamination of skin, individual protective equipments, etc.

4. CONCLUSIONS

Nanomaterials based decontamination formulation composed of mixture of 90 per cent TiO_2 nanoparticles, 8 per cent MgO nanoparticles, and 2 per cent ZnO nanoparticles demonstrated excellent decontamination properties against chemical warfare agents HD and GB. It exhibited more than 99 per cent of physical removal efficiency on contaminated surfaces including that of animal skin. It minimised the injurious effects of liquid toxicant on contaminated skin. It also demonstrated superior chemical degradation property against deadly toxic CWA such as HD and GB hence was found to be suitable for use in "decontamination kit" with nanomaterials based decontamination formulation.

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CONTRIBUTORS

Dr G.K. Prasad obtained his PhD from Jiwaji University, Gwalior. He is working as Scientist E in Protective Devices Division, DRDE, Gwalior. His areas of research include development of protection and decontamination devices for CBRN defense.

Dr Anshoo Gautam obtained her PhD from Jiwaji University, Gwalior. She worked as a Scientist D in Pharmacology and Toxicology Division, DRDE, Gwalior. Her research includes studies on biochemical changes induced by chemical warfare agents, screening of antidotes, and nanotoxicology. She is presently working as Scientist D in Division of CBRN Defence, Institute of Nuclear Medicine, Timarpur, New Delhi.

Dr G.M. Kannan did his PhD in Jiwaji University, Gwalior. Presently he is working as Scientist E in Pharmacology and Toxicology Division, DRDE, Gwalior. His research includes studies on biochemical changes induced by chemical warfare agents, screening of antidotes, and nanotoxicology.

Dr J. Acharya did his PhD in Jiwaji University, Gwalior. Presently he is working as Scientist E in Process Technology Development Division, DRDE, Gwalior. His research area includes development of antidotes against CWA.

Dr A.K. Gupta did his PhD in Jiwaji University, Gwalior. Presently he is working as Scientist F in Process Technology Development Division, DRDE, Gwalior. His research area includes development of novel methods for synthesis of compounds of defense interest.

Dr Vikas B. Thakare obtained BTech in Textile Technology from reputed institute V.J.T.I, Mumbai and PhD from ITM University, Gwalior. He worked as scientist in ADRDE , Agra and his area of interest is technical textiles for defense applications and extensively contributed to the development of high performance textiles for Parachutes and recovery systems for space mission. He is presently working as Scientist F in Protective Devices Division, DRDE, Gwalior on the development of next generation protective gears for CBRN Defense.

Dr Beer Singh obtained his PhD from the University of Delhi in 1985. Presently, he is working as Scientist G in Protective Devices Division, DRDE, Gwalior. His areas of research include, Development of advanced physical protection, decontamination, and detection systems against CBW agents. Presently, he is working on advanced individual and collective protection systems against NBC agents, NBC ventilation systems, futuristic NBC carbons, and heterogeneous catalysts, water purification systems, and advanced oxidation technology.