

Activated Carbon Fabric: An Adsorbent Material for Chemical Protective Clothing

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ABSTRACT

Activated carbon fabric or fiber (ACF) is a novel carbonaceous material with exceptionally high adsorption rate and larger adsorption capacity, that has emerged as a rising star in the field of adsorbents. ACF has many advantages over other commercial porous storage materials such as granular activated carbon and powdered activated carbon in terms of adsorption capacity, well defined microporous structure, stability, flexibility and ease of lamination to various substrates. In the last few years, activated carbon fabrics have gained greater choice of interest for use as an adsorbent material in several fields including nuclear, biological and chemical (NBC) protection suit. Viscose rayon, acetate, polyacrylonitrile, pitch, and phenolic based materials are mainly used as precursors for preparation of ACF. ACF or fibres are generally prepared by process comprising stabilisation, carbonisation and activation of precursors. Reviews recent advances and developments in the field of ACF and their utility as an adsorbent material in various fields including NBC scenario. ACF with unmatched pore structure and surface characteristics at present, with continued innovations and attention to its key challenges, it is expected that ACF will play a pivotal role in diverse environmental, defence, and civil applications.

Keywords: Activated carbon fabric; Adsorption; Viscose rayon precursor; Chemical protection; Carbonisation; Activation

1. INTRODUCTION

Carbon is one of the magnificent elements, which leads to a large variety of compounds and structures with a very wide array of properties which have revolutionised the field of adsorbents. Among the different carbon based adsorbents, activated carbon (AC) is non hazardous, carbonaceous product, having a porous structure, large surface area and widely used for the removal of volatile organic compounds and contaminants from the liquid and gaseous streams. Powder, granular, spherical and fiber or fabric forms are the different variant of the AC. ACs are used in various fields such as removal of heavy metals and toxic gases, biomedical application, catalysis, natural and biogas storage¹⁻⁴. Recently, activated carbon fibers (ACFs) or fabrics have gained increased attention of scientific community over the traditional adsorbents owing to their excellent adsorption potential. Common type of activated carbon has a ladder like pore structure; however, in ACFs, micropores are directly available on surface of fabrics for adsorbate gas. In granulated AC, adsorbate gases first enter to macropores followed by mesopores and micropores⁵. Activated carbon adsorbents have broad pores sizes and shapes, low cost and unique properties as compared to zeolites which make ACs versatile adsorbents. Specific surface area about 2000-2500 m²/g and micropore volume of 1.6 ml/g have been achieved for ACFs using different precursors^{2,6}. Activated carbon impregnated with metals like aluminium, manganese, zinc, iron, lithium,

calcium, palladium, silver have been used for removal of toxic chemicals^{2,7-9}.

Preparation of ACF is challenging because well developed pores with better tensile and compressive strength is required to prevent damage and dust formation⁶. Polyacrylonitrile (PAN), pitch, cellulosic materials such as viscose rayon and acetate, saran and phenolic resin are utilised as precursors for commercial production of ACFs. The different steps for preparation of ACFs from precursors involve spinning, stabilisation (200 °C–400 °C in air), carbonisation, and physical or chemical activation¹⁰⁻¹². After stabilisation, fibers or fabrics are subjected to heat treatment (carbonisation) at about 800 °C to 1,000 °C temperature in an inert atmosphere. To prepare high strength carbon fibers, graphitisation of carbonised fibers have also been carried out at about 3,000 °C to yield high carbon (%) and Young's modulus¹³⁻¹⁴. Supercritical water and carbon dioxide were also studied for preparation of ACF¹⁵. ACFs also have potential in various novel applications such as cell therapy, in medicines as enteroadsorbents and support in growth of stem cell¹⁶. The ACFs are used for adsorption of liquid pollutants and toxic gases, in electrical and electrochemical applications¹⁷⁻¹⁸, microstructured reactors¹⁹, substrate for nanotube growth²⁰, separation and purification of biomolecules²¹ and adsorption of chemical warfare agent. Due to direct linking of micropores with external surface area in ACFs, it provides minimum resistance to mass transfer and also minimises pressure drops²²⁻²³. Commercially available ACFs with their characteristics is given in Table 1.

Individual protective clothings or protective suits against

chemical warfare (CW) agents may play a very important role in the chemical defence during war scenarios. Chemical protective clothing based on various adsorbent materials is available globally²⁴. These adsorbent materials are activated carbon powder, activated carbon spheres²⁵ or activated carbon fabrics. For manufacturing of chemical protective suits, first these adsorbent materials are adhered on base fabrics using a polymeric adhesive followed by another layer of nonwoven fabrics using adhesive or hot melt techniques. Further, fabrications of chemical protective suits are carried out to manufacture complete individual protective suits. In powdered activated carbon or charcoal based chemical protective suits, there is a launderability issues. Thus, the reuse of this suit is not favourable. The activated carbon spheres based chemical protective suits can be used even after multiple laundries, but having a certain limits. ACF based chemical protective suits are light in weight, comfortable and also sustain multiple laundries as shown in Table 2 and address the limits of ACS based suits. Provide an overview of ACF and its application in chemical protective clothing.

2. PRODUCTION OF ACTIVATED CARBON FABRIC

ACF are prepared by pyrolysis to obtain enhanced specific surface area and micro pore volume. Before starting heat treatment process, it is also ensured that suitable precursors or raw materials are selected to achieve desired quality of ACF. Heat treatment conditions are dependent on type of precursors such as PAN, pitch and rayon. Stabilisation, carbonisation and activation processes are optimised to achieve desired quality of ACF.

2.1 Precursors used for ACF Preparation

ACFs are produced from precursor fabrics or fibers of viscose rayon²⁶⁻²⁸, phenolic resin²⁹, polyacrylonitrile (PAN)³⁰⁻³¹ and pitch³²⁻³³. For the selection of suitable precursors, several factors such as cost of precursor with high carbon and low ash

content, and higher density with volatile matter are considered. During carbonisation, pores are created due to volatile material evaporation and better structural strength resulted because of the high density material.

PAN is prepared using solution or suspension polymerisation process from acrylonitrile. For this purpose, initially PAN solution is used for fiber spinning dope and then PAN with linear polymers and copolymer for spinning with molecular weights of 70,000 to 260,000 is made¹³. The PAN based precursor provides good tensile strength and more resistant to compressive failure than other precursor fabric.

Pitch is produced by distillation process and mesophase pitch is prepared by polymerisation and condensation of raw pitch which change pitch from an originally isotropic to an anisotropic substance^{4,34}. Highly pure and low cost synthetic pitch results into fast stabilisation. Pitch as compared to PAN gave high degree of orientation and enhanced yield. The manufacturing cost of carbon fibers from pitch based materials is high³⁵.

The precursors such as cellulosic fibers have also been studied for preparation of ACFs³⁵. For this purpose, viscose rayon prepared from the wood pulp is utilised. Solution spinning has been used to prepare viscose rayon from wood pulp. The production of carbon fibers from rayon has low carbon yield (20–30%)^{13,36}. The research has mostly been focused to increase the carbon yield from rayon. For preparation of carbon fabrics or fibers from rayon includes thermal stabilisation, and carbonisation. For rayon fibers without pre-treatment, the thermal decomposition takes more time. The use of flame retardant chemicals enhanced the dehydration that ultimately decreases process time. Flame retardants are used to impregnate the precursor fibers or fabrics in their aqueous dispersions^{13,37}.

Phosphoric acid as an impregnating reagent and its influence on specific surface area, weight yield, pore volume, morphology and chemical composition of ACF was studied. This study established that the both BET surface area and

Table 1. Characteristics of a few commercially available ACF used for development of NBC protective clothing

Product name with precursor	Mass/surface density	Thickness	BET surface area	CTC activity (%)	Air permeability at 10 mm w.g.	Applications	Manufacturer
FM30K (Knitted) from viscose rayon	110 g/m ²	0.4 mm	NA*	55-70	75 cm ³ /cm ² /s	Combat suits, first responder suits, escape masks, undergarments, gloves, overgarments, socks, CBRN filtration media, CBRN decontamination wipes, low weight reduced resistance respirator canisters, Antimicrobial wound dressing, missile decoy media	Chemviron Carbon, UK
FM50K (Knitted) from viscose rayon	130 g/m ²	0.5 mm	NA*	55-70	75 cm ³ /cm ² /s		
FM10W (woven) from viscose rayon	120 g/m ²	0.5 mm	NA*	55	100 cm ³ /cm ² /s		
AW1107 PAN based	150 g/m ²	0.6 mm	1100 m ² /g	NA*	NA*	Gas mask, anti-smoke hood, NBC garment, hunting suit	Taiwan Carbon Technology, Taiwan
AW1109 PAN based	115 g/m ²	0.4 mm	1100 m ² /g	NA*	NA*		

*Not available in manufacturer's website

Table 2. Characteristics of some NBC protective clothing fabricated using ACF along with their manufacturers for NBC protection

Product name	Fabric assembly / suit mass	Water & oil repellency/ flame retardancy	Water vapor resistance (m ² Pa/W)	Chemical protection	Wash cycle (Times)	Shelf life (Years)	Manufacturer/ developer
NBC Combat suit based on woven ACF (Karcher Safe Guard 3002A1)	460 g/m ²	Yes	6.5	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)	520 g/m ²	Yes	7.5	24 h against HD liquid and 6 h against HD vapor	5	10	Karcher, Germany
New Generation CBRN combat suit based on knitted ACF	420±20 g/m ²	Yes	6	24 h against HD liquid	NA*	10	Paul Boye, France
CBRN over suit activated carbon liner based [RAPTOR 2(&2+)]	1.8 kg	Yes	NA*	24 h against HD liquid	20	15	CQC, UK
CBRN over suit activated carbon liner based [TARGA 2(&2+)]	1.6 kg	Yes	NA*	24 h against HD liquid	20	15	CQC, UK
TCT-NBC garment	83-230 g/m ²	Yes	NA*	24 h against HD liquid and 6 h against HD vapor	6	NA*	Taiwan Carbon Technology, Taiwan

*Not available in manufacturer's website

micropore volume enhanced with increasing phosphoric acid. However, the weight yield and microporosity (%) decreased with increasing impregnating reagent²⁸. Influence of heat treatment temperature and activating agents on the quality of ACFs derived from viscose rayon knitted fabrics have also been established that the use of steam and air at higher temperature enhanced specific surface area and microporosity²⁶. Similar findings were also found at higher temperature using carbon dioxide as activating agent²⁷. Typical flowchart for production of ACF from viscose rayon, PAN or pitch precursors is shown in Fig. 1.

Biorenewable lignin phenolic compounds and poly(ethylene oxide) (PEO) as precursors were also studied for preparation of carbon fibers³⁸. Other natural fibers such as silk, chitosan and eucalyptus tar pitch have also been investigated³⁹⁻⁴¹. However, most of them resulted in ACFs with lower strength.

2.2 Preparation of ACF

ACFs are manufactured by the pyrolysis of variety of raw materials (precursors) such as polymeric, cellulosic, mesophase pitch, PAN and phenolic-based materials as described above followed by physical or chemical activation of chars⁴². The heat treatment process used for preparation of ACF from these precursors mainly involves stabilisation, carbonisation, and activation. Controlled heating is necessary during the stabilisation because this process is exothermic process. In case of PAN precursor, linear structure changed into cyclic structure. Stabilisation with air resulted enhanced carbonisation yield and tensile strength.

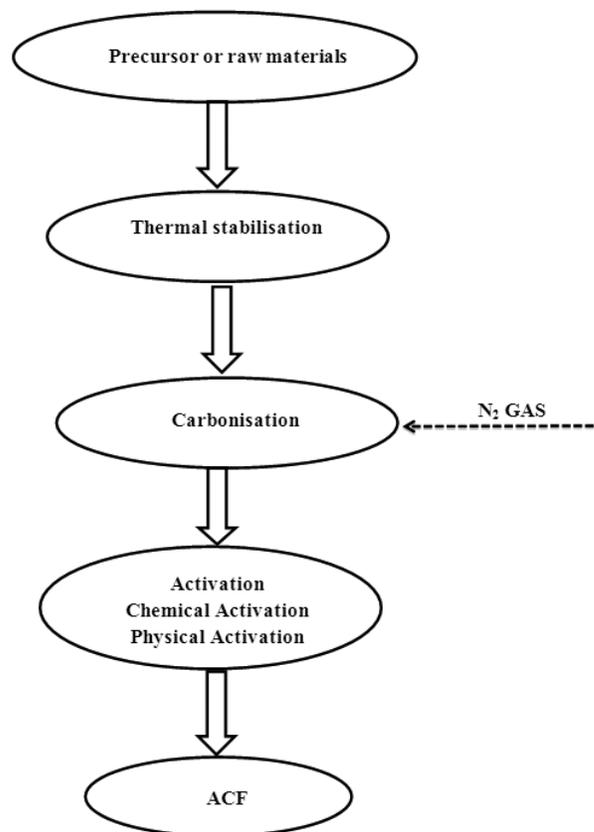


Figure 1. Process flow diagram for preparation of ACF.

2.2.1 Thermo-oxidative Stabilisation

In this process, precursor fibers or fabric are stabilised in the presence of air or nitrogen (inert environment) or both at more than 150 °C. This stabilisation process resulted in conversion of linear polymer to infusible ladder polymer. Slight tension for fibers or fabric is also applied some times which depends on morphology, diameter and composition of fabric^{13,43}.

2.2.2 Carbonisation

Stabilised precursor fibers or fabric are converted to carbon fiber or fabric through carbonisation. Carbonisation process is done in presence of inert gases such as nitrogen or argon in order to avoid the oxidation at higher temperature. Carbonisation involves thermal decomposition of precursor fibers, eliminating non-carbon species yielding a fixed carbon mass and rudimentary pore structure⁴⁴. At about 500 °C, the basic microstructure of carbon fibers is produced and carbonisation process at 1000 °C and above resulted in hard structure of carbon fibers. The carbonisation step is very crucial which determines the properties of the final product

2.2.3 Activation

Carbon fibers produced after carbonisation process is found to have low adsorption potential due to filled or partially blocked free interstices present in the carbon. Activation of carbonised fabrics or fibers is done to increase the surface area, to create the new pores and clear the pores filled with tarry materials. These pores were created during the carbonisation process. Activation process resulted in formation of some new pores thus formation of well developed and readily accessible clear pore structure established. These pores have high internal surface area. The process of activation for carbonised fibers or fabric is done by two ways namely chemical and physical activation. In physical activation, the activating gases generally used are carbon dioxide, steam or their combination. The carbonised products developed porous structure of molecular dimensions and extended surface area after heat treatment. In chemical activation, different chemical reagents are used.

The process optimisation parameters which should be considered during physical activation steps are stabilisation and carbonisation conditions (heating rate and temperatures), activation agents, rate of gas flow, activation temperatures (750 °C - 850 °C for viscose rayon) and burn-off degree. High ACFs yield with breaking load and high adsorption capacity were established at low heating rate for carbonisation during physical activation^{2,6}. During activation of carbon fibers or fabric with CO₂, specific surface area, adsorption potential and microporosity have been increased at low temperature. The tensile or breaking strength of ACFs was also influenced by the starting carbonisation environments and during activation, it decreases with the extent of activation time⁴⁵⁻⁴⁷.

In chemical activation, the influence of chemicals and their concentration during impregnation of fabrics were studied in terms of breaking strength, product yield and pore structures⁴⁸. The chemical treatment of precursor has been reported to be done by various methods including chemically modifying the precursor by padding, adding in the spinning

dope and coating on the precursor. Chemical modification and padding are more popular due to ease of application and resulting properties. Aqueous solutions of AlCl₃, ZnCl₂, NH₄Cl, FeCl₃, CuCl₂, H₃PO₄, and Na₂HPO₄ (1–12 %, w/v) have been used for preparation of ACFs by chemical activation. The highest product yields (36 %) were achieved using 5% of AlCl₃ and H₃PO₄. It has been established that the aluminium chloride (5 %) as impregnating reagent resulted in improvements on tensile strength of ACFs^{45,48}. The process parameters which can influence the ACFs quality in chemical activations are heating temperature, ratio of precursor and activating agent, and contact time with impregnating agent. The chemical treatments prior to carbonisation and activation are considered to be important in the process of making ACF. This is because of one or combination of effects such as increasing the adsorption capacity, reduction in activation temperature and improvement in final properties like tensile strength. Production of ACF cloths using rayon precursor (viscose) were well studied in detail including carbonisation, activation (physical), effect of CO₂ activation and chemical activation^{6,46-49}. Recently, microwave radiation assisted activation has also been used to produce activated carbon fibers with 1370 m² surface area in presence of H₃PO₄. The microwave power used for this purpose was 640 W⁵⁰.

3. CHARACTERISTICS OF ACF

The pores in activated carbons are distributed as macropores (> 50 nm), mesopores (2–50 nm) and micropores (< 2 nm). Gas adsorption isotherms, mercury porosimetry and scanning tunneling microscopy have been applied to get distribution of pore size in activated carbons². Turbostratic, graphitic or a hybrid structure layer planes are formed in carbon fabric. The turbostratic model however is able to explain lower tensile and higher sorption properties of this materials¹. Adsorption is a process used for removal of solutes (from gaseous or liquid mixtures) on solid surface. It is carried out using two ways either physical adsorption or chemical adsorption. Van-der-Waals forces are the driving forces for physical adsorption whereas chemical bonding is responsible for chemical adsorption. ACFs have high adsorption capacity due to high surface area (>2000 m²/g) and high pore volume (0.8 cm³/g to 1.2 cm³/g)⁶. BET surface area and pore volumes (micro, meso, and macro) are calculated using adsorption isotherms⁵¹⁻⁵². ACFs are also effective in protection from the attack of toxic chemical agents, making it ideal for Chemical protective clothing and filters. ACFs can be easily converted or fabricated into cloth for variety of applications. Thus, ACF is a choice for chemical protective clothing.

4. ACF AS PROTECTIVE MATERIALS IN CHEMICAL PROTECTIVE CLOTHING

Nuclear biological and chemical (NBC) threat is a major challenge in present day war scenario. To protect defence personnel against NBC threat, personnel protection takes most important role. Many manufactures around the world have developed personnel protective ensembles using ACF. These ensembles include NBC suit, socks, wipes,

tents, hoods, masks, undergarments, canisters, etc. ACF have very high level of comfort because of its lower weight, excellent air permeability, less water vapour resistance and flexibility without compromising chemical protection. Use of impregnated charcoal cloth for the treatment of air polluted with hydrogen cyanide, a well established chemical warfare agents and industrial toxicant was studied and established the utility of ACF⁵³. The breakthrough behaviour of ACF cloth against an oxygen analogue (OA) of sulphur mustard was also studied and suggested that the ACF may be used in foldable masks for protection against chemical warfare agents⁵⁴. Characteristics of some commercially available ACF along with their manufacturers are given in Table 1. For development of chemical protective clothing, ACF adsorbent layer is laminated with both sides or one side using cotton, non-woven or polyester fabric to achieve better strength along with high air permeability and less water vapour resistance. Characteristics of some of the international protective clothing fabricated using ACF is given in Table 2.

5. APPLICATIONS OF ACF

ACFs are used in adsorptive removal of toxicants such as hydrocarbons, hydrogen sulphide, chloride, ethers, acids, formaldehyde, sulphur dioxide, nitrogen and other sulphur compounds⁵⁵⁻⁵⁶. ACFs are also used for adsorptive removal of waste liquids from different industries like textile, dye and metal processing that contain heavy metals which are highly toxic in nature⁵⁷⁻⁵⁸. The adsorptive removal of toxic metals such as cadmium, lead, copper, nickel, chromium and zinc were also carried out using ACFs. Textile dyes, pesticides, surfactants and organic acids are organic pollutants which were studied for adsorptive removal using ACFs⁵⁹. Dyes used in textile industries are stable and degradation of these dyes are difficult⁶⁰. ACFs showed their efficiency in removal of volatile organic compounds and harmful air polluting gases. ACFs also offer better adsorptive potential for removal of H₂S⁶¹. The adsorptive removal of benzene and n-hexane in vapor phase was successfully carried out using ACFs⁶². ACFs are good adsorbent for natural gas and methane storage application^{4,63}. Pitch-based ACF also showed reduction of oxides of sulphur in the presence of H₂O and atmospheric NO to harmless compound⁶⁴⁻⁶⁵. ACF was used as replacement to granular activated carbon for purifying water as it can be easily regenerated. Antibacterial activated carbon fabric with addition of silver was also developed for water treatment and resulted in antibacterial activity too⁴.

6. OUTLOOK, PROSPECTS AND FUTURE CHALLENGES

Reviewed the recent developments in the field of activated carbon fabrics for their applications as an adsorbent material in chemical protective clothing. It summarises the literature on the recent progress in the development of ACF based on different raw materials particularly, cellulosic (viscose rayon), pitch and polyacrylonitrile precursors. Further the key requirements and challenges in this important area have also been discussed. The key parameters which are critical for preparation of ACFs with low cost are breaking load, compressive strength and new

raw material development. A considerable research effort has been devoted to optimise and to make the balance between three crucial steps of ACF preparation namely stabilisation, carbonisation and activation in order to get well dispersed micropores with large surface area, high breaking load and air permeability (for chemical protective clothing). Still, it is needed to develop a balance approach or methodology to make low cost ACF with greater strength, well defined microporous structure and high surface area. The microporous structure of ACFs relies on structure of raw materials and process environments (temperature and heat treatment time). Process optimisation parameter which includes stabilisation, carbonisation, and activation conditions is necessary to achieve low cost ACFs along with selection of cheap raw material source. This optimisation could lead to achieve better pore size distribution and pore structure for many novel applications. The technology associated with the ACF preparation aiming to make the chemical protective suit to first responders is still in their infancy in most of the countries due to its low strength, less air permeability (after lamination with base fabric) and high cost, though the immense market potential is driving development towards that end. There are several aspects which need to be addressed before translating the ACF into real product in form of chemical protective suit as discussed above. Activated carbon fabrics have better adsorptive removal efficiency, large micropore volume along with BET surface area and it can be easily fabricated into clothing with ease of fabrication, enhanced comfort and product life. ACFs also showed fast adsorption as well desorption potential. ACFs are mainly utilised in adsorptive removal of toxicants from gases and liquid, separation of gases from mixture by incorporating impregnating agents, gas storage, chemical catalysis, medical applications like surgical dressings, purifying water, and many more such applications. Still many potential areas are there in which activated carbon fabrics may play important role to make environment free from pollutants. Chemical protective clothing has been developed for personnel protection against chemical warfare agents using ACF. In future wide scope is there to develop other protective clothing and ensembles using ACF as an adsorbent material such as haversack, escape masks, boots, socks, wipes, hoods, tents and fire protective suits for NBC applications.

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