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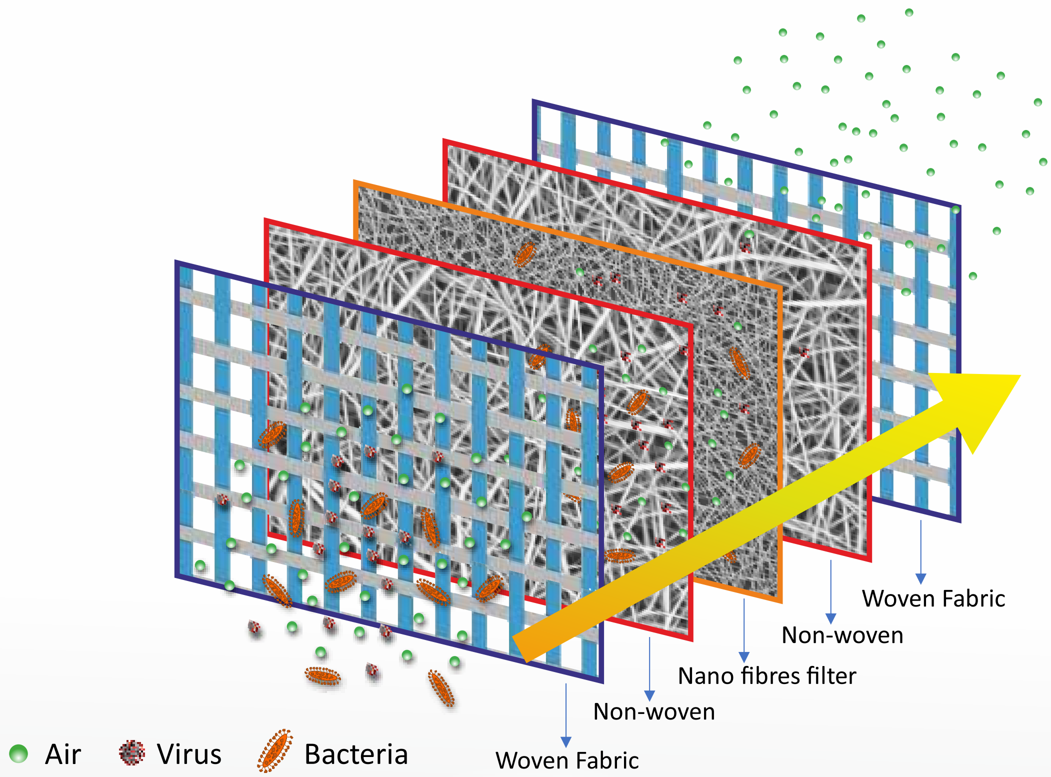

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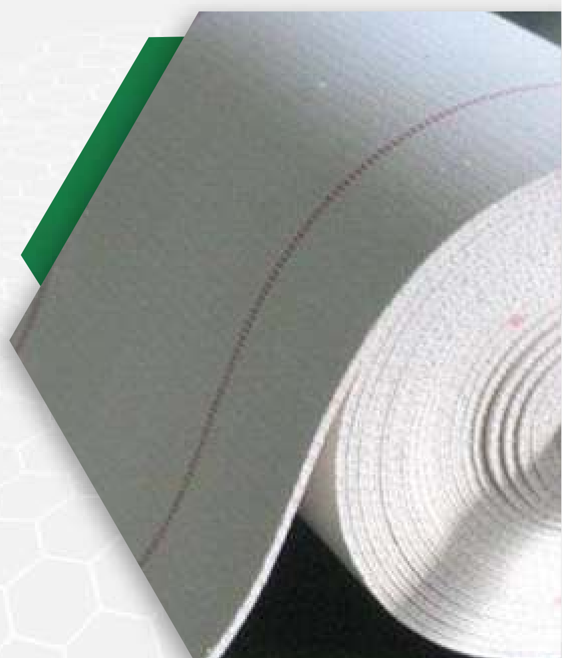

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EDITOR'S DESK

Dear Readers,

Greetings!!

Research with persistent and focused efforts lead to a positive result. Fostering research and providing a platform to publish quality research papers and related articles has been a continuous effort of BTRA Scan. In continuation to this effort, I am delighted to present to our readers the 1st issue of 51 Edition of BTRA SCAN. We are facing now the 3rd wave of the pandemic on the way of our development. We have to take precautions and focus on our progress and development work.

This issue has papers from the different domains such as comfortable reusable protective face mask developed by BTRA, conductive textile, super critical carbon dioxide dyeing of textiles and dispersion of CNT in the PAN. Now we are open for authors from outside so researchers can send their original articles, case studies, research reviews or empirical contributions for publication in our journal.

Inspite of the third wave, we are fully focusing on our work. I feel we will have a great time ahead. Hope, we all are following the safety practices to defeat the pandemic from our life.

Our sincere thanks to all the reader and contributors for their support and interest.

T V Sreekumar, PhD
Director, BTRA

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Installation damage of geosynthetics

The geosynthetics are prone to some amount of damage during their installation. To assess the quantity of the installation damage, a standard method was initially developed by Watts and Brady of the Transport Research Laboratory in the United Kingdom. The procedure has also discussed in the ASTM D 5818 with similar requirements. We are at BTRA doing the test following same ASTM D 5818 method followed by respective tensile strength. For the time being we are using the construction site for the sample preparation. If customer will agree, BTRA will collect the sample from site after standard procedure and provide ethe report.







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Improved Sustainable, Environment Friendly, Green Technology For Textile Dyeing Using Supercritical Fluid

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Abstract

Deven Supercriticals, India (DSPL) has developed innovative Supercritical (SC) Carbon Dioxide (CO_2) based dyeing and finishing technology that is uniquely suitable for not only polyester but also for cotton and blended textiles. Further, it uses conventional dyes (No special dyes required) and recipes as used in the conventional process, to get the desired shade BUT without the use of water in the dyeing process. This innovative process shows improved dye utilization, makes scale-up easy and has less than half dyeing time vis-a-vis prior-art SC CO_2 based dyeing processes. There is no need for reduction clearing for polyester, cotton with no salt added, dyes blends in a single step, reduces overall auxiliary chemicals. Thus substantially reducing the pollution, water and energy load. This has truly made the SC CO_2 technology viable, versatile and simple.

Keywords

Sustainable Dyeing, Water-less Textile Dyeing, Supercritical, Carbon Dioxide, SCF, Green Technology, Wet processing

1. Introduction

Traditionally, water has been a popular medium used in dyeing, finishing, cleaning textile materials. It makes the textile industry one of the largest consumers of water resources. On the other hand, the cost of input water and wastewater treatment is ever increasing along with the pollution control norms becoming more stringent each year. Also globally, usable water resources are becoming alarmingly scarce. Recently in December 2020, water has even started trading on Wall Street as a 'Futures commodity' to join the likes of Gold and Oil.

In this regard, it has become very critical that Textile processes that use minimum or no water are developed and adapted on a commercial scale. In recent years, the use of supercritical fluids as a replacement for water as a solvent, in the Dyeing process has attracted the attention of the Textile industry. Carbon Dioxide (CO_2) has emerged as the most preferred supercritical solvent. Major advantages of Supercritical CO2 (SC CO_2) based Textile Dyeing process which also improves its 'Economic Viability' and 'Consumer preference' are as follows:

1) Zero discharge: Elimination of waste water streams, Pollution.

2) Shorter process and dyeing times because:

   I) SC CO_2 penetrates in the polymer matrix and swells it to help in faster diffusion of dye molecules within the polymer matrix.

   ii) SC CO_2 has negligible surface tension resulting in efficient wetting of polymer surface and faster penetration in voids of textile material.

   iii) SC CO_2 has a low viscosity which helps in efficient and easy circulation of the solution of SC CO2 and dye, through the textile material.

   iv) SC CO_2 has higher diffusivity which helps in faster mass transfer.

3) Efficient process because of Lower dye consumption, no wastage and dye can be reused.

4) Energy saving process due to minimum requirement of expensive 'heat energy' and resource required for post dyeing repeated water washing and drying of dyed fibre or fabric.

5) SC CO_2 is recyclable. inert, nonexplosive, Generally Regarded as Safe (GRAS) solvent.

6) There is no damage to the fibre or fabric.

7) Many pre and post treatments of textile material are simplified or eliminated.

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Limitations of Prior Art Technology / Motivation for Innovation:

The conventional/prior art supercritical dyeing processes that were available in the world had the following major limitations which have also resulted in limited adoption of the said prior art supercritical fluid-based sustainable processes:

(i) The dye needs to be first dissolved in SC CO_2 and then transported to the Textile in placed in a Dyeing vessel.

(ii) Dyes have Low solubility in SC CO_2 resulting in low dye concentration in the dye solution.

(iii) The low residence time of dissolved dye flowing through the Dyeing vessel, limits the contact, interaction of Textile material with dye molecules.

(iv) Some part of dissolved Dye that is flowing through the 'Dyeing vessel' may not come in contact with the Textile surface. Also, Non-uniform flow / Channelling of SC CO_2 solution through a Textile roll in a Dyeing vessel can lead to non-uniform contact and thus non-uniform dyeing in large scale operation. Thus, it may require special, complicated additional devices to impart say rotational motion to the textile roll in dyeing vessel, to improve the uniformity in dyeing.

(v) Thus, only part of the available dissolved dye may take part in SC CO_2 Dyeing to achieve desired colour Intensity on Textile material.

(vi) The final shade of dyed cloth depends on the extent of exposure as the shade keeps getting darker with the passage of contact time with fresh dye solution entering the Dyeing vessel, making it difficult to control Batch Batch variation.

(vii) Mainly useful for applying Dark shades with a single colour at a time.

(viii) All the above limitations make the prior art SC CO_2 dyeing process Less versatile, Slow and Less efficient.

Hence, the objective of innovative work at DSPL was to develop an improved dyeing process:

- To get Uniform, Reproducible Interaction between Dye molecules and the entire surface of Textile material.
- To Improve the rate of Solubilisation of Dye molecules in Supercritical CO_2 solvent to increase the Rate and Efficiency of the SC CO_2 dyeing process.
- To achieve easy scale-up to large scale dyeing while maintaining desired Uniform & Reproducible Colour intensity on textile material.
- To get dyeing of the textile materials with a single or multi-colours in various shades, patterns etc. in a single step of dyeing operation.

2. Methods & Materials:

2.1 Details of Innovative SCF Dyeing Process from DSPL:

Innovation has been carried out by following steps:

Making a dye solution: By mixing the dye material and auxiliary chemicals with a suitable solvent. We preferably use water as a solvent for the conventional dyes along with the dispersing, levelling agents.

Pre-treatment: Pre-coating the surface of textile material to be dyed with an optimum quantity of the above dye solution to obtain a dye coated textile material. Any standard method of coating can be used such as Roller coating, Ink-Jet Printing etc.

Supercritical CO_2 process: Placing dye coated textile material inside the supercritical 'Dyeing vessel' on a supercritical fluid processing plant.

Adding the supercritical CO_2 into the 'Dyeing vessel'. Exact operating conditions are optimized as per the type of dye, auxiliary chemicals and textile used, wherein the supercritical CO_2 solubilizes the dye molecules that were earlier coated on the surface of the textile material and further diffuses the solubilized dye molecules inside the surface, pores and capillaries of the textile material;

Depressurizing the supercritical fluid dyeing vessel to precipitate and entrap the dye material inside the textile material.

Post-Treatment: Mild soap washing of the dyed & finished textile with and Stentering.

2.2 Innovative Elements of Patented Process from DSPL:

Novel / Inventive step: Pre-coating of textiles to be dyed, with the optimum quantity of dye & auxiliary chemical molecules, per unit area of textiles to increase the surface area of solute and improve the rate of solubilisation of dye & other molecules in supercritical CO_2. This also improves uniformity, reproducibility of dyed shade, washing fastness and finishing effect.

Non-Obviousness: Use any pre-coating method such as inkjet printing, Roller coating or similar process for having a controlled pre-coating of optimum quantity of dye molecules, auxiliary chemicals on the textile material to achieve a single or multi-color/light or dark shade dyeing of textiles with post-processing with SC CO_2.

Industrial applicability: Patented improved technology from

DSPL eliminates major limitations of prior art Supercritical dyeing processes available in the market. Its innovative features make it very simple, easily scalable, most efficient and economically viable, to truly achieve the sustainability goals of the user industry.

3. Results and Discussion:

Yellow Magenta

Cyan Black

Fig. 1 Photos of 'R-Elan GreenGold' # polyester fabric dyed with disperse dyes with SC CO_2 based patented process from DSPL

(# 'R-Elan GreenGold' is a brand of Reliance Industries, India, for a special Polyethylene Terephthalate (PET) fabric from recycled PET bottles to address environmental pollution)

a) Patented SC CO_2 Dyeing from DSPL | b) Conventional Water-based Dyeing | c) Prior art SC CO_2 Dyeing

Fig. 2 Photos of R-Elan GreenGold polyester fabric dyed with Navy Blue (1.3 % Shade) using Coralene Navy Blue 3G H/C disperse dye from ColourTex using three different dyeing processes

As is seen in Figure 2, Patented SC CO_2 Process from DSPL gives 18 to 24 % darker colour shade on same "GreenGold" fabric, as compared to samples obtained from conventional water-based dyeing as well as Prior art supercritical dyeing (with same dye & same quantity of dye being used).

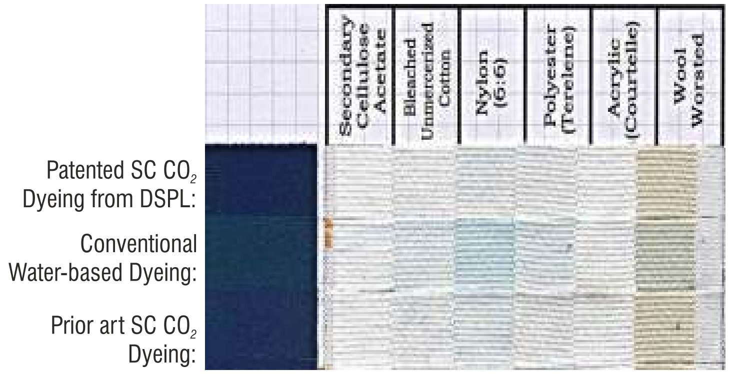


Fig. 3 COLOUR FASTNESS Results as per ISO:105:E01, for dyed GreenGold Fabrics as stated in Fig. 2

As seen in Figure-3 innovative, patented process from DSPL is more efficient and gives better Colour, Washing Fastness.

Navy Blue (2.1 % Shade) by SC CO_2 based Dyeing & Finishing from DSPL | *Navy Blue (3.0 % Shade) by Conventional Water-based Dyeing & Finishing*

Fig. 4 Photos of SORONA# Polyester fabric dyed using disperse dye Dianix Navy XF2 from DyStar using two different dyeing processes

 # "Sorona" is DuPont's brand for an eco-efficient performance Polyester produced by using one of the monomers: 1,3-propanediol, which is obtained from renewable (Plant-based) sources.

As seen in Figure-4, the improved SC CO_2 Dyeing and Finishing Process from DSPL matches the required Navy Blue shade with about 30 % less Dye as compared to the conventional Water-based dyeing process, carried on the same Sorona fabric.

Dyeing of Micro-Denier Polyester Fabric with Patented Process from DSPL:

The micro-denier polyester fabrics have a very high surface area, which poses challenges in dyeing with the conventional Water-based dyeing process. It shows problems like unlevelled dyeing, lower colour depths, lower washing fastness etc. with regular types of dispersed dyes.The above issues are sorted by improved, patented SC CO_2 Dyeing technology from DSPL. As seen in the right side photo of micro-denier polyester fabric (Microsupersoft (125/288), Plain Interlock) dyed with our technology using regular disperse dye (0.75 % Shade of Golden Yellow GG 200%, from Spectrum). A very uniform, levelled dyeing achieved with good colour depth and excellent colour fastness to washing of 4-5.

Amber (1.5 % Shade) on 100 % Cotton fabric (130 GSM)	Yellow Brown (1.5 % Shade) on Polyester-Cotton (67:33) Blend fabric (125 GSM)	Blue (1.5 % Shade) on Polyester-Cotton (67:33) Blend fabric (125 GSM)

Fig. 5 Photos of Cotton and Polyester-Cotton Blend Dyed with SC CO2 based patented process from DSPL

Results of Life Cycle Analysis (LCA) carried out using 'GaBi' software :

INNOVATION

Patented, Most Efficient Supercritical (SC) CO_2 based sustainable single step Dyeing as well as Finishing technology

FEEDSTOCK/INPUT

- Innovative SC CO_2 based dyeing from DSPL is suitable for man-made, natural and blended textiles.
- Allows use of traditional dyes with Improved dye utilisation
- Finishing chemicals can be applied along with dyeing in single step
- Much lesser quantity of Auxiliary chemicals required as compared to conventional water based dyeing

END OF USE

The End of Use would depend on the type of fabric used for dyeing

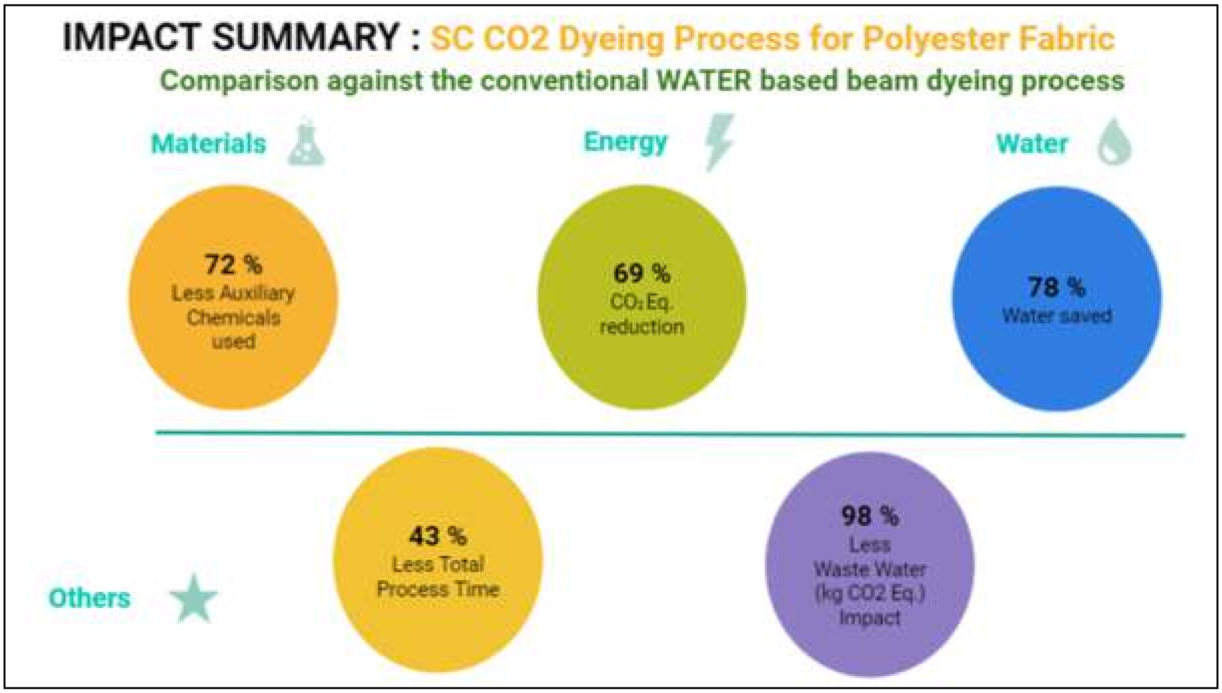
CERTIFICATIONS

None at this stage. We have 3rd party Test Reports from DuPont, ColourTex, BTRA, about the efficient dyeing and Colour Fastness by innovative technology from DSPL

'SCREENING LCA' OUTCOME: SC CO2 Dyeing Process for Polyester Fabric

Comparison against a conventional WATER based beam dyeing process

Name	Beam Dyeing with WATER (kg CO2 eq.)	SC CO2 Dyeing (kg CO2 eq.)	% Impact Reduction
Process Water	0.0640	0.0140	78
Electricity	0.0150	0.0090	40
Steam	1.0000	0.3100	69
Acetic Acid	0.0130	0.0005	96
Disperse Dye	0.0850	0.0680	20
Soaping Agent	0.0092	0.0046	50
Carbon Dioxide	0.0000	0.0470	-
Waste Water	0.4570	0.0110	98
Total	1.6432	0.4641	72



Improved Economic Viability due to Innovation:

Innovative Supercritical CO_2 based dyeing process from DSPL has improved economic viability due to the following important factors:

1) Process from DSPL is simpler, versatile & efficient with less than half dyeing time vis a vis Prior-art processes, increasing processing capacity & reducing processing cost.

2) We can use conventional dyes traditionally used by industry. Thus not necessary to use expensive special dyes required by prior-art processes, improving viability.

3) Here thin, a controlled layer of dye is Pre-coated on the surface of textile to be dyed. This increases the effective surface area of solute (Dye) and thus increases interaction and rate of solubilisation insolvent (SC Co_2).

4) With the availability of the optimum and uniform quantity of dye molecules on the entire surface of textile material (in form of pre-coating of the very thin layer), the supercritical fluid efficiently dissolves the dye molecules and make them penetrate inside the textile matrix to achieve uniform and efficient dyeing all over.

5) Thus, in the process from DSPL Dye molecules are not required to be transported as a Dye solution in supercritical medium, from the 'Dye-Mixing vessel' to the textile material kept in 'Dyeing Vessel'. Also, contrary to the prior-art process, it does not remain critical for the said dye solution to flow and distribute uniformly, over each part of the role of textile material for achieving uniform, reproducible dyeing, even for lighter shades.

6) Pre-coating of the Textile surface with the optimum quantity of dye also minimises wastage of dye in overall dyeing operation. Thus lower dye quantity is required for achieving a specific shade as compared to the conventional dyeing process.

7) This also enables efficient Dyeing with desired Uniform, Reproducible colour shade, on man-made, natural or blended textile materials (fibres & fabrics), in a single step.

8) It also saves expensive 'heat energy' resources otherwise required for post dyeing repeated water washing and drying of dyed Textiles.

9) This is a 'Zero Discharge' process, minimises ETP costs. SC CO_2 solvent is recycled.

10) Any dye recovered in 'Separator' can be reused as there is No hydrolysis or degradation of dye in SC Co_2.

11) This innovation also makes the scale-up of the Dyeing process easier as desired Dye molecules are already made available on the entire surface of Textile material kept in the Dyeing vessel, minimising the fluid and mass transfer related issues.

12) Better premium and higher preference from customers for Genuine 'Eco Friendly', 'Green' dyeing processes: giving major economic and marketing advantage.

13) Innovation from DSPL allows dyeing & finishing process with softeners, antimicrobials etc. in a single step. Thus Saves on process steps, chemicals, water, time & energy.

4. Conclusions

Supercritical fluid-based dyeing and finishing technology from Deven Supercriticals Pvt. Ltd., India is uniquely suitable for not only man-made fabrics like polyester, Nylon but also for cotton and blended textiles. Also, it allows the use of conventional dyes with no requirement for special expensive dyes. The same recipe of dyes as used in the conventional water-based process can be used in this innovative process but without the use of water in the dyeing process. It further shows improved dye utilization, makes scale-up easy and has less than half dyeing time vis-a-vis prior-art SC CO_2 based dyeing processes. There is no need for reduction clearing for polyester, no salt added for cotton dyeing, single-step dyeing possible for blend textiles, reducing the overall requirement for auxiliary chemicals. Thus substantially reduces the pollution, water and energy load. It has made the SC CO_2 technology truly viable, versatile and simple. Thus at present and in the future, the improved, efficient and patented supercritical fluid dyeing and finishing technology from DSPL, which takes care of the limitations of the prior-art SC CO_2 based technologies, has great potential to truly Accomplish Environment friendly, Green objectives of Textile Industries around the world for utilising Sustainable processes vis-a-vis the traditional processes which have a negative impact on health and environment.

References

1] "Process for dyeing of textile materials using supercritical fluid", Inventor: Dr Swapneshu Baser, Indian Patent no. 298213 granted in 2018.

2] "Process for dyeing of textile materials using supercritical fluid", Inventor: Dr Swapneshu Baser, United States of America Patent No. US 11015289 B2 granted in 2021.

Basics of Electrically Conductive Textiles: Manufacturing, Characterization, and Applications

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Abstract

Textiles that carry electricity are known as conductive textiles. It is made up of nonconductive substrates like cotton, polyester, and nylon that are either coated or implanted with electrically conductive materials including nickel, copper, gold, silver, titanium, carbon and so on. Metal mesh, aerospace textiles, taser or stun gun jackets, conductive threads or yarns, fabric sheets used for thermal heating, and other products based on these conductive textiles These fabrics can be employed in a variety of applications due to their conductivity. Low-conductivity fabrics can be used for antistatic or ESD clothing, medium-conductivity fabrics for smart textiles or wearable electronics, and very high-conductivity fabrics for EMI shielding applications. The ways of producing conductive textiles, their characterization in terms of surface/volume resistance are discussed in this work. This paper also mentions BTRA's Conductive textile testing facility which includes Electrometer, EMI shielding analyzer, and Static honestometer.

Key words

Electrically conductive textiles, Resistivity, EMI Shielding, antistatic clothing

1.0 Introduction

The opportunities for conductive fibre applications have greatly expanded as a result of today's technological advancements. Engineers and manufacturers are working on ECG sensors, blood pressure sensors, body glucose level sensors, strain sensors, temperature sensors, and other products that may be worn for sports and health monitoring. According to IDTchEx's research "Wearable Technology Materials 2015-2025," more than $25 billion will be spent on formulations and sophisticated textiles for wearable technology by 2025[1]. The E-textiles or E-fabrics sector is expanding, and conductive fabrics are being used in the electronics industry to help manufacture smaller goods. E-fabrics are far more flexible than normal metal wire, making smaller electronics possible.

Applications of textile materials depending on their surface resistance are shown in Table.1.

Table-1 Application based surface resistivity of conductive fabrics [2,3]

Surface Resistivity (ohms/square)	Applications
$>10^{12}$	Insulating textiles
10^{9} to 10^{12}	Antistatic textiles
10^{6} to 10^{9}	Electrostatic discharge clothing
10 to 10^{3}	Wearable sensors, heat generation
<10	EMI Shielding

Flat textile materials' electrical conductivity is determined by the electrical conductivity of their constituents, such as fibres and threads. Electrical conductivity is influenced by the structure of textile materials. Metal-dielectric composites can be compared to electro-conductive woven and knitted fabrics in general [4,5]. Interlaced conductive threads and holes filled with dielectric air make up such systems. The interlaced threads have contact points and a contact surface. As a result, the electrical resistivity of a woven or knitted structure is determined by the electrical resistivity of linear components, component contact resistance, and pore volume fraction[6]. The fundamentals of conductive textile in terms of applications, manufacturing techniques, and characterisation are explained in this paper.

2. Applications of Conductive Textiles :

Functional and interactive clothing is one of the areas that have expanded the working domain of textiles beyond their traditional uses. These futuristic textiles have the potential to revolutionize the way of communicating, health monitoring, handling emergencies, fashion, and recreation [7,8]. The enormous development in the field of material science in terms of smart and functional materials and their merging into textiles has resulted in textile materials with smart functions, high performance, and novel functionalities. Imparting electrical conductivity to textiles is one of the functionalities that assume significant importance in application areas of static and electrostatic discharge (ESD) protection, electromagnetic interference (EMI) shielding, heat-generating textiles, and microwave

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attenuation[8,9,10,11]. These applications of conductive textiles cater to diverse areas ranging from mundane domestic items such as static shockproof carpets to static dissipative garments and accessories for the electronics & semiconductor industry and high-tech applications such as stealth technology for defense establishments[10].

Fig.1: Prominent applications of conductive textiles

Recent progress in the field of Smart and interactive clothing has put a thrust on electronic textiles which has tremendous applications in the field of fashion, entertainment, healthcare, fitness monitoring, personal protection, and defense[10-12]. The current thrust is on imparting electronic functions such as sensors and actuators into textiles themselves. This has presented unprecedented scope for the utilization of conductive textile substrates for the sake of functions such as sensors, actuators, and data transmission. The main reason behind turning towards electrically conductive textiles for the above-mentioned applications areas not only lies with their flexibility and lightweight but also in their undisputable presence in all spheres of human life.

Smart textiles are the area that has its applications in the field of medical, health care, sports, fitness monitoring, fashion, entertainment, protection, and military[13,14]. The role of electrical conductivity is paramount in such textiles considering the involvement of tasks such as sensing, actuation, and data transmission. Some of the earlier smart textile systems relied on the attachment of electronic devices to the textile matrix for achieving desired functionality. Gradually, the focus of research has shifted towards replacing hard electronic parts with textiles embedded with electronic functionalities. The convergence of electronics, electrical engineering, and textile technologies has the potential to combine the positive attributes of each technology, the speed and computational capacity of modern electronics with the flexible, comfortable, and continuous nature of textiles.

A closer look at literature showed that conductive textiles can be broadly divided into two regimes with regards to their applications areas. Firstly the low conductivity regime whereby a minor conductivity level of textiles is employed for applications that demand controlling electrostatic charge generated on the clothing and work surface. The static dissipation has acquired a critical role not only in shock prevention in the day-to-day domestic situation but also in the form of ESD damage in the semiconductor and electronics industries[15]. Most importantly spark generation from static charge may cause explosions hazards in the work areas where flammable gases, vapours, or powders are handled. Hence there is a need for antistatic garments. In comparison, the second regime of high electrical conductivity can cater to applications where a superior level of conductivity is desired. For instance, EMI Shielding, RADAR Wave attenuation, and smart textiles are some of the application areas. Here high conductivity is a primary requirement for textile substrates for delivering optimum performance. EMI shielding is important in the electronics industry to safeguard delicate instruments from damage due to interference caused by unwanted EM radiations emitted from electric and electronic instruments. Further, the ever-increasing EM radiations emitted by electronic equipment and cell phones antennas are believed to pose certain hazards to human health such as insomnia, languidness, headache, and nervousness[16]. Typically metal-based conductive textiles have been used to block EM radiations.

3.0 Manufacturing of conductive textiles:

3.1 Metal-based technologies:

High conductivity $(<10^{5}\Omega\ x\ cm)^{-1}$ in textiles can be obtained by the draw blending of metal slivers and slivers composed of textile fibers. Metal fibers are expensive and they can also be as much as 5 times heavier than some textile fibers. Also made of a homogeneous blending difficult to produce. As metal fibers are brittle and can abrade the spinning equipment. we can blend them with other fibres. So that the friction with the equipment can be reduced and metal will be in less contact with spinning equipment.

Metal yarns can be produced as core-spun yarn or wrapped yarn. A core-spun yarn can be made on most spinning frames. After the passes through two drafting rollers, the core is introduced. To produce a conductive yarn, which has been coated with a conductive substance could be introduced as the core or sheath component. The wrapper yarn can be either a monofilament or a multifilament. It can be wrapped in a right-hand direction (Z) or a left-hand direction (S).

Electrical properties can be incorporated over the fabric in form of coating. Application of metal to textiles substrate can be done by electroless or electrocoating)

The electroless coating is a method of plating a metal without using an electric current and as a result of a chemical reaction between a reducing agent and metal ions. Nickel and copper are the most common metals used today. Some of the common reducing agents are sodium hypophosphite, formaldehyde, hydrazine, and organoboron compounds. Each combination of metal and reducing agent requires a specific pH range and bath formulation. The coating thickness varies between 0.01 µm to 1 mm. Advantages of this electroless coating process are it possess uniform coating thickness(irrespective of the shape of substratum) and unique mechanical, magnetic, and chemical properties.

Electrocoating of metal involves processes such as the sputtering process, evaporative deposition, and electroplating method. The sputtering process takes place in the vacuum chamber equipped with a target (cathode) and a substratum (anode) to be coated. It involves the application of an electric potential between the target and the substratum. In response to the positive ions, the target ejects atoms into the gas phase. The ejected atoms reach the substratum at a high velocity. Once the ejected atoms from the target reach the substratum, they condense and form a thin layer. Similarly, evaporative deposition takes place in an evacuated chamber. Here the metal is heated to a temperature that maximizes evaporation and then the fabric passes over a water-cooled drum where it is exposed to the vapor of the molten metal. The metal condenses on the fabric surface and is deposited in a very thin layer of thickness. Electroplating takes place in electrolyte cells.

General limitations of these Metal-based conductive textiles include Difficulties during fibre processing, Poor flexibility, Metallized handle, Costly process, and involvement of complex manufacturing processes.

3.2 Coating fibers with conductive particles suspended in a resin

Fibers can be coated with a resin in which a high concentration of conductive particles has been dispersed. Some of the earliest conductive products were produced by applying high concentrations of carbon resin to a fiber. Carbon fibers have fairly high conductivity, (10^5-10^0[Ω x cm]$^{-1}$) , especially if pure carbon is used. Pure carbon can produce fibers with conductivities as high as (10^2 [Ωx cm] $^{-1}$). However, carbon can be difficult to process and any amount of carbon fibers will impart black color to the end product.

3.3 Coating Textiles with Conductive Polymers:

Intrinsically conductive polymers (ICP's) such as polypyrrole, polyaniline, polythiophene can be coated over the fabric using oxidative in situ chemical polymerization. The high level of conjugation along the doped polymer chain required for electrical conductivity caused the polymers to be rigid and stiff. A high melting temperature and poor solubility of monomer resulted in difficulties in processing. Because of the high melting temperatures and poor solubility of the polymers, they could not be processed by conventional methods such as melt or solution spinning. Low mechanical strength and poor stability to electrical and environmental conditions limit its industrial use.

4. Spectrum of materials based on conductive properties

Based on the conducting properties materials can be classified into the following four categories.

i. Conductors: Material capable of carrying an electric current, i.e. material which has "mobile charge carriers" (e.g. electrons, ions,..)e.g. metals, liquids with ions (water, molten ionic compounds), plasma

ii. Insulators: Materials with no or very few free charge carrierse. g. quartz, most covalent and ionic solids, plastics

iii. Semiconductors: Materials with conductivity between that of conductors and insulators; e.g. germanium Ge, silicon Si, GaAs, GaP, InP

iv. Superconductors: Certain materials have zero resistivity at very low temperatures.

Based on this spectrum electrically conductive textile can also be classified as conductor, semiconductor, or insulator based on its application.

5. Characterization Techniques:

Following are some important terms and electrical characteristics that to be known for analyzing the performance of developed conductive fabrics as the degree of conductivity is directly indexed to the performance of textiles in certain applications.

5.1 Conductance: It is reciprocal of resistance.

5.2 Electrical conductivity: It is a measure of how well a material accommodates the movement of an electric charge. It is the ratio of the current density to the electric field strength. Its SI-derived unit is the Siemens per meter.

5.3 Electrical resistivity: It is the reciprocal of conductivity. It is the opposition of a body or substance to the flow of electrical current through it, resulting in a change of electrical energy into heat, light, or other forms of energy. The amount of resistance depends on the type of material. Materials with low resistivity are good conductors of electricity and materials with high resistivity are good insulators.

The SI unit for electrical resistivity is the ohm meter.

This electrical resistance is proportional to the sample's length and the resistivity and inversely proportional to the sample's cross-sectional area.

$$R = \rho\, l/A$$

Where:

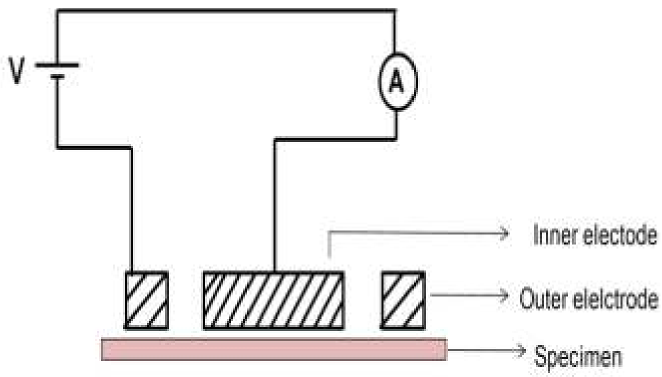
ρ = Resistivity

l = length

A = Cross-sectional area

5.4 Surface resistivity : It is defined as the electrical resistance of the surface of an insulator material. It is measured from electrode to electrode along the surface of the insulator sample. Since the surface length is fixed, the measurement is independent of the physical dimensions (i.e., thickness and diameter) of the insulator sample. Surface resistivity measurement can be done by using two types of the electrode, circular probe, and flat plate electrode. Resistivity calculation will be differ based on the use of the electrode.

Circular probe electrode



$$\mathbf{Sr = PR/g}$$

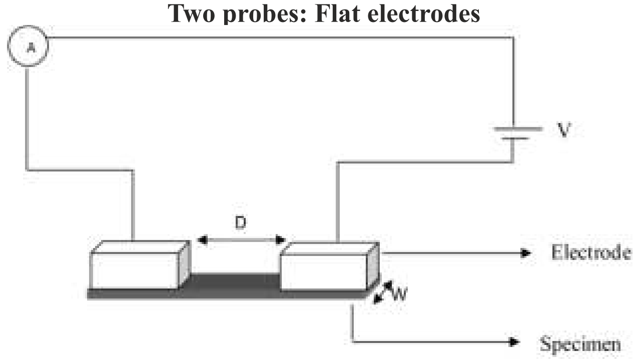
Sr = surface resistivity (per square)

R = measured resistance in ohms (V/I)

P = the effective perimeter of the guarded electrode (mm)

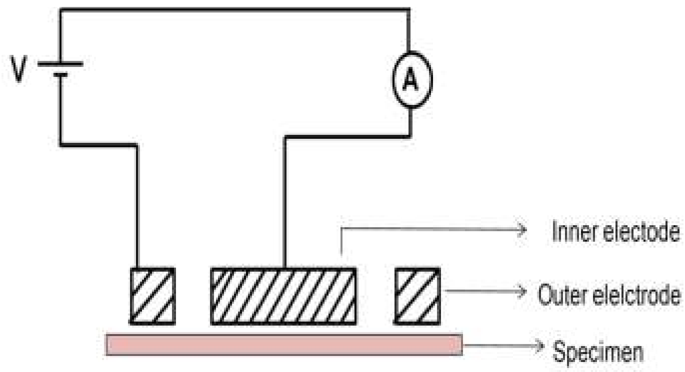
g = distance between the guarded electrode and the ring electrode

Two probes: Flat electrodes



Surface resistivity = $\dfrac{\text{Resistance x Width}}{\text{Distance}}$
(ohm/square, Ω/sq.)

5.4 Volume Resistivity: Volume resistivity is defined as the electrical resistance through a cube of insulating material. When expressed in ohm centimeters it would be the electrical resistance through a one-centimeter cube of insulating material.



$\rho V = K v R/t$

KV = the effective area of the guarded electrode for the particular electrode

T = average thickness of the sample (mm)

R = measured resistance in ohms (V/I)

6.0 Conductive Textile testing facility of BTRA:

For the last 12 years, BTRA is actively working in the areas of conductive textiles.BTRA is having a fully-fledged facility for conductive textiles testing. For the measurement of surface resistivity, there are various standard test methods are followed such as ASTM (D257:2007) and AATCC 76:2013 for textile and clothing. The characteristic feature of these standards is the type of electrodes which essentially consists of two circular concentric rings or flat plate electrodes.

Fig. 6(a). Actual photograph of instrument for two probe concentric rings electrodes

In this method, two concentric ring electrodes are placed onto a specimen surface. The application of voltage to the inner electrode results in the passage of current through another electrode via contact specimen surface (Fig. 6a). This method has been adopted by standards about static and ESD protection fabrics.

In comparison, for the measurement of high electrical conductivity, the two probes flat electrode method has been reported.

Fig. 8. Actual set-up devised for twp probe flat electrodes

Fig 8 demonstrates an actual photograph of a two-probe flat electrode system existing in BTRA. These two probe method is based on the AATCC test method 76-2006; it is expressed by the units 'ohms/square'(ohm/□, Ω/□). For measurement purposes, the electrode assembly based on two flat copper electrodes (30 mm X 20 mm) separated by a distance of 20 mm was designed. A voltage source was connected to the electrodes through a multimeter. The measurement involved placing the electrode assembly over a fabric specimen (30 mm × 60 mm) with a weight of 5 kg. The electrical resistance values in each case were obtained by plotting the current versus voltage values (I/V curves).

$$\textbf{Electrical surface resistivity} = \frac{\text{Resistance} \times \text{Width of electrodes}}{\text{Distance between electrodes}}$$

Electromagnetic shielding effectiveness (EMSE) of fabrics can be measured according to ASTM-D-4935-99, for planar materials using a plane-wave, far-field electromagnetic wave. The SE of the sample can be measured over the frequency range from 100kHz to 1.5GHzusing circular coaxial transmission line holder. Network analyzer (9KHZ-4.5GHz) from Agilent Model E 5071C and SE test Fixture from Electrometric Model EM-2107A here used for measuring Shielding Effectiveness. Analysis of static charge, half decay time and performance evaluation of antistatic oil, filters &fabrics can be analyzed on static honestometer (Model S-5109).

7. Conclusion:

In summary, electrically conductive fabrics' tuneable conducting properties make them appropriate for a wide range of applications. This study contributes to a basic knowledge of the mechanisms involved in the fabrication of metal, carbon, and intrinsically conductive polymer-based conductive fabrics, as well as their characterisation in terms of surface/volume resistivity. Antistatic filters, dust-free and germ-free clothes, microwave attenuation, electromagnetic interference (EMI) shielding, and resistive heating textiles are all examples of electrically conductive fabrics. The surface or volume resistivity of conductive textiles is an important parameter to consider when evaluating their performance. BTRA's facility of conductive textile testing (such as surface or volume resistivity, EMI shielding effectiveness, and electro propensity measurements) can be utilized by the industry for accurate assessment.

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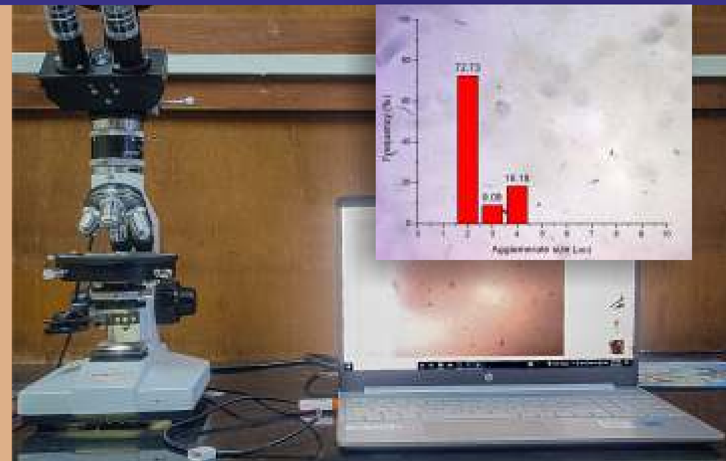
Dispersion Studies of Single Wall Carbon Nanotubes in Polyacrylonitrile



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Abstract

With the increasing demand for advanced materials in the world, carbon nanotubes (CNT) have always been the first choice for material properties enhancement but their uniform dispersion is a big challenge for obtaining desired results in various applications. The recent developments of CNT and Polyacrylonitrile composite show that they not only act as a good reinforcing agent but also as property enhancers of carbon fibres produced from it. However, agglomerate size and dispersion of CNT play an important role in resultant carbon fibres, since big agglomerate size results in a defect in fibre. In this research work, we are reporting the dispersion of ultrasonicated single wall carbon nanotubes in polyacrylonitrile dope solution by mechanical stirring. Significant improvement in the properties of polyacrylonitrile was obtained due to the effective dispersion of single carbon nanotubes which were characterized by optical microscopy, Brookfield viscosity measurement, and solvent resistance test.

Key Words:
Polyacrylonitrile, Single walled carbon nanotubes, microscopy, nano-composite, CNT dispersion.

1. Introduction

Carbon nanotubes (CNT)are one the best reinforcing agents used for making high ends products. It is well known that CNT also acts as a nucleating agent for polymer crystallization[1]. This behavior has been observed with many polymers like Polypropylene, Polyethylene, Nylon, Polyacrylonitrile (PAN), etc., [1-4]. Being a precursor for carbon fibre production, PAN has significant importance than the other polymer nano-composite. CNT in PAN matrix not only acts as a nucleating agent but also behaves like the template to form a graphitic structure at the carbonization stage [5].

The addition of multiwall carbon nanotubes (MWCNT) and single wall carbon nanotubes (SWCNT)in PAN has been reported by several research groups. Though the dispersion of MWCNT is better than SWCNT resultant performance of SWCNT reinforced showed better performance than MWCNT, because of its low diameter of few nano-meter and greater surface area [6]. This increases the effective number of CNT present along the axis of carbon fibre which affects the carbon fibre modulus and tensile strength. The presence of more CNT oriented along the direction of the fibre axis results in better mechanical properties of the resultant carbon fibre which is greatly related to the dispersion of the CNT in the PAN matrix. Better dispersion of CNT in PAN not only gives them a more effective number of CNT along the carbon fibre axis but also gives more surface area to PAN polymer chains for nucleation and increase in strength.

Though the addition of a single wall carbon nanotube in the PAN matrix has many benefits but finding a suitable solvent for both is a very tough task. PAN being polar and SWCNT being non-polar, complete dispersion of SWCNT is always a challenge in any PAN dissolving solvents [7]. Arias-Monje et, al, reported 2-3 times better mechanical strength in 15wt% CNT reinforced carbon fibre than virgin PAN carbon fibre [8]. Loading of that high amount of CNT had surely made the carbon fibre stronger but limited its use for industrial application.

For industrial application purposes, similar strength is required for low CNT loading as low as 1wt% which will make it high-performance carbon fibre. The same can be achieved by functionalizing the CNT which will provide better dispersion of nanotubes in the PAN matrix [9]. Quan et. al, has investigated the dispersion of amine-functionalized CNT in PAN matrix against the virgin CNT [10]. Due to amine-functionalization, surface cohesion energy of nanotubes was greatly reduced which allowed better dispersion and interaction of CNT with PAN polymer chains but its effect on resultant carbon fibre is still unknown.

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Since, producing carbon fibre from PAN involves various reactions like oxidation, cyclization, dehydrogenation and finally, carbonization and effect of functionalization is still undiscovered in these processes.

In this study, we proposed a combination of ultrasonication and mechanical stirring for dispersion of SWCNT in PAN matrix where Dimethyl acetamide (DMAc) was used as a common solvent. Dispersion of SWCNT in PAN is characterized by Optical microscopy, Brookfield viscosity, Opaqueness of PAN/SWNCT composite films, and Solvent resistance test.

2. Experimental:

2.1 Material

Polyacrylonitrile was procured from Technorbital Advance Materials Pvt Ltd of molecular weight 116000g/mol. SWCNT was procured from AdnanoTechnologies Pvt Ltd with a purity of 99%. Dimethyl Acetamide was procured from Venus Trading Corporation, of commercial grade.

2.2 The procedure of making PAN-SWNT composite

Stock solution preparation of PAN in DMAc:

Before proceeding with solution making, PAN polymer was dried in a vacuum oven @ 50°C overnight to remove residual moisture content. The solutions of PAN in DMAc in the concentration of 15wt% were prepared by dissolving 225g of dried PAN in 1000 ml of DMAc respectively. All solutions were prepared under a hot bath by maintaining 55±2°C bath temperature and mechanical stirring at 500 rpm. Mixing was done until PAN was completely dissolved.

SWCNT stock solution preparation:

Concerning PAN concentration suitable amount of SWCNT was added to 50 ml DMAc to obtain 0.1 wt%, 0.2 wt%, 0.5wt%, and 1wt% PAN/SWCNT composite solution. After the addition of SWCNTs in DMAc in the respective amount, it is subjected to ultrasonication for 4 hours.

PAN-SWCNT composite preparation:

Respective composite solutions were prepared by mixing the PAN solution and SWCNTs solution in a mechanical stirrer @ 3000rpm for 6 hours and samples from each composite sample were drawn after every 2 hours of mixing time for Optical microscopy.

3. Characterization

3.1 Optical Microscopy:

Optical microscopy was done with an Almicro optical microscope (model P12). Samples for microscopy were drawn at the interval of 2 hours sonication, 4 hours sonication, 2 hours shear mixing, 4 hours shear mixing, and 6 hours shear mixing respectively. The analysis of agglomerate size was done by Biohazard v4.0 software (Microscope software) for calculation.

3.3 Brookfield Viscosity:

Brookfield viscosity of all samples was measured with BrookfieldSynchro-Lectric Viscometer (ModelLVF), made by Brookfield engineering laboratories Inc, Stoughton, Massachusetts at the speed of 3-30 rpm with spindle numbers 3 and 4 at 35°C.

4. Result and discussion:

4.1 Optical microscopy

Ultrasonication is a very effective and powerful tool in breaking the SWCNT agglomerate size but uniform dispersion with only sonication is very difficult to obtain. Employing the combined technique for agglomerate size reduction and dispersion was effectively achieved with sonication and shear mixing. The vibration produced during sonication of SWCNT in DMAc decreases the cohesion energy between SWCNT agglomerates gradually but complete dispersion with sonication alone will take a very long time. The SWCNT dispersion will remain stable under the influence of sonication only, on the removal of vibration energy from sonication these SWCNT will again tend to combine and form agglomerates. Thus, shear mixing of the SWCNT/DMAc in PAN/DMAc dope solution allowed PAN polymer chains to penetrate in the sites of SWCNT agglomerate which were opened by sonication. The drastic reduction in the agglomerate size just after 2 hr of shear mixing gives evidence the PAN polymer chains are very effective in dispersing and keeping the SWCNT stable in the composite solution. Figure 1a)-1d), represents the Optical microscopy images of PAN/SWCNT solution at various stages.

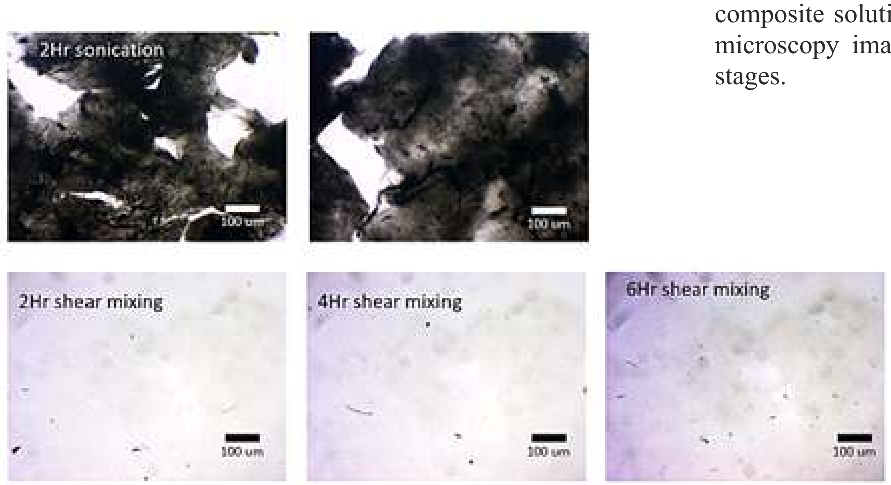

Figure 1a) Optical images of 0.1wt% SWCNT in PAN at various stages

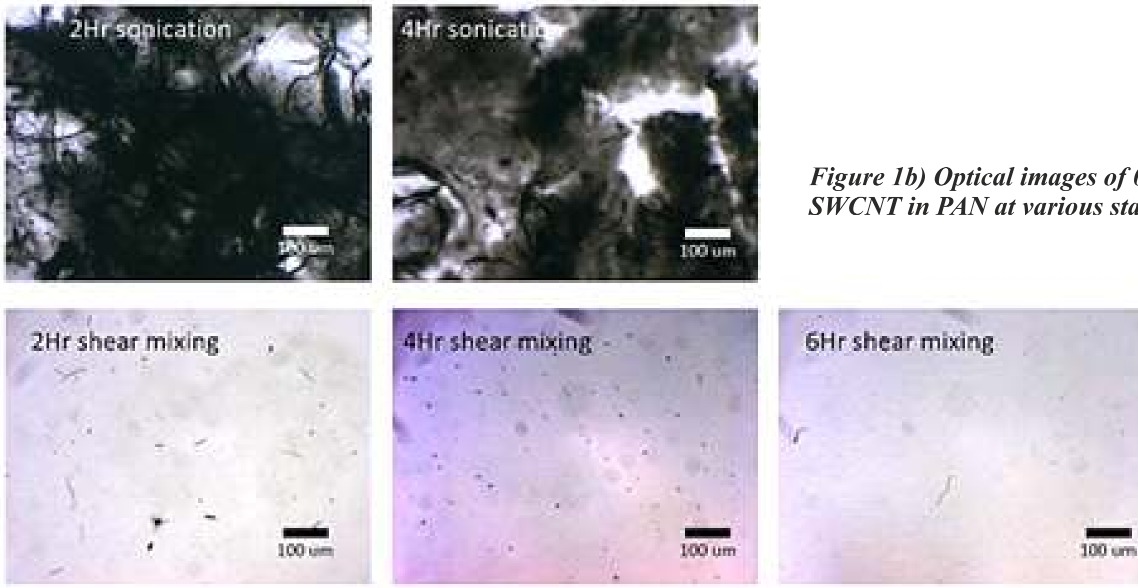

Figure 1b) Optical images of 0.2wt% SWCNT in PAN at various stages

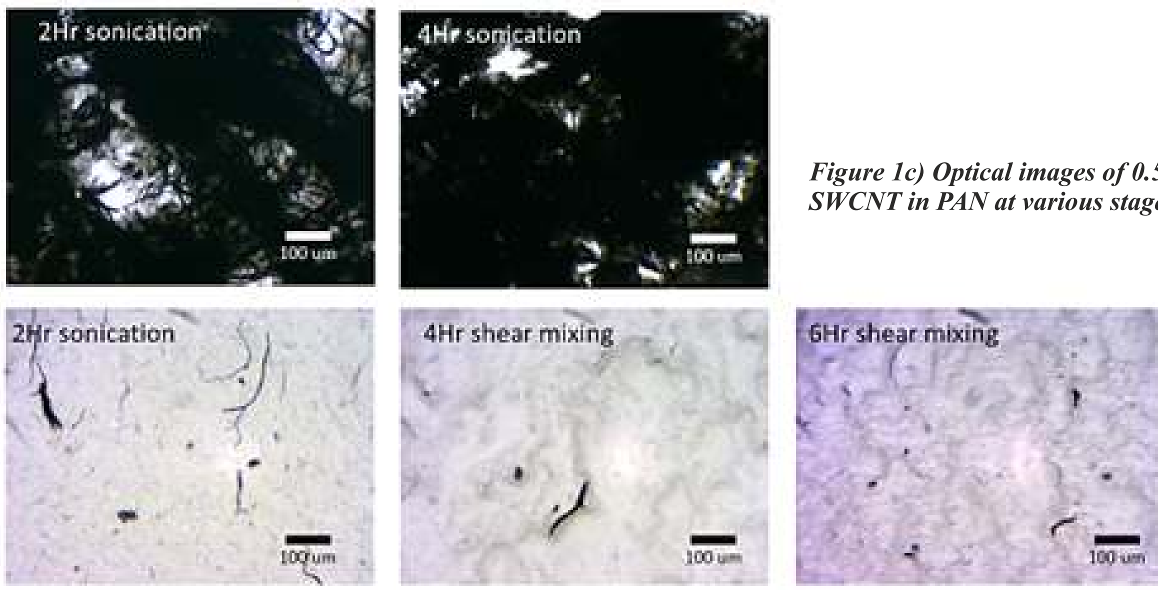

Figure 1c) Optical images of 0.5wt% SWCNT in PAN at various stages

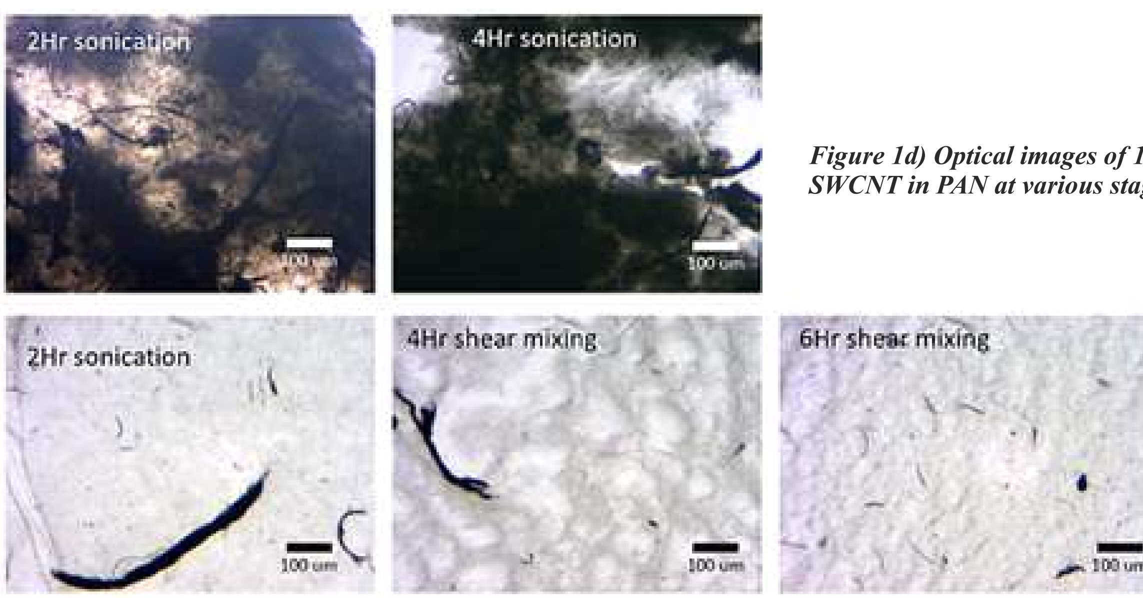

Figure 1d) Optical images of 1wt% SWCNT in PAN at various stages

Along with agglomerate size reduction and agglomerate size gives more information on the dispersion of the SWCNT in the PAN matrix. Agglomerate size distribution here is doing the same work as polydispersity index in the case of polymer. Agglomerate size distribution represents the presence of different sizes of SWCNT agglomerates present in the PAN matrix where its value closure to 1 indicates the presence of all similar sizes of agglomerates in SWCNT/PAN. Calculation of both agglomerate size and agglomerate size distribution is done as per given below equation 1, equation 2, and equation 3:

- Number average Agglomerate size (An):

$$An = \frac{\sum_n^i Ni\ Ai}{\sum_n^i Ni} \ldots(1)$$

- Size average agglomerate size (As):

$$As = \frac{\sum_n^i Ni\ Ai^2}{\sum_n^i NiAi} \ldots(2)$$

- Agglomerate size distribution = $\frac{As}{An} \ldots(3)$

Where N is the number of agglomerate particles

A is the size of respective agglomerate particles

With optimized sonication and shear mixing, all PAN/SWCNT composites attained~1 Agglomerate size distribution which indicates homogeneous network formed by SWCNT and also represents PAN behavior as a kinetic stabilizer for SWCNT (as shown in Figure 2).

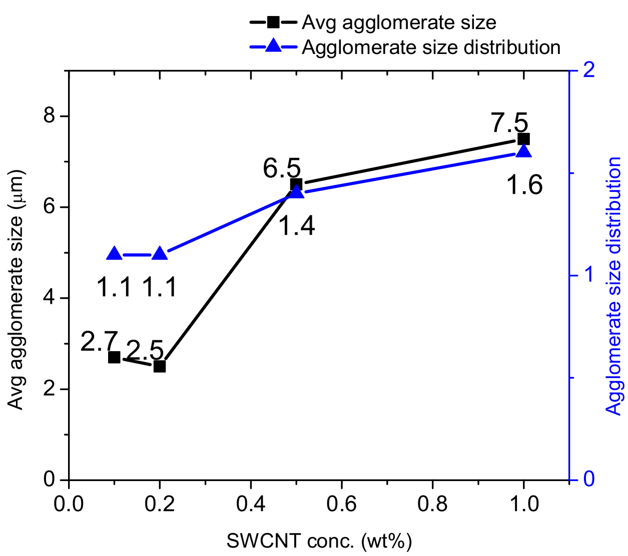

Figure 2: Effect of SWCNT concentration on the agglomerate size reduction and agglomerate size distribution after 4 hours ultrasonication and 6 hours shear mixing.

Though uniform dispersion of SWCNT bundles was achieved with the combination of ultra-sonication and shear mixing agglomerate size was found to be increasing with SWCNT concentration in PAN matrix. The smaller agglomerate size in C1 PAN/SWCNT composite can be attributed to the low concentration i.e., 0.1 wt% of SWCNT, since a greater number of PAN polymer chains were present to interact with SWCNT and making more favorable conditions in preventing SWCNT aggregation and de-bundling.

4.2 Brookfield viscosity

Brookfield viscosity is the resistance to flow of a polymer solution under shear rate which is directly related to the molecular weight, degree of entanglement, and interactions between polymer chains. Figure 3 represents the Brookfield viscosity data PAN and PAN/SWNCT composite solution.

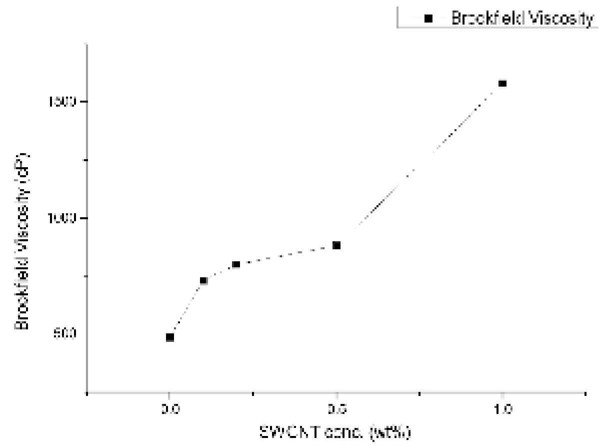

Figure 3: Effect of SWCNT concentration on Brookfield viscosity with images showing opaqueness of films prepared with respective PAN/SWCNT composite solution

As shown in the figure with increasing the SWCNT in PAN matric films are becoming opaque and also the viscosity of the PAN/SWCNT solution is increasing with SWCNT content. This result also conforms with the optical microscopy results which represent uniform dispersion of SCWNT in PAN matrix. The drastic increase in viscosity was observed for 0.1 wt% and 1wt% SWCNT addition which represents the uniform dispersion of SWCNT was stabilized by PAN polymer chains where they are interacting and wrapping the SWCNT and preventing its aggregation. Further, it also shows PAN polymer chains also act as a stabilizing agent which is reducing the surface cohesion energy of SWCNT agglomerates. Table 1 represents the detailed data of Brookfield viscosity of respective PAN and PAN/SWCNT composite solutions.

Table 1: Brookfield viscosity of PAN and PAN/SWCNT composite solution

S. No	15% PAN Solution (ml)	CNT Conc. (%)	Brookfield Viscosity (cP) at 35°C
1	200	0	485
2	200	0.10%	730
3	200	0.20%	800
4	200	0.50%	880
5	200	1.00%	1580

4.3 Solvent resistance test

A solvent resistance test was done to study the interaction between PAN and SWCNT. All the composite films i.e., pristine PAN as well as PAN/SWCNT films were kept in Dimethylformamide (DMF) solvent for 1 hr at the controlled temperature of 25°C. We observed DMF acted as a strong solvent for completely dissolving PAN polymer, however, PAN/SWCNT composite showed resistance towards the complete dissolution in DMF solvent. It can be related to the strong interactions between PAN polymer chains and SWCNT. A similar effect can be seen when SWCNT concentration in PAN increased from 0.1 wt% to 1wt% where an increase in SWCNT concentration residual weight after the test period was observed increasing in PAN/SWNCT composites films (as shown in Figure 4 and Table 2).

Figure 4: PAN and PAN/SWCNT dried composite films in DMF, (left to right): PAN, 0.1wt%, 0.2wt%, 0.5wt% and1wt% SWCNT respectively

Table 2: Solvent resistance test

S. no.	Sample	SWCNT conc. (wt%)	DMF resistance Before wt (mg)	After wt (mg)	wt loss (%)
1	PAN	0	102.1	0	100.00
2	C1	0.1	127.5	96.3	24.47
3	C2	0.2	115.5	90.8	21.39
4	C3	0.5	125.6	106.8	14.97
5	C4	1.0	113.5	100.8	11.19

As indicated from the optical microscopy and Brookfield viscosity, the uniformly formed network of SWCNT in PAN is not only restricting the penetration of DMF in composite films but also keeping PAN polymer chains strongly attached on the SWCNT surface. This effect is said to be very prominent on just the initial introduction of carbon nanotube in the PAN matrix.

5. Conclusion:

Dispersion of SWCNT in PAN matric was successfully obtained from ultrasonication and mechanical stirring. Optical microscopy results show mechanical stirring aids in reducing the agglomerate size by more than 10 times. Though the near 1 value of agglomerate size distribution was obtained in all PAN/SWCNT composite solutions agglomerate size increased with an increase in SWCNT concentration. Brookfield viscosity and physical images of PAN/SWCNT solution also show uniform dispersion of SWNCT in the PAN matrix. Solvent resistance test and drastic increase in Brookfield viscosity show good interaction between PAN polymer chains and SWCNT.

Acknowledgment

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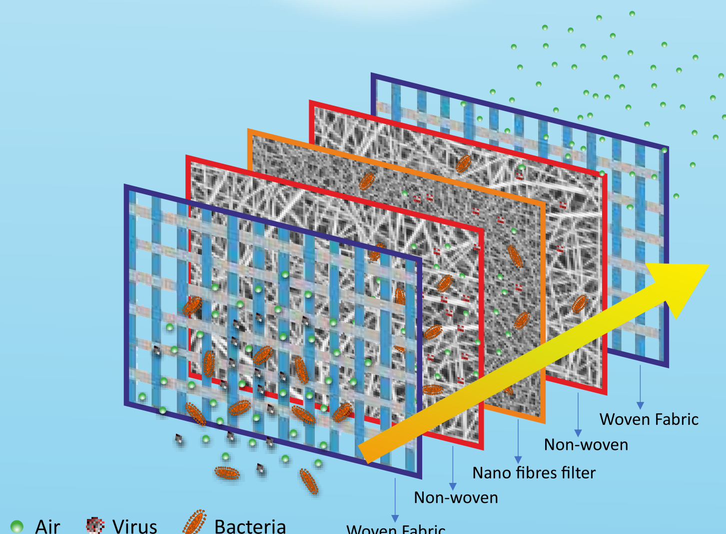

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MAKE IN INDIA

Highly Protective Reusable Face Mask – Designing and Development

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Abstract

Wearing a face mask is a compulsory requirement for today's lifestyle due to Covid -19 pandemic. The non-woven based disposable protective face is available at a low cost; however, they have comfort issues while wearing (fibre formation) and disposal problems. The fabric-based mask available in the market does not guarantee protection. Roughly face masks are categorised into three categories as fabric mask, surgical mask and respirators. Fabric mask provides less protection on the other hand surgical mask and respirators gives good protection but are not recommended for common people. Hence, we at BTRA have designed and developed unique cotton-based 5layers highly protective face masks with improved comfort, breathability, and above 95% bacterial filtration efficiency. BTRA developed mask gives very good protection, is comfortable to use and is recommended to use by common people. The mask is tested as per the requirements of Surgical Face Mask IS 16289 class 3 and passed all test requirements. Further, the wash durability to normal home laundering is also studied.

Keywords

Face Mask, Covid-19, comfort, disposable, breathability, durability, bacterial filtration efficiency

1.0 Introduction:

As a consequence of the Covid-19 pandemic, it is mandatory for the wearer a protective face mask. The government of India has also imposed strict regulations for use of face masks in public areas to prevent the spread of covid-19. Therefore, everyone must buy it and hence the demand for face covering is boomed. Disposable protective masks (N95 and similar) are the most commonly used protective mask, however, suffocation due to the synthetic fibres and fibre formation inside the mask makes it very uncomfortable to use and disposal of such used masks is another big issue [1,2]. Therefore, some manufacture grabbed this opportunity to make reusable and washable face masks however their efficacy is unknown. Hence, it is important to develop a washable, reusable mask with reliable protection and comfort properties [3].

Reusable cotton face masks are preferred over disposable non-woven masks due to their comfort. However, the fabric used for the mask is normally open weave with large pores size. The porous nature of textiles means that viruses and bacteria can be trapped within the fabric structure, which possibly lowers the risk of the viruses being transferred. On the other hand, the size of bacteria, microbes and viruses are in the range of 0.012 to 0.5 microns. However, the pore size of the fabric is much larger than the pathogens and they can easily pass through the fabric pores. Reduction of the pore size without affecting the breathing comfort is necessary to improve the efficacy of the mask. The use of nanofibre membrane as a filter may help in solving the problem and improve filtration efficiency [4]. Hence, in this work, we have used the nano fibre-based membrane filter to reduce the pore size and improve the bacterial filtration efficiency. This work aimed to develop the washable, reusable, comfortable and highly protective face mask.

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2. Materials & Methods

2.1. Materials:

100% Cotton fabric with 95 GSM-having 96 ends per inches and 62 picks per inches with warp and weft count of 40Nes and 34Nes respectively was procured from India mart supplier. Polypropylene spun bond nonwoven with 30 GSM was used. Elastic, nose wire and elastic adjuster were procured for the local market.

2.2. Methods:

2.2.1. Electrospinning

The measured amount of acetic acid and formic acid in the required ratio was taken in a conical flask and stirred using a magnetic stirrer. The polymer was added slowly during stirring and kept for 2h. The needleless electrospinning machine from ELMARCO (NS IS500 U) with wire electrode was used for the nanofiber spinning. Electrospinning parameters such as concentration of polymer, positive electrode voltage, negative electrode voltage, the distance between the electrode and relative humidity were standardized. Morphology and diameter of Nylon 6 nanofibers were observed by Scanning Electron Microscope (SEM JEOL JSM 5400). Quanta chrome's 3G porometer operating under windows ® the 3G win software was used for the analysis of pore size. Nanofiber layer spun at optimised parameters was used to reduce the pore size of the designed mask.

2.2.2. Mask design:

The designing of the protective mask was the critical part. Considering the different patterns available and drawbacks of the theme, we have designed a uniquely comfortable and proper fitting mask in four different sizes as shown in table 1.

Table 1. Different sizes as per weight groups

Sr. No	Weight (kg)	Size	Dimensions	
			length	Width
1	10-20	XS	19	10
2	21-40	S	21	12
3	41-65	M	23	15
4	65+	L	25	16.5

Mask should be worn in such a way that the nose and mouth should be covered fully, there should be minimum leakages from the sides and it should stay at the proper place. Figure 1 shows the correct methods to take the measurements for the proper fitting mask. The upper edge of the mask should be a little below the eye to provide clear vision and 0.5 to 1inch under the chin. The horizontal length of the mask must cover the full mouth and be 1 inch away from the ear.

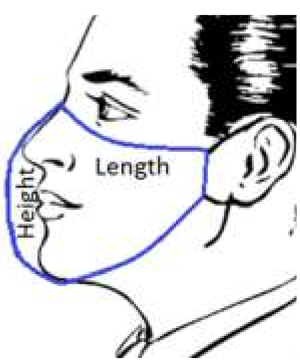

Figure 1. Measurement of proper fitting face mask.

One more important point to be noted is that the mask should not have a through cut at the centre to provide maximum protection. The mask having though out cut at the centre, stitching line may create pinholes at the centre through which the bacteria and viruses can penetrate though the mask directly near the nose and purpose of wearing a protective mask can diminish. Therefore, our mask does not have a throughout cut at the centre. The pattern design is shown in figure 2.

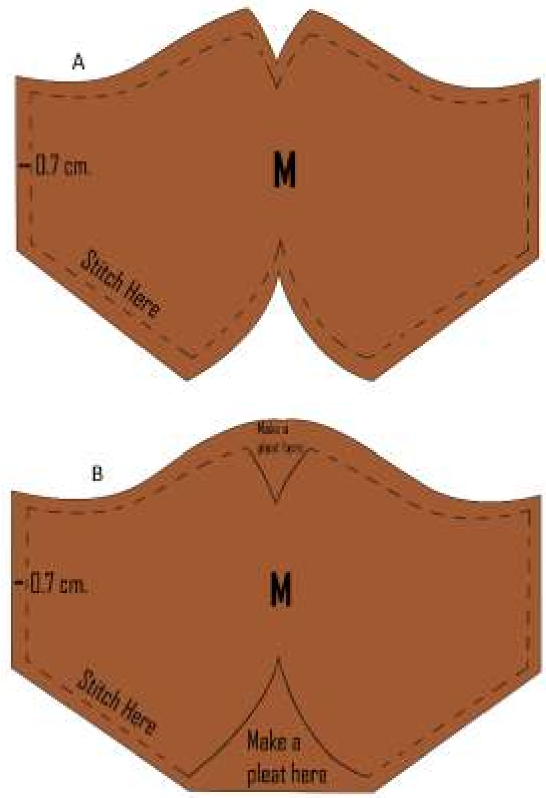

Figure 2 Master pattern of the mask – A-for fabric cutting, B- for nonwoven filter cutting

3. Characterisations:

Performance characterisation: Good performance of the protective face mask is one of the most important requirements. There are two Indian Standards (IS) wherein requirement criteria for the protective mask is given. Specification of the mask as per two different IS are listed in table 2.

It may be noticed from Table 2 that, bacterial filtration efficiency (BFE) and Breathability as differential pressure and breathing resistance are the most important parameters for surgical masks and respirators. Hence in our study, we have studied both the parameters for our developed mask.

Table 2. Specification of the mask as per IS 16289 and IS 9473

Parameters	Surgical Face Mask IS 16289			Respiratory protective devices IS 9473		
	Class 1	Class2	Class3	FFP 1	FFP 2	FFP 3
Bacterial filtration efficiency	95	98	98	--	--	--
Differential pressure @8L/min, pa	29.4 pa	29.4 pa	49.0pa			
Breathing Resistance @95l/min, mbar				2.1	2.4	3.0
Splash resistance	--	--	120	--	--	--
Sub-micron particulate filtration efficiency	--	--	98% @ 0.1µ	--	--	95% @ 0.3µ
Leakage	--	--	--	<25%	<11%	<5%
Penetration- Paraffin oil	--	--	--	NA	2%	1%
Flammability	--	--	--	Yes	Yes	Yes

3.1 Differential pressure:

Differential pressure test of the samples was performed according to IS 16289 as prescribed in annexe C at an airflow rate of 8 L/min. Five readings were taken from five different mask specimens and the average reading was recorded differential pressure value of the mask.

3.2 Bacterial filtration efficiency:

BFE of the BTRA developed face mask samples was performed as per the ASTM F-2101. The test samples were challenged to Staphylococcus aureus bacteria with a mean aerosol particle size of 3.0 ± 0.3 micron with a flow rate of 28.5 L/min. the bacterial aerosol passed through the mask was collected on the Tryptic soya agar and incubated for 24hr at 37°C. The growth of the bacteria was counted as several CFU and the percentage of the BFE was calculated. Similarly, the BFE of the washed samples was also studied after 5 and 10 items of washing.

3.3 Fabric characterisation:

All the cotton fabrics were subjected to colour fastness to washing, light and rubbing tests using respective IS standards.

4. Results and discussions:

4.1. Differential pressure:

Differential pressure (DP) essentially measures the difference in pressure between two given points. When considering the DP of any mask it measures the resistance created by the mask at constant airflow. Lowering the resistance better is the breathability and the mask will provide more comfort. We have measured the DP of our samples at an airflow rate of 8L/min as per IS 16289. We received the DP value of 55.5pa/cm2 which is a little higher than that of the standard requirement as shown in table 2 for the class 3 mask. However, we have also compared our face mask with various N95 masks available in the market and found that the DP values of the commercial N95 mask samples are very high than that of our developed samples.

Table 3 Differential pressure of the samples at 8L/min airflow

Sample Name	Differential pressure (pa/cm^2)
Fabric only single layers	6.12
Nonwoven filter	26.9
Mask	55.5

Table 3 shows the DP of various components of the mask. It may be noticed that the DP fabric is the result of its weaving pattern and cover factor by changing the weaving structure we can further reduce the DP of the mask and meet the requirement of 49 pa/cm2 for class 3 surgical masks.

4.2 Bacterial filtration efficiency:

BFE of the unwashed and washed samples is depicted in figure 3. It can be seen that initially before washing the BFE was 98%. This indicates that there is only 2% of bacterise can pass through the mask and the remaining 98% are filtered out. This high BFE can be attributed to the nano-membrane filter used as the middle layer of the mask. Nano membrane effectively works to reduce the pore size of the filter media without affecting breathability. This meets the requirement of IS 16289 class 3 masks. Further, after washing up to 10 washes the BFE is not reduced and can provide 98.5% bacterial protection.

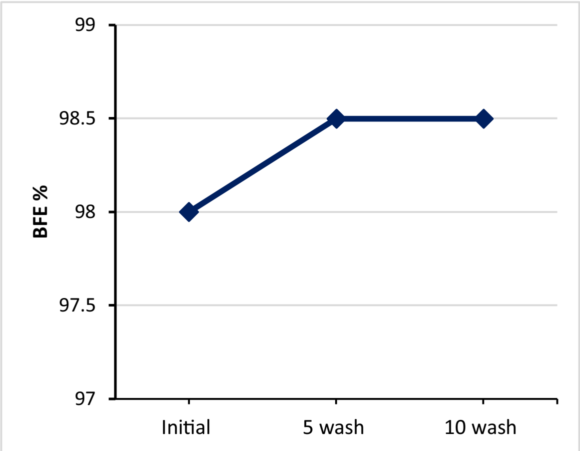

Figure 3 BFE of the mask samples up to 10 wash

4.3 Fabric characterisation:

The cotton fabrics used for the outer and inner layer of the mask was subjected to colour fastness to washing at 60°C for 30 min as per IS standard 105 C10 test method. The results and given in table 4. As can be seen from Table 4 that all the samples have goo colour fastness to washing. Similarly, colour fastness to natural sunlight was tested by accelerated test using IS 105 B02 standard method. It was found that all the samples have good colour fastness to light rating. Further, it can be seen from the table that only the black sample has poor colour fastness to wet rubbing.

Dimensional changes after washing were also analysed and reported in table 4 as % shrinkage.

Table 4 colour fastness properties of the mask samples

Fabric colour	Colour fastness				Shrinkage (%)	
	Washing	Light	Rubbing			
			Dry	Wet	warp	weft
White	NA	5	NA	NA	6.4	2.1
Black	4-5	5	4-5	2	5.4	2.6
Navy blue	4-5	5	4-5	3	5.1	6.8
Olive green	4-5	4	4-5	4	6.0	1.3
Mustered yellow	4-5	4	4-5	4	4.6	4.8
Purple	4-5	4	4-5	3	5.2	5.6

4.4 Design and comfort:

The design of the mask is made in such a way that it should meet the specifications of both the standards and provide comfort to the wearer. Cotton fabric, polypropylene nonwoven and nanofibre membrane are arranged in five different layers to make a protective mask. Adjustable nose chip provided at the top centre of the mask ensure the proper fitting and keep the mask at the proper position, eliminating the leakages and reducing the fogging effect. Further, elastic with little wider width (8mm) reduce the tress on the ear and an elastic adjuster makes it more comfortable to fit as per the choice of the individual.

The design and comfort of any face mask is the subject matter of an individual and hence cannot be tested quantitatively using any instrumental techniques. Therefore, the design and comfort of the mask were evaluated through the feedback of users. Total 50 reviews from different users of 25 to 55 age groups were analysed and found that more than 95% of the users are happy with the comfort that is the breathability of the mask and outer fabric feel. More than 70% are happy with the colours, print and look of the mask. It is well understood that the colour, print and design is the individual's choice and I defer from person to person. Further, a few people were unhappy with the size of the mask. This issue can be resolved by choosing the proper size as suggested in table 1. Overall, it was concluded from the feedback that, the BTRA developed mask provides comfort and also comply with the requirements of the standard.

5. Conclusions:

We at BREA have developed a highly protective face mask. Differential pressure of the mask was found to be 55.5 pa/cm2 which gives better breathing comfort. 98% BFE provides very good protection against bacterise. The mask passes the requirement of IS 16289 class 3 surgical masks. Front and back cotton fabric is skin-friendly and absorbed more sweat. The adjustable nose clip avoids the fogging on specs. The ear loop adjuster provides more comfort by reducing the stress on-ear.

Acknowledgements:

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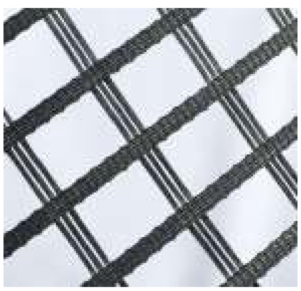

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