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Chemical Protection Studies of Activated Carbon Spheres based Permeable Protective Clothing Against Sulfur Mustard, a Chemical Warfare Agent

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ABSTRACT

Technological advancements in the field of chemical threat have made it possible to create extremely dangerous chemical warfare agents (CWA). Hence, the effective protection of personnel is very important in a chemical warfare scenario amidst the current climate of terrorism awareness. In particular, body protection plays a substantial role in the chemical defence considering the urgency of situation in the nuclear, biological and chemical environment. Activated carbon spheres (ACS) based permeable chemical protective clothing (coverall) was developed for protection against CWA. The adsorbent material *i.e*, ACS used in this protective clothing provided higher adsorption capacity (1029 mg/g in terms of iodine) and low thermal burden (34 °C WBGT index) compared to earlier indigenously developed NBC suit. This article focuses on the extensive evaluation of chemical protective clothing against sulfur mustard (HD), a CWA. The results revealed that the developed protective clothing provided more than 24 h protection against HD. This chemical protective suit is light weight (< 2.75 kg for XL size). It also has higher air permeability (> 30 $cm^3/s/cm^2$) as well as less water vapour resistance (< 9.6 m^2Pa/W). With continued innovations in materials and attention to key challenges it is expected that advanced, multifunction chemical protective suit will play a pivotal role in the CWA protection scenario.

Keywords: Chemical warfare agents; Chemical protective suit; Activated carbon spheres; Sulphur mustard; Chemical protection; NBC protective suit

1. INTRODUCTION

Nuclear, biological and chemical (NBC) threat is a global concern in the wake of terrorism and several nations having acquired the capability to build these weapons of Mass destruction. In the current scenario, due to increasing science & technological innovation and the proliferation of knowledge through the Internet across the world, the production, storage and dissemination of chemical warfare agents (CWA) has become cost effective, easier to operate and easy execution for the Mass destruction[1]. Hence, appropriate and adequate protective measures are required to protect the combatant and as well as civilian from the lethal effects of CWA. Protection is very in-depth field and it is the first and most important field which prevents the exposure of CWA and it allows the combatant to be effective with minimal performance degradation. Protective clothing is required to protect the wearer against a variety of chemical and biological agents[2]. Protection is required against liquid droplets, aerosol and vapour of chemical warfare agents which can penetrate dermally and are threat to the combatant[3]. Therefore, there is a requirement to develop the NBC protective clothing which can be used effectively under NBC scenario without affecting the tactility of combatant.

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During World War I, the protective clothing used against CWA was impermeable in functionality as it was made up of rubber[4]. As such impermeable protective suits do not offer any adsorption capacity; they are simply impermeable barriers for chemical warfare agents[5]. The material restrains the passage of CWA and also prevents the passing out of perspiration which leads to heat buildup and makes personnel very uncomfortable. Hence, it can be worn for short period of time, and limits its applicability. The technological advancement has been taken place and many improvements[6,7] have been pursued after World War-I, particularly focus was directed towards the development of NBC suit by emphasising on what the use of the technology is for and who is going to use it[8].

To address the aforementioned shortcomings, impermeable protective clothing and permeable clothing was designed and developed, by using a layer of fine powder of activated carbon[9], either bound in polyurethane foam or other adhesives came in to the International market. In case of permeable NBC suit, an adsorbent layer permits water vapour in the form of perspiration released from the body to pass through and simultaneously adsorbs CWA and thereby prevents them from passing through to the skin[10]. Afterwards, many improved versions of protective clothing was developed for military and defence application based on charcoal coated fabric layers, impregnated polyurethane foam, non-wovens with bonded charcoal and carbonised and activated carbon cloth came in existence[11]. The properties of activated carbon continue to be improved by further refining the present technology. However, all of these approaches have still two major limitations, *i.e.* a) inability to provide sufficient carbon on the surface of the carrying material without blocking off its breathability; and b) degradation of the charcoal due to aging of the bonding process. Other common limitations present in some of these systems are: shedding of charcoal through abrasion, lack of launderability, lack of breathability and high thermal insulation[11]. Thus, the reuse of this suit is not favourable.

In the past few decades, various types of adsorbents including zeolites[12], metal organic frameworks (MOF)[13,14], functionalised porous silica, porous polymers and carbonaceous materials[15] have been investigated for development of advanced NBC suit. Among them, MOF[13] and nanoporous carbons have gained much attention due to its higher surface area. Despite the excellent adsorption capacities of MOFs and nanocarbon this materials could not replace especially commercially available activated carbons due to their cost, size and cumbersome synthesis. In addition, MOFs are water sensitive, they can chemisorb water and their porous structure can be destroyed upon exposure to water vapour[16]. Some new generation of permeable NBC protection clothing based on semi permeable membrane based material have been explored for hot climate condition, however, this membrane technology is still at their infancy stage. Therefore, there is an urgent need to develop adsorbent materials which can address all the above aforementioned drawbacks without compromising its protective potential and comfort to the wearer or combatant. To overcome these drawbacks, carbonaceous materials, particularly microporous activated carbon spheres (ACS) as an adsorbents are of great interest because of it high mechanical strength, higher water resistance, higher thermal stability, good chemical resistance to both alkaline and acidic media, easy preparation, tunable pore structure and low ash content[17,18]. Thus, ACS is considered to be one of the most potential as adsorbent for the fabrication of chemical protective clothing.

In the present work, ACS was utilised for the fabrication of chemical protective suit as a NBC coverall for the adequate protection against CW agent. The performance evaluation of developed NBC suit is comprehensively carried out and tested against live CWA sulfur mustard (HD). In order to mimic operational field conditions, different chemical tests based on different working principle were carried out on swatch samples of NBC suit permeable Mk V by challenging against HD. As per literature search, this is the first comprehensive report on chemical protection studies of NBC suit permeable Mk V against HD. This NBC suit permeable Mk V is based on state-of-the-art technology of indigenous developed ACS sandwiched between the fabric layers. The list of some internationally available chemical protective clothing is as given in Table 1.

2. MATERIALS AND METHODS

2.1 Chemicals and Instruments

Chemicals were obtained from Sigma-Aldrich (India) (unless otherwise stated). Solvents were analytical grade for synthesis**.** XAD-2 (Sigma) resin was washed prior to its use in preparation of adsorption tubes in order to facilitate the adsorption of CWA. Three colour detector paper as purchased from M/s Raksha Polycoats Pvt. Ltd, India. HD was synthesised in our establishment as per international norms with 99 per cent purity.

Permeation chamber for placing suit fabric, rotameter, adsorbent tube, vacuum pump (Pall, USA) and gas chromatograph (Agilent, USA) equipped with FPD, micro pipette (Eppendorf, Germany), Mirror, Glass tube (41 mm interior diameter), three colour detector paper (TCD), Stainless steel 316 made cylinders of weight 70 g and 234 g were are used in this study. Agilent @ 6890 GC Coupled with an Agilent 5975 MS was used for detection of HD permeation after exposure. Syringe drive vapour generation system, trap sorbent [Supelco DRBO 608 Amberlite XAD-2 (1500/75 mg)] tubes with plugs, Drager To 7000 Deluxe Tube Opener, Tygon tubing and PTFE tubing for gas flow lines were also used in chemical protection testing. Surface area of ACS was determined by using Surface area analyser (Quantachrome, USA). The CHNS analysis of raw material and ACS were carried out on Vario Micro Cube Elemental Analyzer. Crushing strength of ACS was measured using strength tester (M/s Hemtech, India) and expressed in kg/sphere.

2.2 Methods

2.2.1 *Preparation and Characterisation of Activated Carbon Spheres*

The activated carbon sphere was prepared as per our earlier communication[17]. In brief, sun dried sulfonated polystyrene divinylbenzene (Ion exchange, India) resin was heated in the fluidised bed reactor (FBR), up to 200-250 ºC under air to release the inherent moisture. Around 250 ºC, the cross linking of the polymer started breaking and results in exothermic reaction. This can be observed by the drastic increase in temperature. At this condition the purging of air was stopped and the fluidisation was carried out under nitrogen atmosphere. If the temperature falls below the set point (200 ºC), a mixture of nitrogen and air was used. The stabilisation was done for 30 min at the temperature 250-300 ºC. The furnace was further slowly heated up to 800-850 ºC under nitrogen environment. This process is called carbonisation, where the material releases lot of volatile matters. At 800-850 ºC, steam was supplied for certain duration till we achieved the desired iodine no and crushing strength. Prepared ACS was characterised for iodine number, surface area, bulk density, crushing strength, particle size, CHNS% according to the reported procedures[17].

2.2.2 *Preparation of ACS Adhered Laminated Fabric, Fabrication of Suit and their Characterisation*

For preparation of ACS laminated fabric, first adhesive was applied on the surface of knitted cotton fabric. The ACS was scattered with the help of a powder scattering machine over the base knitted fabric, which has a layer of hot melt adhesive (polyurethane based) over it. After getting scattered, the wound roller is getting cured for a period of about 24 h. The base knitted fabric with ACS was further sandwiched with non-woven material (PP blend) using hot melt adhesive.

Table 1. Characteristics of some NBC protective clothing fabricated based on ACS or activated carbon fabric (ACF) as adsorbent materials along with their manufacturers for NBC protection

Product name	Fabric assembly / Suit mass	Water & oil repellency/ Flame retardency	Water vapour resistance	Chemical protection	Wash cycle (6 times)	Shelf life / Storage time (Year)	Manufacturer/ developer
		Specified Requirement: Yes	Specified Requirement: < 9.6 m^2Pa/W)	Specified Requirement: HD-BTT: >24 h			
SARATOGA® - JSLIST Chemical warfare Protection Overgarment (ACS based)	NA*	NA*	NA*	24 h against HD liquid	6	15	Blucher, Germany
Air permeable NBC Protection Suit (AC beats/ foam based)	600 g/m^2	NA*	10^3 m^2m Bar/W	NA*	NA*	NA*	Shalon, Israel
NBC Combat suit based on woven ACF (Karcher Safe Guard 3002A1) (ACF based)	460 g/m^2	Yes	6.5 m^2Pa/W	24 h against HD liquid and 6 h against HD vapor	10	10	Karcher, Germany
NBC Combat suit based on non-woven ACF (Karcher Safe Guard 2002HP) (ACF based)	520 g/m^2	Yes	7.5 m^2Pa/W	24 h against HD liquid and 6 h against HD vapor	5	10	Karcher, Germany
New Generation CBRN combat suit based on knitted ACF(ACF Based)	420±20 g/m^2	Yes	6 m^2Pa/W	24 h against HD liquid	NA*	10	Paul Boye, France
CBRN over suit activated carbon liner based [RAPTOR 2(&2+)](ACF based)	1.8 kg	Yes	NA*	24 h against HD liquid	20	15	CQC, UK
CBRN over suit activated carbon liner based [TARGA 2(&2+)](ACF based)	1.6 kg	Yes	NA*	24 h against HD liquid	20	15	CQC, UK
TCT-NBC garment (ACF based)	83-230 g/m^2	Yes	NA*	24 h against HD liquid and 6 h against HD vapor	6	NA*	Taiwan Carbon Technology (TCT), Taiwan
Present developed chemical protective suit	< 2.75 kg	Yes	<9.6 m^2Pa/W	24 h against HD liquid and 6 h against HD vapor	6	10	DRDO, India

*Not available in manufacturer's website

After that, the composite layer of base knitted fabric with non-woven material encompassing activated carbon sphere material is getting cured for 24 h. The cured laminated fabric was then evaluated towards mass, bursting strength and air permeability. For manufacturing of chemical protective suits, laminated fabric was stitched with outer fabric to result complete individual protective suit. Preparation of laminated fabric & fabrication of suit were carried out in collaboration of M/s Shiva Texyarn, Coimbatore.

2.3 Chemical Protection Studies

All the tests as given below which was used for evaluation of swatch sample taken from NBC suit have been carried out as per US Military standard[19,20]. Air permeability and water vapour resistance were determined using ASTM D737 and ISO 11092 respectively. The detailed methodologies of following tests are given in these standard; however we have described the methodology in brief.

2.3.1 *Mandrel Test*

A test swatch was taken from the protective suit while maintaining the orientation of suit structure. The swatch sample was kept above the detector paper (TCD paper) on glass mandrel. The stress was created by hanging 234 g weight to each end of the test swatch. A 4 µl (5 mg) of HD droplet was dispensed on test swatch in six different places. Visual examination was carried out periodically to check the penetration of HD. In case no penetration was observed after 1 h the test swatch is declared as qualified sample[19].

2.3.2 *Expulsion Test*

A square (5.1 x 5.1 cm) test swatch was kept above the detector paper (TCD paper) on glass plate. A 4 µl (5 mg) of HD was challenged in the centre of test swatch in such a manner that outer fabric was faced in upward direction. The detector paper was observed post 15 s of agent challenge. In case of no sign of agent penetration, a 454 g weight was applied which creates a pressure of 70.2 g/cm^2 on the swatch. If no penetration was observed after 1 h the test swatch is declared as qualified sample.

2.3.3 Inverted Expulsion

Test specimen was prepared using a square (5.1 x 5.1 cm) test swatch placed above detector paper (TCD). First about 4 µl (5 mg) of HD was placed on a glass plate and then test specimen was placed on the HD drop by keeping outer fabric in contact with HD and detector paper faced upward. Similar to expulsion test a pressure of 70.2 g/cm^2 was applied on test sample. If no penetration was observed after 1 h the test swatch is declared as qualified sample.

2.3.4 HD-liquid Challenge

The testing was carried out with swatch samples from suit. A 50 µm polyethylene film was placed in the permeation chamber containing test specimen in such a way that the outer fabric always faces upward. A dual flow configuration was used to perform the HD- liquid challenge. A stream of conditioned air (80±5 % RH) was drawn across the top of a swatch challenged with droplets of agent. A clean airflow was drawn across the underside of the swatch for sampling. This method causes the agent challenge to evaporate over time. The top surface air flow rate has been maintained at 0.25 L/min using rotameters, and the underside airflow rate at 0.30 L/min. Challenge concentration of HD on fabric was 10 g/m^2 for 24 h. The HD was uniformally distributed on swatch sample using calibrated microsyringe in such a way that HD droplets of size approx 1 µl with 10 g/m^2 contamination density. The chambers were closed and adsorbent tubes filled with XAD-2 was placed in lower cups and empty adsorbent tubes at upper cups. RH was maintained at 80±5 % through water bubbler. The amount of penetrated agent was collected in an adsorbent filled tubes. At the end of the test, the amount of agent in the adsorbent filled tubes was determined by extracting the adsorbent material with 1 ml of ethyl acetate through vortexing (vortexing at least for 5 min). The extracted samples were analysed by GC-MS with respect to standard calibration plot.

2.3.5 HD-vapour Challenge

In vapour challenge test, a conditioned swatch sample of 3.56 cm diameter is placed in a closed test cell assembly. The required concentration of HD vapours *i.e* 20 mg/m^3 was generated by syringe drive vapour generated system. The temperature and RH were kept at 30 ºC and 80±5%. Swatch was continuously exposed upto 6 h at given concentration. The air speed was 1.6 cm/s to the perpendicular of fabric surface. The amount of penetrated agent is collected through XAD-2 sorbent tube by using air sampling pump. At the end of the test multiple point internal standard method was used to quantitate and validate 20 mg/m^3 concentration of HD in XAD-2. The dose (mg.min/m^3) as reported is calculated by dividing the penetrated amount of agent (after test duration) by the flow in ml/min.

2.3.6 HD Breakthrough Time (HD-BTT) Measurements

HD-BTT method used for evaluation of swatches are described elsewhere[21]. In brief, a specially designed 50 mm brass assembly, the coated fabric (ACS sides facing each other) are exposed to an atmosphere saturated with HD vapours at 20 ºC. A detector paper prepared using a congo red-impregnated filter paper spotted with SD reagent (2,4-dichlorophenylbenzoylchloroimide) was kept on top of the samples to follow the BTT of HD. First appearance of blue colour on the detector paper indicated BTT, the protection time against HD.

2.3.7 Analysis of Liquid and Vapour Penetration Using GC-MS

The post analysis of CWA permeation was carried out with method described elsewhere[22] with little modification. For this purpose GC–MS (7890A GC and 5975 quadrupole MS; Agilent Technology, USA) was used with DB5-MS capillary column (J&W Scientific, Folson, CA). The flow rate of helium was maintained at 1.0 ml/min and the sample was in split less mode. The temperature of injection port was 250 ºC where as for transfer line temperature was 280 ºC. The solvent delay of mass spectrometric was initiated 3 min post sample application. Selected ion mode was used as the ionisation for the quantitative analysis. The temperature was maintained at 230 ºC for ion source and 150 ºC quadrupole. The m/z range 50-500 was used for data acquisition purpose.

3. RESULT AND DISCUSSIONS

3.1 Preparation and Characterisation of Activated Carbon Spheres

ACS was produced in a pilot scale fluidised bed reactor using polystyrene based resin as raw material. The prepared ACS has BET surface area of 877 m^2/g after 7 h of activation and having 2.3 kg/sphere of crushing strength. The activation temperature varied between 800-850 ºC and steam flow was rate of 5-7 kg/h. The carbon content was increased from ~ 40 % as in precursor to more than 90 % in final product. The produced ACS was 0.3 mm to 0.8 mm in size range. This ACS was used for preparation of inner laminated fabric.The characteristics of ACS used for the chemical protective suit development is as given in Table 2.

3.2 Preparation of ACS Adhered Laminated Fabric, Fabrication of Suit and Their Characterisation

The picture of outer fabric, base fabric, non-woven and ACS are as given in Fig. 1. Figure 1(a) shows the camouflage multifunctional outer fabric which is flame retardant, oil and water repellent used for fabrication of NBC suit. Figure 1(b) shows the image of knitted fabric used as carrier to adhere the ACS, and SEM image (Fig. 1(c)) shows the morphology of prepared ACS depicted as spherical geometry. Non-woven fabric (Fig. 1(d)) is used as base fabric to sandwitch the ACS and also provides the diffusivity of CWA. ACS adhered

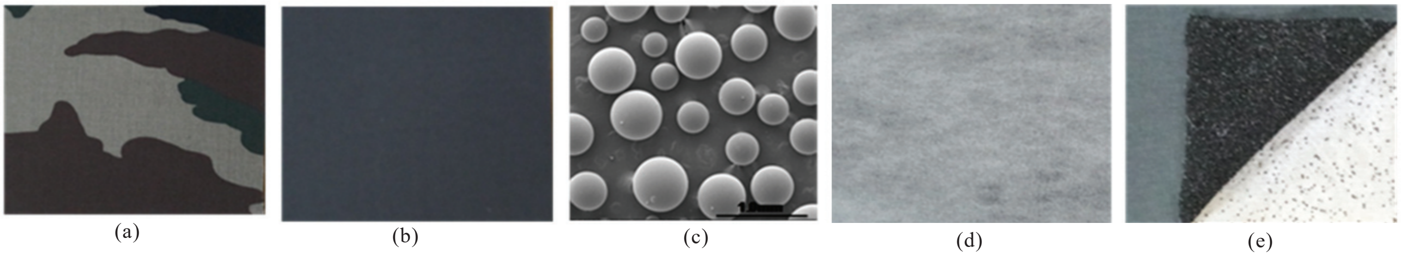

Figure 1. Pictures of component used for fabrication of chemical protective suit: (a) Outer fabric, (b) Base fabric used for ACS coating, (c) Activated carbon sphere, (d) Non-woven fabric, and (e) ACS adhered laminated fabric.

Table 2. Characteristics of Activated carbon spheres used for development of inner laminated fabric

Parameters	Value
BET (Brunauer-Emmett-Teller) surface area Specified requirements: ≥800 m^2/g	877 m^2/g
Iodine Number Specified requirements: ≥1,000 mg/g	1029 mg/g
Crushing strength Specified requirements: ≥1.5 kg/sphere	2.3 kg/sphere
Carbon (%) Specified requirements: >90%	91%
Particle size Specified requirements: (0.3-0.8 mm)	0.3-0.8 mm

laminated fabric is as represented in Fig. 1(e) showing the uniform distribution of ACS over the carrier fabric.

Coating of ACS over the fabric is one of the critical parameter to influence the chemical protection, comfort and low thermal burden for chemical protective suit. Coating and lamination enhance and extend the range of functional performance properties of technical textiles and the use of these techniques is growing rapidly as the applications for technical textiles in particular defence sector. The characteristics of base fabric (knitted cotton fabric), non-woven fabric and laminated fabric is as given in Table 3.

Table 3. Characteristics of inner ACS laminated fabric used for development of chemical protective clothing

Characteristics parameters	Value
Base fabric (Knitted) Max 120 g/m^2	Cotton with 111.7 g/m^2
Non-woven fabric 35 - 50 g/m^2	Polypropylene blend with 42 g/m^2
Mass of laminated fabric (consisting of cotton base fabric+ACS+Non-woven fabric with adhesive) 616 g/m^2	343 g/m^2
Bursting strength of laminated fabric Min. 500 kPa	717.1 kPa
Air permeability of laminated fabric Min. 30 $cm^3/s/cm^2$	57.93 $cm^3/cm^2/s$

The key challenge of adhesive selection is to keep minimum amount of adhesive so that better binding of ACS resulted along with air permeability. The air permeability of the laminated fabric was determined as per standard and given in Table 3. Air permeability of the laminated fabric is a parameter of critical importance and play a key role to decide the heat stress of the protective clothing for the wearer. Good transmission of air across the fabric, offers active sweat managements and thus higher wear comfort. However, increased air permeability will directly reduce the chemical protective performance of the suit[23]. In order to achieve a good compromise between comfort and protection it is useful to understand the relationship between the chemical protective performance and air permeability. For this purpose, various combinations of ACS loading on fabric were evaluated and optimised to achieve air permeability greater than 30 $cm^3/s/cm^2$ and lighter weight < 2.75 Kg (XL). It was also compared with the commercially available NBC suits and found to be better in terms of air permeability and cost effective. It can provide continuous protection to the personnel for a period of more than 24 h with chemical agents in liquid and vapour forms. The physical characteristics of chemical protective suit is as given in Table 4. The image of indigenously developed NBC suit Mk V is as represented in Fig. 2.

Table 4. Characteristics of chemical protective suit fabricated using camouflage pattern outer fabric and inner laminated fabric

Characteristics parameters	Value
Outer fabric Min. 100 g/m^2	Aramid fabric with 115.18 g/m^2
Suit weight for S, M, L and XL size <2.75 kg	2.28, 2.34, 2.44 and 2.60 kg
Tear strength of outer fabric (Min.) Warp 3.0 kgf Weft 3.0 kgf	Warp 4.1 kgf Weft 3.67 kgf
Water repellency (Rating) of outer fabric Min. 90	90
Oil repellenacy (Rating) of outer fabric Min. 6	7
Flame retardancy of outer fabric Yes	Yes
Air permeability Min. 30.0 $cm^3/cm^2/s$	31.6 $cm^3/cm^2/s$
Water vapour resistance Not to exceed 9.6 m^2Pa/W	9.38 m^2Pa/W
Anti static < $5.3x10^{12}$ Ohm	7.27 x 10^{10} Ohm

Figure 2. Chemical protective suit.

3.3 Chemical Protection Studies

Chemical tests qualitative and quantitative were conducted for the evaluation of suit using swatch. Chemical protective performance tests include HD-BTT, mandrel, expulsion and inverted expulsion as qualitative tests and liquid and vapour challenge as quantitative tests for performance assessment. The results of chemical protection studies are as given in Table 5. Expulsion (under pressure) method simulates a situation in which swatch sample of chemical protective suit is subjected under pressure against HD contaminated surface. Mandrel test (under stress) simulates a situation in which swatch sample of chemical protective suit is subjected under stress against HD contaminated surface. The above tests are qualitative in nature and depict the suitability of developed chemical protective suit in real combat field situation. As shown in Table 5, the developed chemical protective suit meeting all the specified parameters of Indian Army. After qualitative tests, quantitative tests were also conducted against HD liquid and vapour form to simulate the real field conditions to measure the permeation of HD through suit swatches over a 24 h and 6 h, respectively. These tests are very critical and performed worldwide to predict the protective potential of developed NBC suit. These tests are of paramount importance to assess how well the suit materials perform against HD -liquid and vapour permeation. Swatches were taken from the chest area, thigh area and crotch upper arm seam. The results of liquid and vapour challenge as shown in Table 5 revealed that the developed suit provided protection against liquid and vapour form of HD. The experimental results revealed that greater than 180 g/m^2 ACS loading on fabric meets the requirement of specified protection against HD along with physical and chemical parameters as well as total weight of suit is < 2.75 kg for XL size (Tables 4 and 5).

Table 5. Chemical protection studies on suit against HD (CWA)

Test parameters	Results/Value
Mandrel test Fabric under stress No penetration in 1 h	No penetration in 1 h
Expulsion test Fabric under pressure No penetration in 1 h	No penetration in 1 h
Inverted expulsion test Fabric under pressure No penetration in 1 h	No penetration in 1 h
Liquid chemical agent challenge test Maximum allowed penetration 4 µg/cm^2	0.4 µg/cm^2
Vapour chemical agent challenge test Maximum allowed penetration 500 mg. min/m^3	411 mg.min/m^3

4. CONCLUSIONS

In the present study, ACS of high mechanical strength was utilised for the fabrication of chemical protective suit as a NBC coverall for the adequate protection against chemical threat. The sphere being hydrophobic does not get deteriorated by laundering. This makes the suit reusable and more affordable. Carbon loadings were evaluated with respect to desired physical and chemical parameters. A series of chemical tests mimicking the on-site field conditions were performed such as under stress, under pressure, liquid and vapour form for evaluation of this suit. The test results revealed that the fabricated suit can efficiently protect the wearer from chemical threats. It also has high air permeability and can provide protection in contaminated environment for more than 24 h against specified concentration. On the basis of performance evaluation of NBC Mk V suit it is evident that the developed suit meets the international standard. Furthermore, a possible integration of advanced material onto textile filter fabric can be done to achieve self detoxifying properties of the NBC suit. Such practical application would further enhance the horizon of NBC protective suits.

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