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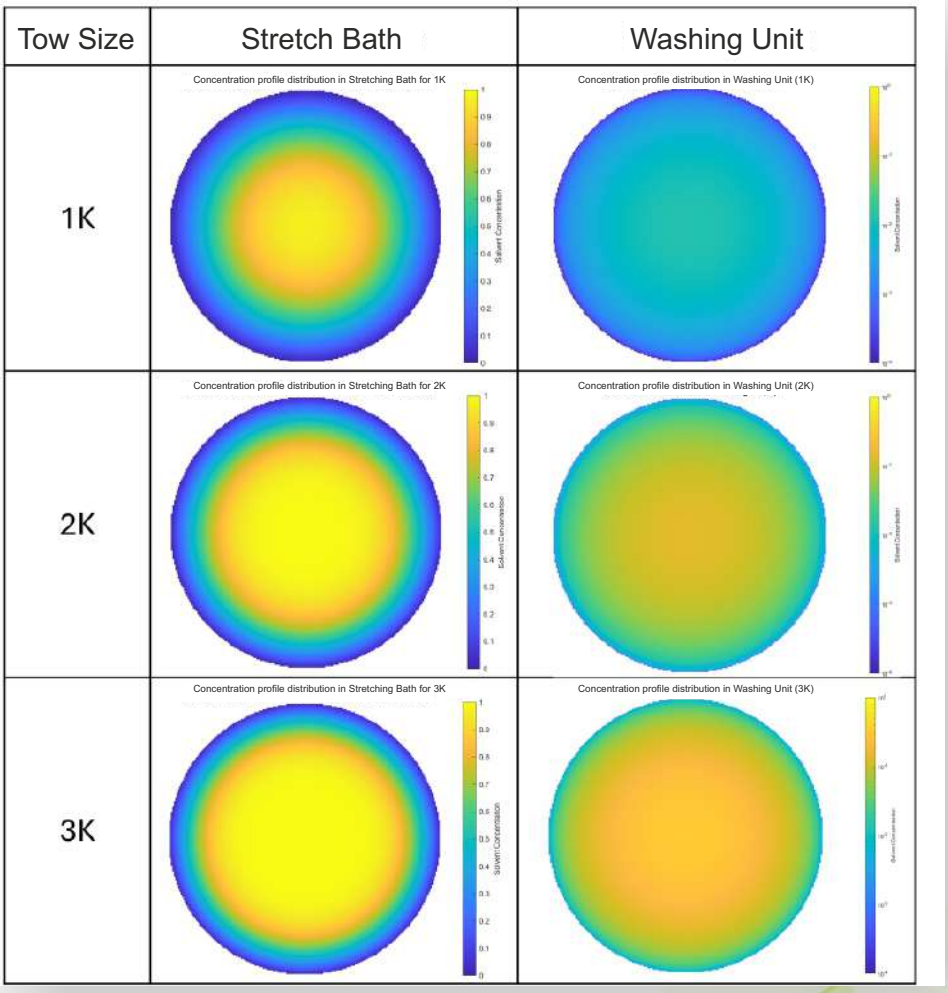

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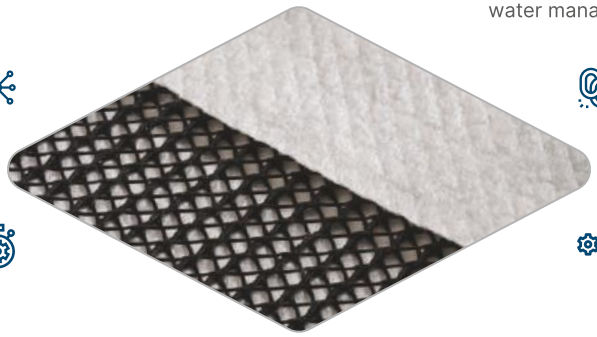

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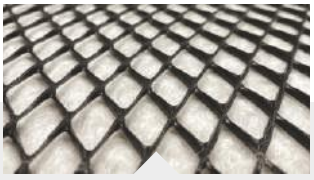

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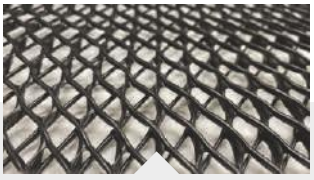

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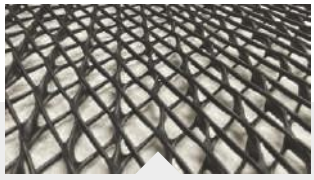

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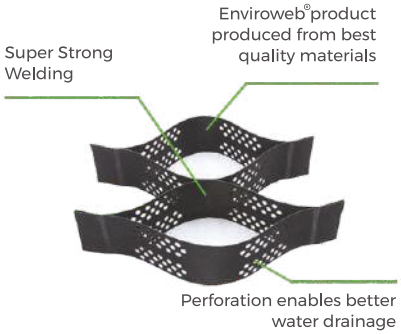

+91 76079 00000,
+91 96216 60000,
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envirogeosystems@gmail.com
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A-6/1A/7-8 AT Kursi Industrial Area,
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EDITOR'S DESK

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. We are working hard to help the journal in climbing up the ranking ladder. In continuation to this effort, I am delighted to present to our readers the 4th issue of 54th Edition of BTRA SCAN.

This issue has 3 papers from the different domains such as dvances in Spinning Techniques for High-Performance PAN-Based Carbon Fibers, Development of Alkaline-Resistant Polyester via PVA-Based Coating for Geosynthetic Applications and Effect of tow size on solvent extraction from PAN fibers during wet spinning. 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.

I thank my entire publishing team for all their support. Together we would work towards making the journal a truly influential publication. Comments and suggestions are always welcome.

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

T V Sreekumar, PhD
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Advances in Spinning Techniques for High-Performance PAN-Based Carbon Fibers

Yogesh Hande
The Bombay Textile Research Association, L B S Marg, Ghatkopar (W), Mumbai 400086

Abstract

Polyacrylonitrile (PAN) fibers are essential precursors for high-performance carbon fibers, recognised for their significant carbon yield and mechanical strength. This review investigates the spinning methods of PAN fibers, focusing on wet spinning and dry-jet wet spinning, while utilising recent research to analyse their mechanisms, chemical characteristics, and results. Wet spinning facilitates controlled coagulation, producing fibers with minimal defects, whereas dry-jet wet spinning improves molecular alignment by introducing an air gap. Essential factors like polymer molecular weight, solvent selection, and coagulation bath composition significantly impact fiber characteristics. Developments such as adding various additives and electrochemical modifications enhance tensile strength and sustainability. Ongoing issues, including solvent recovery and process scalability, remain, with future efforts to incorporate eco-friendly solvents and advanced composite materials. This review consolidates insights to emphasise the best spinning conditions and new trends for producing high-performance PAN fibers.

Keywords

Polyacrylonitrile, Carbon fiber, Spinning techniques

Citation

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1.0 Introduction:

Polyacrylonitrile (PAN), a synthetic polymer mainly made up of acrylonitrile units, is the leading choice for carbon fiber precursors because of its high carbon yield (around 68%), exceptional mechanical characteristics, and its capability to develop ordered crystalline structures during production, rendering it essential for high-performance uses in sectors like aerospace, automotive, wind energy, and defence [1,2]. The origins of PAN fibers trace back to the mid-20th century when their potential as precursors for carbon fibers was identified, prompting extensive research into spinning methods to enhance fiber microstructure, tensile strength, and modulus[2]. The spinning techniques convert PAN polymer solutions into fibers via a series of steps, including extrusion, coagulation, drawing, and stabilisation, where the selected spinning method significantly affects the resulting fiber characteristics[3,4]. The primary methods for producing PAN fibers are wet and dry-jet wet spinning, each providing distinct benefits in controlling fiber structure and performance[1], [5]. Wet spinning entails the direct extrusion of a PAN solution into a coagulation bath, enabling a gradual solvent exchange that reduces defects and permits the addition of sustainable substances like lignin or nano-reinforcements such as cellulose nanocrystals(CNCs) [6,7,8]. On the other hand, dry-jet wet spinning features an air gap between the spinneret and the coagulation bath[9], which allows for initial chain alignment that improves molecular orientation and diminishes die swell, resulting in fibers with enhanced tensile properties [10,11,12]. Over the years, research has aimed at optimising essential factors like polymer molecular weight, solvent choices, coagulation bath compositions, and draw ratios to develop fibers suitable for carbon fiber precursors with high performance[13,14,15]. Recent developments have investigated novel techniques, including electrochemical modifications and nanomaterials, to improve fiber quality further and tackle environmental issues[1,16]. This review systematically compiles findings from recent research to offer a detailed understanding of the spinning processes for PAN fibers, their relative advantages, ongoing innovations, challenges, and potential future directions.

*Corresponding author,
E-mail: carbonfibre@btraindia.com

2. PAN Fiber Spinning Techniques

2.1 Wet spinning

Wet spinning is recommended for PAN fiber manufacturing because it allows for forming fibers with a regulated shape, high tensile strength, and suitability as carbon fiber precursors. The technique enables exact manipulation of fiber structure under regulated coagulation conditions, ensuring scalability and consistency for industrial applications. [4,8]The wet spinning process begins by dissolving PAN or its copolymers in a solvent to generate a spinning dope, usually at concentrations of 15-25 wt%[6]. The dope is extruded through a spinneret into a coagulation bath, where the fibers solidify as the solvent evaporates and a non-solvent, often water, diffuses in.[7] The fibers (jet stretch, wet stretch, and hot stretch) are then drawn to improve molecular alignment, washed to remove residual solvent, and dried at 100-150°C to complete the fiber structure. [1,4] Key processing factors include coagulation temperature (20-60°C), bath composition (solvent/non-solvent ratio), dope concentration, draw ratio, and spinneret design, which all have a substantial influence on fiber quality and performance.[8,17]PAN (usually 10–25 weight per cent) is dissolved in a polar organic solvent, like dimethylformamide (DMF) or dimethyl sulfoxide (DMSO), to create a spinning dope. Impurities are then removed by filtration and degassing.[1,6] Rapid phase inversion hardens the filaments when the dope is extruded through a spinneret into a coagulation bath, which frequently contains a solvent-water mixture (e.g., 50–70% DMF)[4,7]. Crucial elements that affect fiber morphology, decreasing voids and increasing tensile strength are bath mix, temperature (20–50°C), and coagulation rate.[8] The fibers undergo multi-stage drawing (draw ratio 4–12) in hot water or steam to align polymer chains, followed by washing to remove residual solvent and drying to stabilise the structure.[17] This sequence ensures high crystallinity and orientation, yielding PAN fibers with tensile strengths of 200–500 MPa, ideal for carbon fiber production.

2.1.1 Raw Material Characteristics

The production of PAN fibers through wet spinning involves specific chemicals for the spinning solution and the coagulation bath, with factors like molecular weight and concentration being essential in defining fiber quality. The core polymer is PAN or PAN copolymers, frequently including comonomers like methyl acrylate or itaconic acid, with a molecular weight ranging from 80,000 to 1,50,000 g/mol to balance strength and processability.[4,17]The dope is made at a concentration of 15-25 wt% in a solvent such as dimethyl sulfoxide (DMSO, MW: 78:13 g/mol) or dimethylacetamide (DMAc), which can effectively dissolve high-molecular-weight PAN.[6,8]To control diffusion speeds and avoid voids, the coagulation bath is commonly made up of water or a solvent-water mixture with solvent concentrations ranging from 10- 70 wt% (for example, DMSO/water ratio of 30:70 to 70:30)[1,7]

2.1.2 Influence of Processing Parameters in Wet Spinning

Wet spinning of PAN fibers showingtensile strengths ranging from 600 MPa to 7 GPa (post-carbonisation) and tensile moduli up to 130 GPa, notably for lignin/PAN blends, depending on the processing optimisation.[1,8] The most significant results are produced with high draw ratio (jet stretch: 1- 2 times, wet stretch: 2- 5times, hot stretch: 5-10times), low coagulation temperatures (20-30°C), and optimum solvent ratios, e.g., 50:50 DMSO/water, which minimise voids and increase molecular alignment. [4,7]For example, studies demonstrate that controlled coagulation and high stretching improve fiber density and mechanical properties, making them ideal for carbon fiber precursors. These optimised conditions lead to fibers with superior tensile strength and modulus, suitable for high-performance applications in aerospace and structural composites[8,17]

2.2 Dry Spinning

Unlike wet spinning, dry spinning does not require a coagulation bath, which reduces void formation and simplifies solvent recovery. It is used for PAN fiber production when the objective is to produce fine fibers with high surface quality and minimal defects, especially for applications requiring enhanced molecular orientation. For textiles and some carbon fiber precursors, dry spinning is beneficial for creating fibers with diameters between 10 and 50 µm. The dry spinning process contains multiple steps: Initially, a spinning dope is made by dissolving PAN in a volatile solvent. The fibers are then solidified by the solvent's quick evaporation in a regulated gas environment, like hot air or nitrogen, after this dope is extruded via a spinneret into a heated spinning chamber. After being drawn to increase molecular alignment, the fibers are gathered on a winder, where they may undergo optional post-treatments like washing or heat treatment to enhance their qualities. Dope temperature (70–100°C), spinneret temperature (150 - 200°C), air or nitrogen flow rate, draw ratio (2–6 times), and dope concentration (20–30 wt%) are important processing parameters that have a significant impact on fiber quality and performance[18].To create a spinning dope for PAN fibers, PAN or its copolymers must first be dissolved in a volatile solvent, like DMSO or DMF, at a concentration of 20–30 weight per cent and at a temperature of 70–100°C to get the ideal viscosity [1]. After passing through a spinneret with 0.1–0.3 mm hole sizes, the dope is extruded into a heated spinning chamber between 150 and 200°C. The fibers are solidified in this chamber by the quick evaporation of solvents made possible by hot air or nitrogen. The fibers are gathered on a winder after being drawn at ratios of 2–6 times

to align polymer chains and improve mechanical qualities. It is possible to apply optional post-treatments, such as heat treatment to enhance crystallinity or washing to remove leftover solvent. The procedure necessitates exact control over the dope temperature, spinneret temperature, air flow rate, and draw ratio to avoid flaws like unequal fiber diameters or surface roughness.[19,20]

2.2.1 Raw Material Characteristics

In contrast to wet spinning, the dry spinning method for PAN fibers uses certain chemicals for the spinning dope and does not require a coagulation bath. To guarantee adequate chain entanglement for robust fibers, the main polymer is PAN or its copolymers, which frequently include comonomers like methyl acrylate or itaconic acid. Their molecular weight is generally between 80,000 and 150,000 g/mol.[19] Because of their capacity to efficiently dissolve PAN and evaporate rapidly, volatile solvents like dimethylsulfoxide (DMSO, molecular weight: 78.13 g/mol) or dimethylformamide (DMF, molecular weight: 73.09 g/mol) are used to create the dope at a concentration of 20–30 weight per cent.[20]While DMSO is selected for its thermal durability during processing, DMF is preferred due to its lower boiling point (153°C), which allows for speedier evaporation. Additives like plasticisers, such as glycerol, or stabilisers may be used to improve dope stability or fiber flexibility.[20]For instance, one study used PAN with a molecular weight of around 100,000 g/mol in a 25 weight per cent DMF dope to maximise spinnability. Another study used a PAN copolymer with itaconic acid to enhance fiber homogeneity in a 22 weight per cent DMSO dope.[19,20]No coagulation bath is used, as solvent evaporation in the heated chamber produces fiber solidification.

2.2.2 Influence of Processing Parameters in dry spinning

Depending on the processing parameters, PAN fibers produced by dry spinning have tensile strengths between 500 and 900 MPa and tensile moduli between 10 and 15 GPa. Compared to wet-spun fibers, the absence of a coagulation bath produces denser fibers with smoother surfaces, decreasing internal voids and improving surface quality. High draw ratios of 4–6 timesoptimise tensile strength and molecular alignment, and controlled evaporation conditions, including spinneret temperatures of 160–180°C and moderate air flow rates, guarantee homogeneous solidification free of surface defects, and yield the best results. The optimal viscosity-to-fiber quality ratio is achieved at 22–25% dope concentration. As an illustration of the value of ideal evaporation and stretching conditions, one study used a 25 weight per cent dope in DMF with a 5 times draw ratio to report fibers with a modulus of 14 GPa and a tensile strength of 850 MPa[19,20].

2.3 Dry jet wet spinning

For PAN fibers, dry-jet wet spinning is used instead of traditional damp spinning to provide better mechanical characteristics and molecular alignment. By encouraging chain orientation and minimising die swell, the air gap permits the polymer solution to stretch initially before coagulation, improving fiber strength and modulus.[5,10]. High molecular weight PAN, essential for intense carbon fiber precursors, can be used with this technique, which also allows for fine coagulation control to reduce voids and surface imperfections. [11,12] The process is particularly effective for producing fibers with tailored microstructures, making it ideal for high-performance applications. [15,16].The dry-jet wet spinning process consists of several meticulously regulated steps with precise specifications to maximise fiber qualities. To create a PAN dope, PAN or its copolymers are first dissolved in 15–22% DMSO, heated to 60–80°C, and filtered to guarantee homogeneity.[5,16]. A jet stretch ratio of 0.8 to 2.0 is then used to align polymer chains after the dope is extruded via a spinneret with diameters of 0.05 to 0.2 mm into an air gap of 5 to 20 mm at 20 to 30°C.[10,11]. After extrusion, the fibers are placed in a coagulation bath at 10 to 50°C with either water or water/DMSO (30 to 70 weight per cent DMSO). The concentration of DMSO affects the coagulation time, with a larger DMSO content slowing coagulation to minimise voids.[12,15]. To improve molecular orientation, the fibers are first drawn in a coagulation bath with draw ratios of three to six times, and then they are hot-drawn in hot water or steam at 80 to 100°C with draw ratios of six to twelve times.[10,16]. The fibers are then dried under tension at 100 to 150°C after being cleaned to remove any remaining solvent, to establish the structure[11,15]. Thermal stabilisation at 200 to 300°C in air is frequently carried out to prepare the fibers for carbonisation.[5].

2.3.1 Raw Material Characteristics

Various chemicals are used in the dry-jet wet spinning process to get the required fiber qualities. The main polymer is PAN or its copolymers, which are frequently combined with vinyl acetate or itaconic acid. Their molecular weights range from 70,000 to 300,000 g/mol, where greater molecular weights enhance tensile strength and spinnability[10,11]. Because it can successfully dissolve high molecular weight PAN, dimethyl sulfoxide (DMSO) is the most widely used solvent. Dope concentrations are usually between 15 and 22 weight per cent to balance viscosity for extrusion[5,12,15].Lower DMSO concentrations speed up coagulation and affect fiber shape. The coagulation bath is made up of water or water/DMSO mixes with DMSO concentrations ranging from 30 to 70 weight per cent and bath temperatures between 10 and 50°C[10,11,15]. Additives such as carbon nanotubes (CNTs)

at 0.5 to 2 weight per cent or graphene oxide (GO) at 1 to 5 weight per cent enhance mechanical or electrical properties. By fortifying the fiber matrix, CNTs raise tensile strength[12,16]. Furthermore, adding 1–3 mol% itaconic acid to copolymers improves heat stability during subsequent carbonisation, which lowers flaws[11].

2.3.3 Influence of Processing Parameters in dry jet wet spinning

To prepare a dope for dry-jet wet spinning of PAN fibers, PAN or its copolymers—often with additions like CNTs or GO—are dissolved in DMSO at a 10 wt.% concentration of 15 to 22 and heated to 60 to 80°C to guarantee homogeneity. Before the dope enters a coagulation bath of water or water/DMSO (30 to 70 weight per cent DMSO) at 10 to 50°C, it is extruded through a spinneret into an air gap of 5 to 20 mm, where jet stretch aligns polymer chains, decreasing die swell and improving molecular orientation. Defects are reduced by the air gap and regulated coagulation, which promote fiber solidification by solvent exchange. To optimise chain alignment and crystallinity, the fibers are then drawn in stages, first in the coagulation bath at 3 to 6times and then in hot water or steam at 80 to 100°C with draw ratios of 6 to 12 times. To maintain the structure, the fibers are dried under tension at 100 to 150°C after being drawn and cleaned to remove any remaining solvent. With the air gap and additive inclusion offering improved control over microstructure, this technique creates fibers with high strength and modulus appropriate for carbon fiber precursors[10,15]. PAN fibers with tensile strengths of up to 1 GPa are produced by dry-jet wet spinning. This makes them perfect starting materials for high-performance carbon fibers in energy, automotive, and aerospace applications. Fibers with remarkable mechanical qualities and microstructural homogeneity that closely match commercial requirements are produced by incorporating carbon nanotubes (CNTs) and optimising process factors like high molecular weight PAN, controlled coagulation, and high draw ratios.[11,15].

2.4 Electrospinning

Electrospinning is utilised to produce PAN fibers because it can create nanofibers with diameters between 100 and 500 nm, providing high surface area-to-volume ratios and improved mechanical characteristics ideal for advanced applications. In contrast to traditional wet or dry spinning methods, electrospinning allows for accurate control of fiber structure. It is possible to incorporate functional additives such as graphitic carbon nitride or silica nanoparticles, improving properties like photocatalytic activity or hydrophobic characteristics.[17] This method is especially beneficial for creating lightweight, porous PAN nanofibers suitable for oil-water separation, supercapacitors, and filtration, as nanoscale designs enhance efficiency.[21,22] The procedure includes dissolving polyacrylonitrile (PAN) in a solvent, applying a high voltage to create a charged jet, and gathering solidified nanofibers on a grounded collector. Important processing factors consist of voltage (ranging from 10 to 25 kV), solution viscosity, and the distance from the collector (10 to 20 cm), all of which influence fiber diameter and consistency.[23].

2.4.1 Raw Material Characteristics

The main polymer used in electrospinning PAN fibers is PAN, which usually has a molecular weight ranging from 70,000 to 150,000 g/mol and is dissolved in dimethylformamide (DMF) at concentrations between 8 and 15 wt% to create a thick spinning dope[17,21,24]. Chen et al. employed PAN at a concentration of 10 wt% in DMF, along with polystyrene (PS) in a 1:1 ratio to create core-shell structures, but did not indicate the molecular weight of the PS[21]. Wang et al. incorporated 0.5–2 wt% of graphitic carbon nitride (g-C3N4) into a solution of 10 wt% PAN/DMF to improve photocatalytic properties[17].Sabantina et al. reported that using PAN (150,000 g/mol) at 8–12 wt% concentrations in DMF increases viscosity, hindering bead formation at higher concentrations. Yang et al. introduced poly(methyl methacrylate) (PMMA) as a sacrificial polymer at 1:1 with PAN in DMF, maintaining PAN at 8 wt%. Ramalingam et al. utilised PAN at a concentration of 12 wt% in DMF, adding carbon nanomaterials to enhance conductivity[22,23,24]. In electrospinning, a coagulation bath is not utilised because fiber solidification takes place through the evaporation of the solvent in the air, which sets it apart from all spinning techniques.

2.4.2 Electrospinning Process Parameters

The solvent-based electrospinning of PAN fibers uses electrostatic forces to generate nanofibers with well-defined morphology. The process starts with creating a uniform PAN solution, usually within a concentration of 8–15 wt% in DMF, occasionally mixed with additives like g-C_3N_4, PS, or PMMA to improve the functional characteristics of the resulting fibers [17,21,23]. This mixture is placed into a syringe fitted with a needle diameter of 0.5 to 1 mm. A high voltage, typically ranging from 10 to 25 kV, is applied to the needle's tip, creating a Taylor cone that triggers the release of a charged polymer jet[21,24]. The jet experiences stretching and whipping motions caused by electrostatic repulsion and the evaporation of the solvent, which results in the solidification of fibers. These nanofibers are gathered as a nonwoven mat on a grounded collector, like aluminium foil or a rotating drum, placed 10–20 cm away from the needle[17,23]. Research on process optimisation has revealed several variations: Chen et al. employed a

voltage of 12 kV, a distance of 15 cm, and a flow rate of 1 mL/h to produce uniform core-shell fibers [21]. Wang et al. indicated using 15 kV at a distance of 12 cm for fibers modified with g-C_3N_4[17]Sabantina et al. attained stable fibers by applying 18 kV, maintaining a distance of 15 cm, and a flow rate of 0.5 mL/h[24] Yang et al. employed a voltage of 20 kV and a distance of 18 cm for carbonised nanofibers; in contrast, Ramalingam et al. refined their parameters to 18 kV, 15 cm, and a flow rate of 0.8 mL/h for energy storage applications[22,23]. Environmental factors, usually within a 25–30°C range and 30–50% humidity, ensure fiber consistency and stability during processing[24]. After electrospinning, the gathered fibers typically undergo thermal stabilisation at temperatures ranging from 250 to 300°C, followed by carbonisation between 800 and 1200°C. This process enhances mechanical strength and electrical conductivity, especially for carbon fiber applications [22,23]. The electrospinning technique provides scalability, exact control over fiber structure, and adaptability in functionalisation, which has led to its widespread use for producing advanced PAN-based nanofibers.

2.4.3 Influence of Processing Parameters in Electrospinning

Electrospinning polyacrylonitrile (PAN) produces nanofibers ranging from 100 to 500 nm in diameter, characterised by high porosity and specific surface areas that can reach up to 500 m^2/g, influenced by various processing conditions and additives. According to Chen et al., core-shell PAN/PS nanofibers exhibit superhydrophobic characteristics (with a contact angle exceeding 150°) and significant oil absorption capacity (up to 50 g/g), making them suitable for oil-water separation, which was achieved at 12 kV with a 15 cm distance from the collector.[21]. Wang et al. demonstrated that incorporating 1 wt% g-C3N4 into PAN nanofibers improved the photocatalytic breakdown of dyes by 80% when exposed to visible light, resulting in uniform fibers with a diameter of 200 nm produced at 15 kV[25].Sabantina et al. successfully produced stabilised PAN nanofibers that exhibited tensile strengths ranging from 5 to 10 MPa post-carbonisation, with the best results observed at a 10 wt% PAN concentration to reduce the occurrence of bead defects.[24]Yang et al. described carbonised PAN nanofibers possessing a specific surface area of 400 m^2/g, which are appropriate for catalysis, created from a 1:1 blend of PAN and PMMA at a voltage of 20 kV[23]. Ramalingam and colleagues achieved carbon nanofibers that exhibited a specific capacitance of 200 F/g for use in supercapacitors, optimised at 12 wt% PAN and 18 kV[22]. Optimal outcomes are generally obtained with PAN concentrations ranging from 10% to 12% by weight, voltages between 12 kV and 18 kV, and adding additives to customise functionality.[21,22].

3. Comparative analysis

Wet spinning, dry-jet wet spinning, electrospinning, and dry spinning each have unique benefits and drawbacks for producing PAN fibers, especially when considering fibers free of additives, with their appropriateness based on the specific application intended. Wet spinning enables direct extrusion into a coagulation bath (20–50 wt% DMSO, 20–40°C), facilitating controlled coagulation, resulting in uniform microscale fibers (10–20 µm diameter) that exhibit tensile strengths ranging from 0.6 to 2.5 GPa and moduli between 10 and 15 GPa. Still, it may lead to void formation if coagulation occurs too swiftly, necessitating careful management of bath composition and temperature to reduce defects[1,7,21]. The straightforward nature of this process renders it cost-effective for generating fibers intended for general carbon fiber precursor usage. However, its mechanical properties fall short compared to those achieved through dry-jet wet spinning due to less effective chain alignment[4]. Dry-jet wet spinning, characterised by an air gap (5–20 mm) and a coagulation bath (30–70 wt% DMSO, 10–50°C), improves molecular alignment, producing microscale fibers (8–15 µm diameter) with tensile strengths of 0.8–2.8 GPa and moduli ranging from 12 to 20 GPa, attributed to enhanced crystallinity (70–80%)[5,10,15]. Nevertheless, its intricate design and the energy demands associated with the air gap and high draw ratios (up to 12 times) create challenges for scalability[11,12]. Electrospinning generates nanoscale fibers (50–500 nm diameter) with tensile strengths between 0.1 and 0.5 GPa and moduli from 5 to 10 GPa, making them suitable for filtration or biomedical scaffolds due to their extensive surface area. Still, the lower strength and the complicated setup (high voltage and precise flow management) restrict their application in carbon fiber precursors[24,26]. Dry spinning operates by evaporating solvent in a heated chamber (100–200°C), resulting in microscale fibers (15–25 µm diameter) with tensile strengths of 0.5–1.5 GPa and moduli between 8 and 12 GPa, providing simplicity but less control over microstructure, which leads to inferior mechanical properties and increased porosity when compared to wet or dry-jet wet spinning[26]. Wet spinning and dry-jet wet spinning are favoured for producing high-performance carbon fiber precursors, with dry-jet wet spinning demonstrating enhanced strength and modulus. In contrast, electrospinning and dry spinning are more appropriate for specialised or economical applications[1,10,24,26]. A summary of the essential parameters and results without additives is included in the table below.

Parameter	Wet Spinning	Dry-Jet Wet Spinning	Electrospinning	Dry Spinning	References
Molecular Weight (g/mol)	80,000–250,000	70,000–300,000	100,000–200,000	80,000–200,000	[10, 11, 13, 14, 24, 26,]
Dope Concentration (wt%)	15–25 in DMSO	15–22 in DMSO	8–12 in DMSO/DMF	20–30 in DMSO/DMF	[1, 5, 7, 10, 11, 24]
Coagulation/ Evaporation	Water or 20–50 wt% DMSO, 20–40°C	Water or 30–70 wt% DMSO, 10–50°C, air gap 5–20 mm	Air, 20–30°C, 10–30 kV, 10–20 cm distance	Heated chamber, 100–200°C	[1,5,7,10,12,24]
Jet Stretch/Flow Rate	Jet stretch: 0.5–2.0	Jet stretch: 0.8–2.0	Flow rate: 0.5–2 ml/h	None	[1,10,11,24]
Draw Ratios	Wet: 2–5, Hot: 5–10 times	First: 3–6 times, Second: 6–12 times	None (post-drawing optional, 1–3 times)	3–8 times	[4,10,15,21, 24]
Fiber Diameter (µm)	10–20	8–15	0.05–0.5 (50–500 nm)	15–25	[1,10,24,26]
Tensile Strength (GPa)	0.6–2.5	0.8–2.8	0.1–0.5	0.5–1.5	[1,7,10,11, 24]
Tensile Modulus (GPa)	10–15	12–20	5–10	8–12	[1,7,10,11,24]
Crystallinity (%)	60–80	70–80	50–70	55–75	[1,10,12,24,26]

4. Challenges and future directions

Pinning PAN fibers encounters numerous obstacles restricting scalability and sustainability across all methods. The tendency for voids to form in wet spinning at elevated coagulation rates necessitates careful management of bath temperature (20–40°C) and DMSO concentration (20–50 wt%), complicating the process.[1,7,21]. Dry-jet wet spinning faces scalability issues due to its energy-demanding air gap (5–20 mm) and high draw ratios (up to 12times), with maintaining a consistent air gap length being crucial to prevent filament breakage.[10,12,15]. The low throughput and high-voltage needs (10–30 kV) of electrospinning limit its scalability for carbon fiber production, and achieving homogeneous nanoscale fibersremains challenging.[24]. The high-temperature evaporation (100–200°C) required in dry spinning leads to increased porosity and diminished mechanical properties, indicating a need for improved chamber designs.[26]. The dependence on DMSO or DMF in all methods raises environmental and cost issues, with current solvent recovery systems proving ineffective.[20,24]. Future efforts should focus on creating environmentally friendly solvents, such as ionic liquids or aqueous solutions, to substitute DMSO and DMF and enhance solvent recovery technologies to promote sustainability.[24,26]. Investigating process optimisation, including automated regulation of coagulation or evaporation parameters, could enhance scalability and consistency.[10,19]. Progress in stabilisation methods, particularly for fibers produced through electrospinning and dry spinning, could improve their viability as precursors for carbon fibers[24], [26]. Addressing these challenges will be essential for increasing the production of high-performance, sustainable PAN fibers[20,22,26].

5. Conclusion

Spinning of PAN fibers, which includes techniques such as wet spinning, dry-jet wet spinning, electrospinning, and dry spinning, is crucial for creating high-performance carbon fiber precursors. Wet spinning provides a straightforward and consistent method, achieving strengths between 0.6 and 2.5 GPa, while dry-jet wet spinning offers enhanced mechanical properties, ranging from 0.8 to 2.8 GPa. Electrospinning facilitates the formation of nanoscale fibers, although these typically possess lower strengths of 0.1 to 0.5 GPa. Conversely, dry spinning is a more cost-effective, yielding fibers with strengths between 0.5 and 1.5 GPa. The ideal production conditions involve using high molecular weight PAN, specifically 250,000–300,000 g/mol for wet and dry-jet wet spinning, and 100,000–200,000 g/mol for electrospinning and dry spinning. For wet-based methods, a dope concentration of 15–25 wt% DMSO is recommended, alongside regulated coagulation or evaporation processes. Nonetheless, issues such as solvent recovery, intricate procedures, and scalability remain challenges. However, advances in research focusing on environmentally friendly solvents and automated methods are expected to improve sustainability and performance. By overcoming these obstacles, PAN fiber spinning stands to satisfy the increasing demand for high-performance, sustainable carbon fibers in aerospace, automotive, and renewable energy sectors.

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Development of Alkaline-Resistant Polyester via PVA-Based Coating for Geosynthetic Applications

Shyambabu K. Sainik, Prasanta K. Panda*
The Bombay Textile Research Association, L B S Marg, Ghatkopar (W), Mumbai 400086

Abstract

Polyester-based geosynthetics are widely employed in civil engineering applications owing to their high mechanical performance and cost-effectiveness. However, exposure to aggressive alkaline environments, such as high-pH soils and groundwater, significantly reduces their long-term durability. In this study, polyvinyl alcohol (PVA) was employed as a coating material for polyester yarns due to its intrinsic alkaline resistance and water solubility, reducing the need for organic solvents and enhancing environmental sustainability. To improve water resistance and mechanical stability, PVA was crosslinked using crosslinkers,. The crosslinked coating was applied to polyester yarns via a padding mangle machine and dried under controlled conditions. The resulting materials demonstrated excellent alkali resistance up to pH 12.5 at 50°C, with negligible physical changes after prolonged exposure. Gravimetric analyses, including GSM determination, confirmed uniform coating deposition and mechanical reinforcement. This work provides a cost-effective, scalable, and environmentally sustainable strategy to extend the functional lifespan of polyester geosynthetics in harsh environments.

Key words:
Alkaline resistant Polyester, Crosslinking, Geosynthetics, Polyvinyl alcohol, and Tensile strength

Citation
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1. Introduction:

Polyester-based geosynthetics are extensively utilized in civil engineering owing to their outstanding mechanical strength and cost efficiency. However, their vulnerability to alkaline hydrolysis in harsh environments, such as soils and groundwater with high pH greatly restricts their long-term durability. In this study, a surface coating approach is explored to enhance the alkaline resistance of polyester materials. Coating with alkaline-resistant polymers was identified as a practical and scalable technique that effectively improves chemical stability without requiring extensive energy input or complex processing steps. This method offers a cost-efficient and industrially adaptable solution for improving the long-term durability of polyester-based materials exposed to harsh alkaline environments.

Polyvinyl alcohol (PVA) selected as the coating material due to its inherent alkaline resistance and water solubility, which minimizes the need for organic solvents and promotes environmental sustainability. PVA is a versatile synthetic polymer with a carbon–carbon (C–C) backbone and multiple hydroxyl (-OH) groups attached as side chains. These hydroxyl groups impart distinctive characteristics, including water solubility [1]. Owing to its solubility property PVA has found increasing use in packaging and other industries [2]. In addition, PVA offers advantages like excellent film-forming capability, strong adhesion, and high thermal stability [3]. PVA appears as a white, odorless, tasteless powder known for its superior film-forming properties. It is used across diverse sectors, including paper, textiles, adhesives, coatings, films and packaging, pharmaceuticals, construction, and cosmetics [4]. While PVA is water-soluble and exhibits a high degree of crystallinity, this property can limit its performance in humid conditions or in applications requiring water resistance. To overcome this limitation, several crosslinking agents such as cyanuric acid, citric acid, formaldehyde, glutaraldehyde, glyoxal, acetic acid, and Maleic acid are commonly used. However, without adequate modification or plasticization, crosslinked PVA films may become brittle, highlighting the need for optimization, particularly in coating applications.

To address this, a novel method has been developed that achieves effective crosslinking within few minutes. This innovation enhances process efficiency and broadens the applicability of PVA across industries that demand rapid

*Corresponding author,
E-mail: nanolab@btraindia.com

processing. The newly developed coating technique optimizes reaction conditions to achieve rapid crosslinking while preserving or enhancing PVA's mechanical strength and stability. This advancement offers a promising, sustainable, and efficient solution for industries utilizing PVA-based coatings.

2. Materials and Methods

2.1 Materials

PVA (Molecular weight = Approx. 1,15,000, Hydrolysis degree = 98 - 99%), Calcium Hydroxide 94 %, Sodium Hydroxide pellets 98%, Sulphuric Acid (AR) 98%, Sodium Sulphate Anhydrous 99%, 40% Crosslinker -1(CL-1), 37% Crosslinker -2 (CL-2), were buy from Loba Chemie Pvt Ltd, 07, Wodehouse Rd, near Bank of India, Cuffe Parade, Mumbai, Maharashtra, India, 400005. Crosslinker -3 (CL-3), purchased from Tokyo Chemical Industry Co. Ltd, 6-15-9 Toshima, Kiti-Ku, Tokyo, Japan. Shanghai Lingfeng chemical reagents co. LTD (Shanghai, China),

2.2 Methods

2.2.1 Preparation of crosslinked films PVA / Crosslinker -1, Crosslinker -2, and Crosslinker -3

This two-step process ensures efficient crosslinking of the polymer. In the first stage, the polymer solution was prepared, and thin films were fabricated using the solution-casting method. In the second stage, the fabricated films were subjected to crosslinking in an acid bath prepared by dissolving sulfuric acid and sodium sulfate in distilled water. The preparation of crosslinked films of PVA/(CL-1), PVA/(CL-2), and PVA/(CL-3) is outlined below.

Initially, PVA (5% w/v) was dissolved in distilled water by heating the solution to 85°C under continuous magnetic stirring at 800 rpm for 30 minutes to achieve complete dissolution. The solution was then cooled to 30°C (ambient temperature), and crosslinking agents were added in the desired ratios, as specified in Table 1. The mixture was stirred for an additional 10 minutes to ensure homogeneity. The resulting solution was poured into Petri dishes to form thin films, which were dried in an oven at 70°C until complete drying. Once dried, the films were carefully peeled off for subsequent crosslinking. In the second stage, the dried films were immersed in a crosslinking bath containing sulfuric acid (5% v/v) and sodium sulfate (20% w/v) for 5 minutes at 50°C. After crosslinking, the films were thoroughly rinsed with distilled water to remove residual acid and then dried in an oven at 70°C for 10 minutes.

CL-1/PVA	CL-2/PVA	CL-3/PVA
1.0	1.0	1.0
0.8	0.8	0.8
0.6	0.6	0.6
0.4	0.4	0.4
0.2	0.2	0.2

Table1 composition ratio of crosslinking agents and PVA

2.2.2 Water-swelling degree

The water swelling degree was determined by immersing a 3 × 5 cm film sample (Figure 3c) in distilled water for 72 hours. After swelling, the dimensions (ls) and weight of the swollen film were recorded. The film was then placed in a desiccator at room temperature for three days to dry completely, after which the dried film dimensions (ld) were measured. The water swelling degree was calculated using the following equation:

$$DS = [(ls - ld)/ld] \times 100$$

2.2.3 Degree of Cross

The degree of crosslinking was evaluated based on the weight loss of the films before and after immersion in water. All prepared films were initially dried to a constant weight and recorded as m0. After immersion in water for 72 hours, the films were assumed to have reached solubility equilibrium. The remaining insoluble portions were then dried in an oven at 85°C until a constant weight was achieved and recorded as md. The degree of crosslinking was calculated using the following equation:

$$DC = \mathrm{m0/md} \times 100$$

2.2.4 Evaluation of Alkaline Resistance property of PVA-coated Polyester

The alkaline resistance of the coated polyester samples was evaluated in accordance with DIN EN 14030:2003, "Screening test method for determining the resistance to acid and alkaline solutions- Geotextiles and geotextile-related products." This standard specifies a procedure for assessing the chemical stability of geosynthetics under aggressive alkaline conditions. According to the standard, the test specimens are completely immersed in solution of pH-12.5, maintained at a temperature of 60 °C for a period of three days (72 hours). Upon completion of the exposure period, the samples are removed, thoroughly rinsed with distilled water to eliminate residual alkali, and then dried under controlled conditions prior to further evaluation.

3. Result and Discussion

3.1 Water-swelling degree

Since PVA is inherently water soluble, the evaluation of water solubility after crosslinking served as a key indicator of crosslinking effectiveness. The solubility and swelling tests were therefore used to assess both the degree of swelling and the extent of crosslinking achieved. PVA was crosslinked at progressively higher crosslinker-to-PVA ratios of 0.2, 0.4, 0.6, 0.8, and 1.0, under consistent conditions summarized in Table 2. Each experiment was performed in triplicate, and the resulting film thicknesses were approximately 100 µm. Figure 1, illustrates the relationship between the swelling degree (%) and the crosslinkers/PVA ratio, with corresponding data presented in Table 2. Among the

crosslinking agents tested, CL-3 exhibited the most pronounced influence on the swelling behavior of PVA films. At a CL-3/PVA ratio of 0.2, the swelling degree was 18% (±4%). As the CL-3/PVA ratio increased, the swelling degree decreased to approximately 9%, 7%, and 6% (±4%) at ratios of 0.6, 0.8, and 1.0, respectively. This reduction occurs because higher CL-3 concentrations promote reactions at both ends of the CL-3 molecule, thereby increasing the crosslinking density and reducing the free volume within the polymer matrix. Consequently, the water resistance of the film improves significantly with increasing CL-3 content [5].

Table 2: Water swelling degree.

Crosslinker / PVA ratio	CL-1	CL-2	CL-3
1.0	Gel formation	20	6
0.8	Gel formation	18	7
0.6	Gel formation	15	9
0.4	265	26	15
0.2	300	40	18

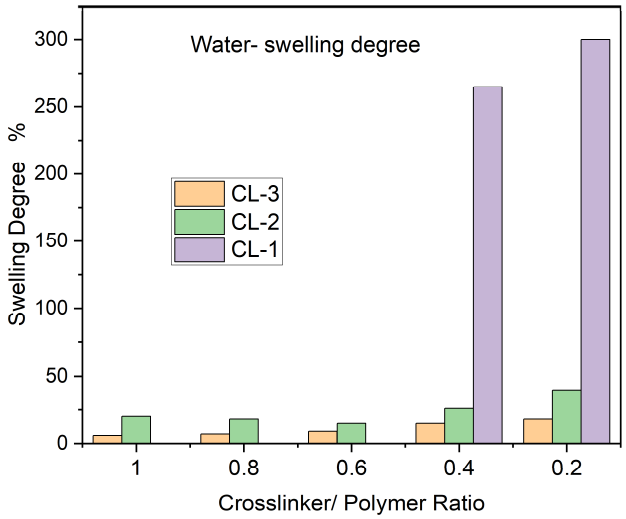

Figure 1 : Water swelling degree

3.2 Degree of Cross-Linking

Figure 2, presents the relationship between the degree of crosslinking and the crosslinker/PVA ratio, with corresponding data summarized in Table 3. The degree of crosslinking was determined using the weight loss method after immersion in water. Among the crosslinker tested, CL-3 exhibited a consistently high crosslinking degree, ranging from 96% to 99% across CL-3 /PVA ratios of 0.2 to 1.0. CL-2 showed a crosslinking degree of 93% at a 0.2 CL-2/PVA ratio, reaching a maximum of 97% at a 0.8 ratio. In contrast, CL-1 achieved a crosslinking degree of 90.2% at the lowest CL-1/PVA ratio, which decreased progressively with increasing concentration. This inverse trend may be attributed to the availability of reactive hydroxyl groups in PVA: at lower CL-1 concentrations, more active sites are accessible for crosslinking, whereas higher concentrations may hinder effective molecular interactions, reducing crosslinking efficiency. These results emphasize the necessity of optimizing crosslinker concentration to balance reactivity and performance in PVA-based systems. The observed variations could also be related to insufficient activation energy for effective reaction initiation at the relatively low experimental temperature [6].

Table 3 : Degree of Cross- linking

Crosslinker / PVA ratio	CL-1	CL-2	CL-3
1.0	64.4	96	98
0.8	66.5	97	99
0.6	68.2	94	99
0.4	76.9	94	97
0.2	90.2	93	96

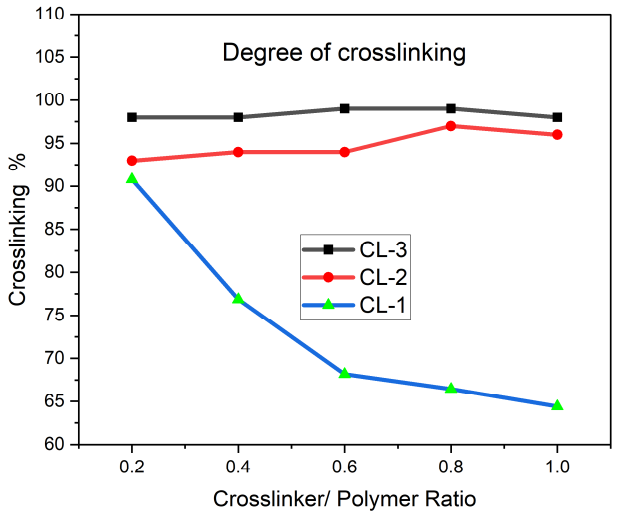

Figure 2 : Degree of cross - linking

3.3 Coating of Polyester Yarn, geotextile, and geogrid with Crosslinked PVA

Initially, a PVA-based emulsion was prepared by dissolving PVA in distilled water under constant stirring and heating to achieve a homogeneous solution. Selected additives and crosslinking agents were subsequently incorporated into the formulation to enhance coating adhesion, flexibility, and alkaline resistance. The resulting emulsion exhibited suitable viscosity and stability for application through the padding process. The prepared PVA emulsion was then applied to the polyester yarn, geotextile, and geogrid using the padding mangle unit operated at a nip pressure of 2 bars and a roller speed of 120 rpm. These parameters were optimized to ensure consistent pick-up and adequate penetration of the coating into the yarn structure without causing fiber distortion or excessive squeeze-out. After coating, the treated yarns were dried in a hot-air oven at 150°C for 4 minutes to facilitate solvent evaporation and initiate the crosslinking reaction between PVA and the crosslinker. This process yielded a thin, uniform, and adherent crosslinked PVA coating on the polyester yarn surface. The controlled drying conditions promoted the formation of a stable, three-dimensional polymer network,

enhancing the chemical resistance and mechanical durability of the yarn. The coated yarns were subsequently conditioned under standard atmospheric conditions (65 ± 2% RH and 25 ± 2 °C) prior to further characterization and performance evaluation.

3.4 Evaluation of Alkaline Resistance property of coated Polyester

The Grams per Square Meter (GSM) of each specimen of virgin polyester textile, coated polyester textile, virgin polyester geogrid, and coated polyester geogrid, before and after alkaline exposure was measured to evaluate the effect of the PVA coating process on material mass and surface coverage. Each specimen was prepared with a fixed size of 20 cm × 20 cm (400 cm²), and the corresponding weights were recorded using a precision balance. GSM values were calculated using the standard relation: The calculated GSM provides a normalized value independent of sample size, allowing direct comparison between coated and uncoated materials.

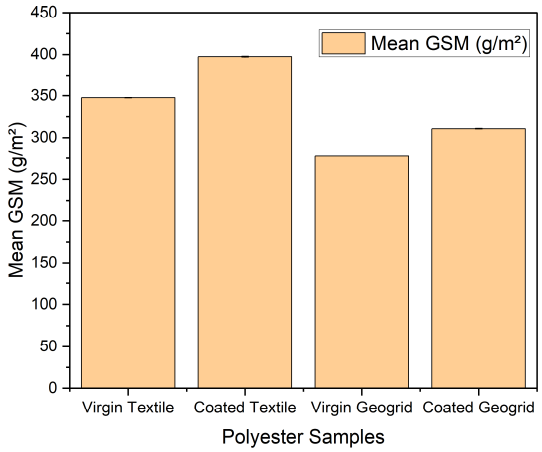

Figure 3 : GSM for Coating uptake Assessment

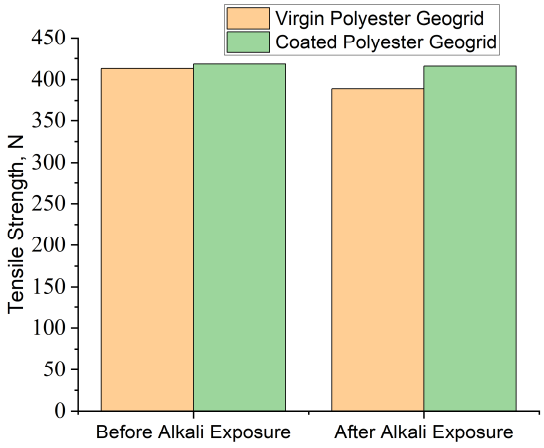

Figure 4 TS for evaluation of Alkaline Resistance

Each GSM value represents the mean of five independent replicates, ensuring statistical reliability. The associated standard deviations were minimal across all samples, confirming excellent repeatability and consistent coating application. Figure 3, presents the relationship between GSM, The virgin polyester textile exhibited a mean GSM of 347.575 g/m², while the coated textile showed an increased GSM of 397.305 g/m², corresponding to an approximate 14.3 % mass gain. This increase can be attributed to the uniform deposition of the crosslinked PVA coating over the textile fibers, indicating effective wet pickup and strong adherence of the coating layer. Similarly, the virgin geogrid demonstrated a mean GSM of 277.145 g/m², which increased to 310.655 g/m² after coating an increment of 12.1 %. The slightly lower percentage increase in geogrid GSM compared to the textile suggests that the open structure of the geogrid allows less polymer retention compared to the denser textile surface.

3.5 Effect of Alkaline Exposure on GSM Retention

Post-exposure GSM measurements were compared with pre-exposure values to determine percentage mass loss. The virgin polyester textile exhibited a -3.20% reduction in GSM, while the coated textile displayed only a -0.10% loss under identical conditions. Similarly, the virgin polyester geogrid experienced a -3.61% GSM reduction, whereas the coated geogrid showed a negligible -0.20% loss. This significant difference highlights the protective efficacy of the crosslinked PVA coating against alkaline hydrolysis. The marked degradation observed in uncoated polyester is attributed to the hydrolytic cleavage of ester linkages in the polymer backbone, which accelerates in highly alkaline environments. In contrast, the coated specimens retained nearly their entire mass, indicating that the crosslinked PVA layer acted as an effective diffusion barrier, preventing direct contact between the polyester surface and the aggressive alkaline medium.

3.6 Tensile Strength Evaluation Before and After Alkaline Exposure

Figure 4, presents the relationship between tensile strength of the virgin and coated polyester geogrids. Tensile strength was determined to assess the influence of the crosslinked PVA coating on mechanical integrity and durability, both before and after alkaline exposure. Each reported value represents the mean of five replicates to ensure statistical accuracy and reproducibility. Before exposure to the alkaline medium, the virgin polyester geogrid exhibited a mean tensile strength of 413.24 N, while the coated polyester geogrid demonstrated a slightly higher mean strength of 418.42 N. This marginal increase (~1.25%) indicates that the PVA coating did not adversely affect the intrinsic mechanical properties of the polyester substrate. Instead, the coating contributed to a minor improvement, likely due to the formation of a thin, crosslinked polymer layer that enhanced fiber–fiber frictional resistance and load distribution during tensile loading. Following alkaline exposure distinct differences were observed between the coated and uncoated samples. The virgin polyester geogrid experienced a substantial decline in tensile strength to 389.02 N, corresponding to a 5.86% reduction compared to its original value. This degradation is attributed to the alkaline

hydrolysis of ester linkages in the polyester backbone, resulting in chain scission, molecular weight reduction, and surface erosion, which collectively weaken the fiber structure. In contrast, the coated polyester geogrid retained a tensile strength of 416.46 N after alkaline exposure, showing only a 0.47% reduction relative to its pre-exposure value. The negligible loss in tensile strength clearly indicates the excellent barrier protection provided by the crosslinked PVA coating. The coating effectively inhibited alkaline species from diffusing into the polyester matrix, thereby preventing hydrolytic degradation. This finding aligns with the GSM retention results, where the coated specimens exhibited minimal weight loss (<0.2%) under identical test conditions.

These results collectively demonstrate that the crosslinked PVA coating preserves both dimensional stability and mechanical strength of polyester geogrids when exposed to high-pH environments. The combination of high tensile retention and low GSM variation after exposure confirms the chemical inertness, structural integrity, and long-term performance reliability of the coated material.

4. Conclusion

This study demonstrated the successful development of a crosslinked PVA coating for enhancing the alkaline resistance of polyester-based geosynthetics. The coating, applied using a padding mangle process and thermally cured at 150 °C for 4 minutes, produced uniform and adherent films on both polyester textiles and geogrids. The GSM analysis confirmed effective coating deposition, showing increased areal weight and consistent uniformity. After exposure to an alkaline solution, the coated samples exhibited negligible GSM loss (< 0.2 %), whereas uncoated materials showed about 3–4 % weight reduction, indicating strong chemical resistance of the coating layer. Mechanical testing further validated the coating performance: coated geogrids retained over 99 % of their tensile strength after alkaline exposure, compared to a 5–6 % drop in the uncoated specimens. These results confirm that the crosslinked PVA layer effectively prevents alkaline hydrolysis, maintaining the structural and mechanical integrity of the polyester substrate. Overall, the developed coating provides a durable, sustainable, and easily scalable solution for improving the long-term chemical stability of polyester geosynthetics. Its compatibility with existing industrial processes makes it highly suitable for civil and geotechnical applications where alkaline exposure is a concern.

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Effect of tow size on solvent extraction from PAN fibers during wet spinning- A MATLAB-based FDM study

[1]Nishant Chandel*, [1]Karishma Hemani, [1]T V Sreekumar, [2]Rani Rohini
[1]The Bombay Textile Research Association, LBS Marg, Ghatkopar (W), Mumbai 40086, India.
[2]Department of Materials Engineering, Indian Institute of Technology Jammu, Jagti, Jammu and Kashmir - 181221

Abstract

Residual solvent content in the precursor fibers from polyacrylonitrile (PAN) is one of the key parameters that severely affects the mechanical properties of resultant carbon fibers to a large extent. Although its significance is well-known, the influence of precursor tow size (i.e., number of filaments) on the kinetics of solvent removal is not yet reported. In order to develop the relationship for better PAN precursor spinning line design, a computational model using MATLAB is presented. The model uses a finite difference method (FDM) approach using Fick's second law of diffusion for dynamically determining the residual solvent content in fibers after passing through various units. It is assumed that the multifilament tow takes the shape of a cylindrical geometry, considering hexagonal closed packing (HCP). The models were simulated for 1K, 2K, and 3 K precursor fiber tows passing a typical wet-spinning line. The model data values are also verified with consistent data recorded from Gas Chromatography-Mass Spectrometry (GC/MS) equipment. According to the data presented, solvent removal efficiency significantly reduces as tow size increases, while the washing unit operation is the most influential on extraction due to longer retention times. While significant size reduction occurs in the stretching unit but its influence on solvent reduction is not comparable with the washing unit. This paper presents a theoretical and reliable methodology for optimizing spinning plant parameters, selecting appropriate units, and developing higher-quality PAN precursors for high-performance carbon fiber applications.

Key words:
Carbon Fiber, Polyacrylonitrile (PAN), Wet-Spinning, Finite Difference Method, Solvent Diffusion, Process Modelling.

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1.0 Introduction

The residual solvent content in PAN fiber is a very crucial parameter for the high-strength Carbon fiber production process. The high-solvent content severely affects the morphology of the PAN precursor, which results in microvoids in the carbon fiber [1]. Thus, an optimal quantity of solvent is desirable in the PAN precursor fiber for optimal strength and modulus in carbon fibers. Various spinning parameters, such as tow size (number of filaments), spinning bath concentrations, spinning line design, and spinning speed, have a direct influence on the solvent content in PAN precursor fibers [2,3]. As reported by Gao et al, the higher spinning line speed resulted in higher PAN precursor strength, but due to the presence of higher solvent content (~820 ppm), it resulted in lower strength in carbon fiber [4]. However, the PAN fibers spun at a lower spinning speed resulted in comparatively lower strength in the PAN precursor along with lower solvent content (~ 493 ppm) in it, resulting in higher strength in the Carbon fibers. This study shows the great significance of solvent content in PAN precursor that significantly affects the carbon fiber properties, where higher solvent content results in a higher number of microvoids in carbon during the heat treatment process. Though the importance of solvent content can be observed, the minor increase in it from ~500 ppm to 820ppm drastically reduces the strength in carbon fibers, but this study is only limited to the effect of one influential factor. However, the influence of other factors is yet to be known, which can help researchers and industry professionals to further improve the properties of the carbon fiber. Thus, this literature gap opens up an opportunity for further research in the related field.

*Corresponding author,
E-mail: carbonfibre@btraindia.com

In this work, a MATLAB-based study is performed along with experimental verification to understand the effect of tow size and spinning unit components on the solvent content present in PAN precursor fibers during the spinning process. The effectiveness of the different types of unit and their effect on solvent extraction capability is also studied through the model. Further, the predicted values from the program were compared with the experimental values of the solvent content obtained through GC/MS. Based on the predicted values & experimental values, an optimized spinning line design is also applied and verified.

2. Experimental

Polyacrylonitrile copolymer (Mw: 100 kg/mol) was procured from Technorbital Pvt Ltd. N,N-Dimethyl acetamide (DMAc) solvent of commercial grade is used in the study was procured from Balaji amines Pvt Ltd. PAN fibers for the experimental sample preparation were prepared using a wet-spinning machine with 3 different spinneret sizes of (1000 holes) 1K, (2000 holes) 2K, and (3000 holes) 3K with a polymer dope concentration of 20%. The spinning parameters, such as jet-ratio (0.8), total draw ratio (4), take-up speed (1m/min), and bath temperatures (90 ^{o}C), were kept the same for all tow sizes.

The MATLAB model based on the finite difference method (FDM) analysis is developed, which determines the diffusion of the dimethyl acetamide solvent in the baths as a function of time and radius of the tow size. The objective is achieved by solving Fick's second law-based mass diffusion equation, considering each filament acting as the nodal point. One more assumption taken for this theoretical study is considering the multifilament tow forms a cylindrical shape as per the hexagonal close packing (HCP) theory in the form of multiple layers under tension. This assumption is taken to simplify the theoretical framework & considering that this form is most suitable for maximum packing efficiency and uniformly distributing the stretching load.

The experimental verification of solvent content from the wet-spun fibers of different tow sizes was done with a Gas chromatograph and mass spectrometer (GC/MS), Shimadzu GC/MS QP2010. 5g samples of the final processed fiber samples were taken & tests were performed using the headspace method. The solvent content obtained from the GC/MS is normalized with the pure DMAc solvent value (taken as standard). The detailed method of normalization and GC/MS reports is represented in the supplementary information.

3. Results and Discussion

The model for the tow geometry is developed based on the HCP theory, which gives the highest packing fraction (74%) and also conforms with the material behaviour to attain the lowest surface area under tension [5,6]. During this transition, the multifilament tow forms the structure by forming multiple layers covering the layers underneath it and preventing their direct contact with the outside medium [7]. Figure 1 represents the schematic of the layering formation by the multifilament tow under tension.

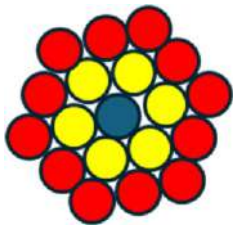

Figure 1: Schematic of the Hexagonal pack structure formed by multifilaments

With reference to the HCP theory for the formation of such cylindrical structure in multilayer form, the number of layers can be determined by the number of multifilaments present in it [8]. Thus, the number of layers obtained from 1K, 2K, and 3K filaments are obtained to be 18 layers, 26 layers, and 32 layers, respectively. Moreover, based on the HCP theory, the effective radius before any effect of the jet-ratio & draw ratio for the tows of 1K, 2K, and 3K can also be calculated (as per equation 1), and determined to be 3.16mm, 4.47mm, and 5.47mm, respectively [9].

$$R_{eff} = r \times \sqrt{(N)} \ldots (1)$$

Where, R_{eff} is the effective radius of the Tow,

r is the radius of the single filament, and

N is the number of filaments

For efficient and faster calculation of the concentration reduction during the various processes, Fick's second law of mass diffusion from a cylindrical geometry system is employed, due to its ability to link concentration with time and spatial dimension. The law states that the rate of change of concentration with respect to time can be determined from the double spatial derivative of the concentration. The governing equation for the same for cylindrical geometry is represented in equation 2 [10].

$$\frac{\partial C}{\partial T} = D\left(\frac{\partial^2 C}{\partial x^2} + \frac{1}{x}\frac{\partial C}{\partial x}\right) \ldots . (2)$$

Where, C is the solvent concentration,

x is the spatial dimension,

D is the diffusion coefficient of the solvent in the outside medium, and

T is the time.

Though the equation is compatible with solving the cylindrical geometry system, but it requires the system to be homogeneous for computation. While in cylindrical geometry, a structure formed by multifilament tow is bound to have gaps in between due to its 74% packing efficiency.

Along with that, the bigger geometry requires a higher value of time difference, which severely affects the accuracy of the system, while lower time difference values may result in higher computing time. Thus, to reduce the computing equations and increase efficiency, the discretization of the cylindrical system is done along a single axis (along the radial direction). The discretized form of the governing equation is represented in equation 3 [11].

Governing equation:

$$C_i^{m+1} = C_i^m + \frac{D\Delta t}{\Delta r^2}\left[\left(1+\frac{\Delta r}{2r_i}\right)C_{i+1}^m - C_i^m + \left(1-\frac{\Delta r}{2r_i}\right)C_{i-1}^m\right] \quad .....(3)$$

Where, C_i^m is Concentration at radial position $r_i = i\Delta r$ and time $t_n = n\Delta t$,

D is the Diffusion coefficient of the DMAc solvent in the water (1.41×10^{-9} m^2/s) [12],

Δr is the Spatial step size,

Δt is the Time step size, and

r_i is Radial position, $r_i = i\Delta r$.

m is a step for time, and

i is the spatial step.

Initial conditions:

$C = C_i^m$ at $0 < r \leq R_{eff}$ …(4)

$C = 0$ at $r > R_{eff}$ … (5)

Boundary conditions:

$C = 0$ at $r > R_{eff}$ for $0 < \Delta t < T$ …(6)

Therefore, the calculation of the concentration profile with reference to time& spatial dimension (radius) is done for various tow sizes (1K, 2K, and 3K filaments) to determine the solvent reduction in the various components of the wet-spinning line. For normalization, the determination of the solvent profile is done by designing a line with similar components arranged in the same arrangement and at similar speeds to give a similar draw ratio. Therefore, the effect of line designing on the solvent extraction from the various tow can be compared. The designed spinning line for the solvent extraction calculation consists of 2 coagulation baths, 2 stretching units, and 1 washing unit respectively. The flow diagram of the line is represented in Figure 2, and details of processing parameters are given in Table 1.

Figure 2: Hypothetically designed wet-spinning line for solvent extraction

Table 1: Parameters of wet-spinning line for solvent extraction

S. No.	Unit	Parameters		
		Temperature (°C)	DMAc Concentration (%)	Residence time (s)
1	Coagulation Bath-1	30	70	120
2	Coagulation Bath-2	30	0	41.63
3	Washing unit	90	0	570
4	Stretch Bath-1	90	0	24.27
5	Stretch Bath-2	90	0	17.65

The calculation of the solvent extraction from the various tow sizes (1K, 2K, and 3K) is performed according to the FDM model with respective initial and boundary conditions. Figure 3 represents the solvent reduced from the various tows at different stages of the wet-spinning line. The effect of layering formed due to a larger number of filaments on solvent extraction was found to be prominent at all stages. The initially reduced value of solvent in 1K, 2K, and 3K filaments after the coagulation bath was found to be 71.5%, 74.1%, and 75.3% respectively. This also correlates well with the coagulation theory, where a single filament undergoes a double diffusion process and coagulates to form the semi-solid gel fiber structure [13]. The residual solvent content in 2K and 3K was obtained to be the same, while 1K shows a comparatively lower value, indicating the role layer structure formed by the tows and their critical limit, which reduces solvent extraction efficiency due to more number of layers. Thus, it also indicates that the major factor responsible for the solvent extraction difference between 2K and 3K mainly depends upon the further components of the spinning line, which are responsible for stretching and washing of the fibers. As shown in Figure 3, the reduction in solvent concentration is more prominent in the subsequent units of the wet-spinning machine. One noteworthy observation is in the solvent reduction data after washing units, where no further notable reduction was observed for the 1K filament yarn. This indicates the efficient washing & solvent extraction in the washing unit. Thus, the difference between the stretching unit and washing unit can be observed from the plots. Though the stretching unit does reduce the fiber size, which exposes the new surface, but residence time remains less. While the washing unit has a much higher residence time, no reduction occurs in size. Still, this prolonged time is enough to efficiently extract the solvent from the tow. For better understanding, the comparative simulation video of both the stretching unit and washing unit for 1K is illustrated as SV1K and WV1K in the supplementary information.

While the higher number of layers present in 2K and 3K showed resistance to the washing process, in comparison to 1K showed a very drastic reduction in the average solvent concentration of yarn. Moreover, the model is also tuned to

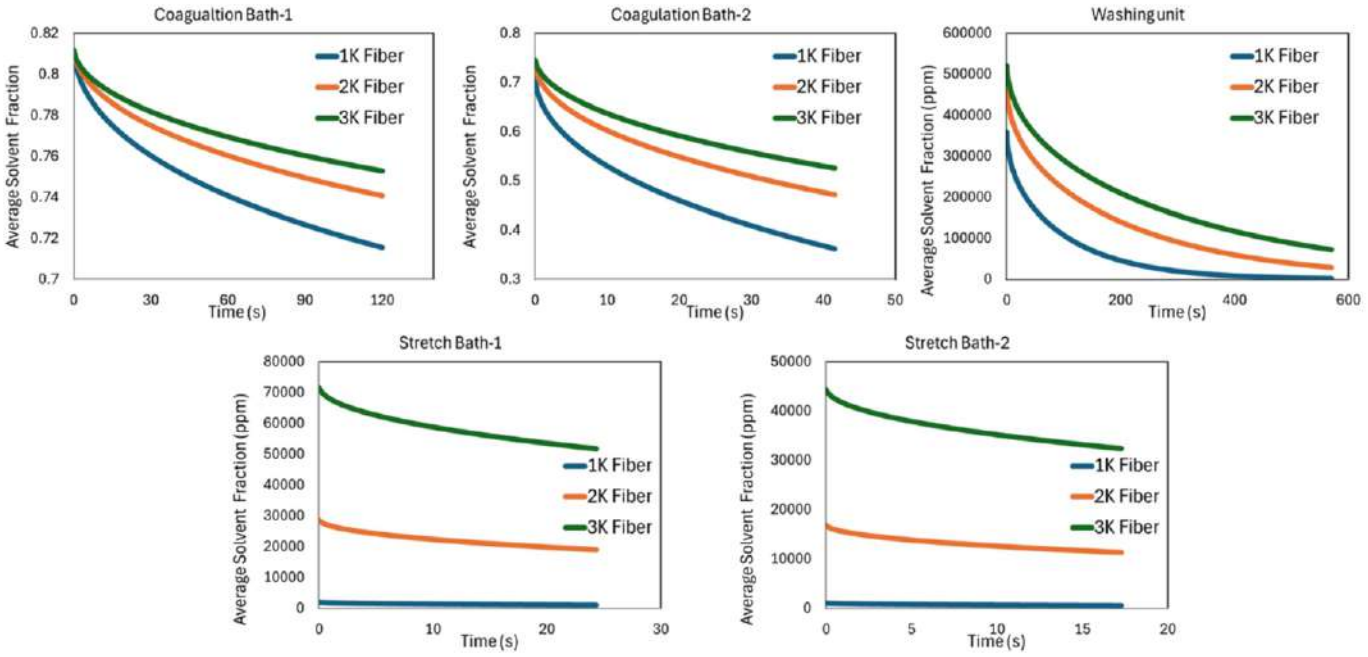

Figure 3: Solvent extraction profile for 1K, 2K, and 3K filaments at various stages

determine the penetration of the water into the core filaments of the yarn is also performed. The 2D colour map plot of the 1K, 2K, and 3K filament yarns after passing through the stretching units and washing unit is represented in Figure 4. It can be clearly observed that the reduction in the concentration is obtained in the washing unit only with a more prominent reduction obtained in 1K than in 2K and 3K. While the clear distinction of solvent extraction can be made in comparison to 1K and 2K & 3K filament yarns in both the stretching units & washing unit. Due to the smaller yarn size & less number of nodal points, 1K showed a drastic and faster reduction in the concentration values than 2K & 3K [14]. It is anticipated that a similar effect will also take place with the actual yarn samples.

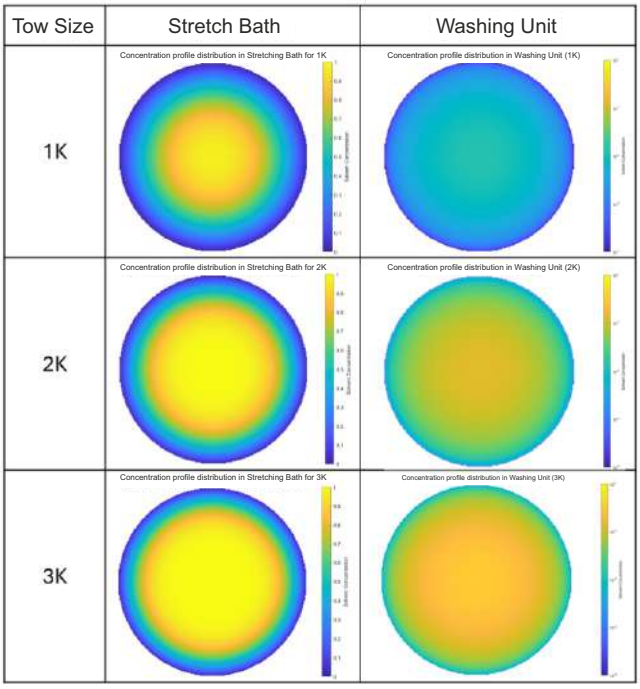

Figure 4: Solvent concentration reduction in: (A) Stretching unit, (B) Washing unit

Thus, the FDM study clearly indicates the effectiveness of the function of the different units used for the wet-spinning process. Multiple process parameters, such as the effective radius of the tow, tow size, and residence time, play a significant role in the residual solvent content in the PAN fiber. However, without experimental validation, these results only give a superficial comparison of the PAN fiber properties with different tow sizes due to the wet-spinning line design. To validate the FDM results with the actual solvent content present in the fibers, the respective fiber samples were tested with a GC/MS machine. The DMAc solvent gives a solvent peak at 7-7.3 R-time in GC/MS, which is obtained from the analysis of the pure DMAc solvent. With reference to the peak properties of DMAc from the pure DMAc solvent (standard), the comparative content present in the yarns was determined to obtain the solvent content present in the fiber sample [15]. The solvent content in 1K, 2K, and 3K fiber tows was tested with GC/MS for the residual solvent content and is represented in Table 2.

Table 2: Residual solvent content in the fibers with different tow sizes

S. No.	Tow size	FDM predicted value (ppm)	Experimental value (ppm)	Difference (ppm)
1	1K	327.1	122.6	204.5
2	2K	11,258.1	10,889.5	368.9
3	3K	32578.5	26005.2	6573.7
4	2K-modified line	897.2	2597.8	-1700.6
5	3K-modified line	5464.4	5983.4	-519

From Table 2, it can be observed that the predicted solvent content values through the FDM model are in accordance with the experimental values obtained from the GC/MS values, indicating the FDM model is valid. It can be observed

that the reduction in the solvent content (in ppm) values holds good for the 1K and 2K filaments HCP model. Whereas above it (for 3K filaments), the packing must follow a different geometry due to their own weight, which increases the surface area & more contact points with the water, along with a reduced number of layers. Therefore, this study shows its direct application in the wet-spinning of PAN, where appropriate unit placement and type not only directly influence the mechanical properties but also the solvent content in the yarn as well.

Therefore, based on the information, the revised hypothetical wet-spinning line is designed with 2 washing units running at the same speed. Since washing is found to be more efficient in extracting the solvent from the stretching unit. The FDM results show that there will be a drastic decrease in the solvent content value in 2K filaments compared to 3K filaments. However, it is observed that the HCP theory gave a better prediction for the 3K filament result than the 2K. For experimental samples, the 2K and 3K filament yarns, right after washing from the washing units, were collected and washed again. The FDM-based value & experimental values obtained through GC/MS are represented in Table 2. It can be observed that the values are in good agreement and show reduced solvent values from the previous single wash values. This shows that through this theoretical framework approach, a wet-spinning line can be designed for high-strength PAN fiber, which may also result in high-performance carbon fiber with fewer microvoids in it.

4. Conclusion

An FDM based computational model was successfully developed in this work to mimic and simulate the influence of tow size on solvent removal from PAN precursor fibers in the wet-spinning process. The results confirmed that smaller 1K tows are significantly more efficient in solvent removal than larger 2K and 3K tows, resulting in solvent content values as low as 327 ppm in 1K. The difference is caused by the smaller effective radius and fewer filament layers in the 1K tow, which decreases the diffusion path length for the solvent. The most important finding was the crucial role of the washing unit, whose longer residence time is indispensable for removing residual solvent from the tow's core since this is less effective with short-duration stretch baths. The predictions of the FDM model showed good agreement with the GC/MS experimental measurements, which validated the FDM model as a robust and inexpensive tool for process simulation. The present model provides valuable knowledge to engineers for better line design and process parameters, which reduces the need for extensive experimental trials and helps to customize the precursor properties for the synthesis of high-strength carbon fibers.

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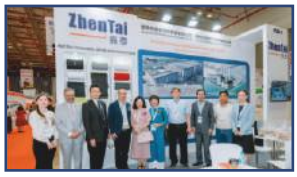

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