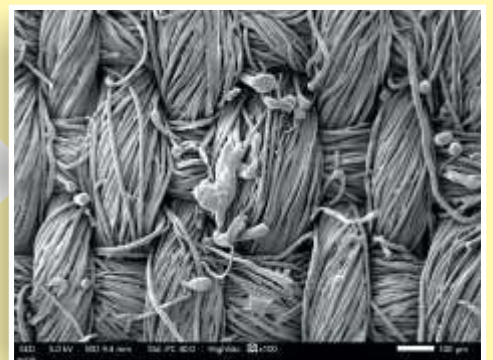
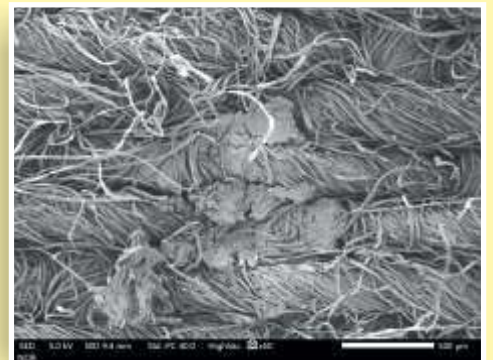
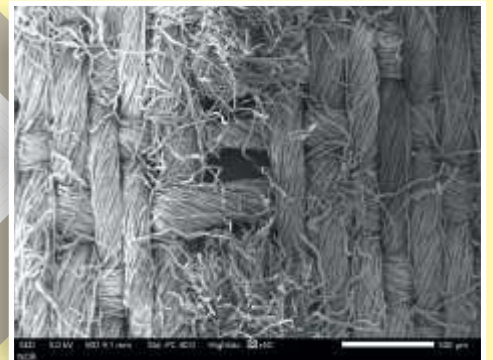


bttra scan

Vol. LIII No. 4 October 2024

BTRA SCAN DOI: 10.70225/874384hydog



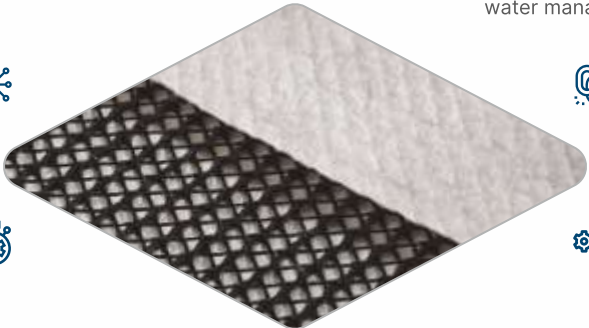
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01 


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08 


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02 


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07 


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03 


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06 

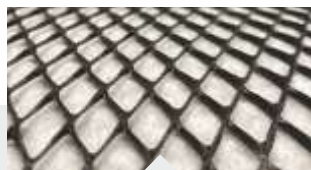
Multi functioned and improved performance

04 


Easy transportation

05 


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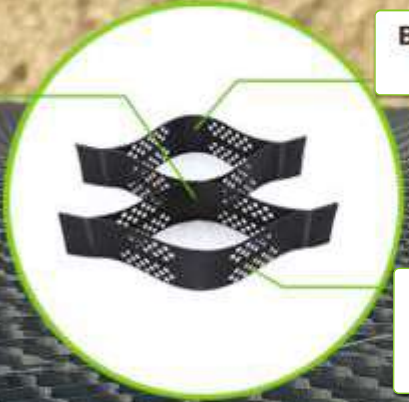




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EDITOR'S DESK

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. 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 53rd Edition of BTRA SCAN.

This issue has papers from the different domains such as analysis of textile defects using SEM, overview on rheology of UHMWPE/ solvent systems and review on high Performance Synthetic Fibers for Composite Materials & it's Application. 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
Director, BTRA

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ISSN 0972 - 8341

BTRA Scan is abstracted by -

- Elsevier Bibliographic Database, UK,
- EBSCO Research Database, USA
- Chemical Abstract Services, USA

BTRA Scan is published quarterly by the Bombay Textile Research Association, Lal Bahadur Shastri Marg, Ghatkopar (W), MUMBAI — 400 086, INDIA; Tel. : 022-62023636; Email : info@btraIndia.com Website: www.btraIndia.com

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The annual subscription price is Rs. 1000/- only (India) / USD 100 (Rest of World)

Root Cause Analysis of Textile Defects Using Scanning Electron Microscopy

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Abstract

Textile manufacturers are extensively working on improving the market with qualities, however, textile defects are a great hindrance to them. This article looks at the standard reasons for commonly occurring defects in textiles by employing an SEM analysis. Playing an analytical role, SEM is a tool that allows one to obtain an image that is of a fully enlarged surface of the material. The analysis describes the diagnosis of some microstructural abnormalities such as fiber breakage, contamination of the surface, and irregularities in the spinning and weaving of different textile samples. SEM helps to understand the physical and chemical environments leading to these defects by facilitating the study of these deformities at the micro and nano scales. The investigation performs a defect root cause analysis where the link is made between the defect and a process of manufacture, the quality of raw materials used, or the state of the environment. This article demonstrates how textile manufacturers can use SEM textile defect diagnosis to make improvement recommendations that are actionable to enhance production and reduce the incidence of defects in the future.

Keywords

Root cause analysis of textile defects; Scanning Electron Microscopy

Citation

Amol G. Thite*, Geeta P. Jawdekar, Manasi D. Kadam, Soumya S. Behera- "Root Cause Analysis of Textile Defects Using Scanning Electron Microscopy", *BTRA Scan* - Vol. LIII No.4 , October, 2024, Page no. 5 to 13, DOI: 10.70225/839567asqpk

1.0 Introduction:

Among the largest and most intricate manufacturing industries in the world is the global textile industry, which has applications in apparel, medical, automotive, and even aerospace textiles [1]. With the increasing popularity of high-end and defectless textiles, maintaining the mass production process has become crucial. Textile defects, which can take place because of a great number of factors, lower not only the mechanical, aesthetic, and functional capabilities of the products but also bring about consumer dissatisfaction, economic loss as well as loss of credibility to the producers [2]. Hence, textile defect analysis becomes one of the essential aspects of quality assurance. Many approaches have been used through the years to detect and minimize textile defects including conventional inspection methods and more sophisticated tools [3].

1.1 Textile Defects and Their Classification

Textile defects can be divided into structural defects, surface defects, and chemical defects. These defects can occur at different points such as in the production of the fiber, its spinning, weaving, dyeing, finishing, etc. [4].

(i) Structural Defects: These are comprised of fracture of the fiber or the filament, irregular twists of the yarn, and weaving imperfections like the breaking of the warps and wefts. This implies that structural defects are mostly due to mechanical stress factors during the different stages of production, including tension during spinning, weaving, or chemical processes.

(ii) Surface Defects: Pilling, fuzzing, and foreign matter rise to the surface during manufacture or post-manufacturing processes, or due to finishing processes that are incorrectly applied, all of which are referred to as surface defects. Apart from this, they are also associated with low surface tension in synthetic fibers or poor heat setting.

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(iii) Chemical Defects: These consist of bad dyeing, chemical leaching leading to fading of fibers, and synthetic textile polymerization defects. It is interesting to indicate that chemical defects are severe in blended textiles where two or three types of fibers are seldom capable of consistent chemical synthesis, leading to wide variation.

1.2 The Significance of Root Cause Analysis in Textiles

Defects may develop at any of the stages in the production process such as fiber, spinning, weaving, dyeing, and finishing and hence it becomes very important to detect them and take corrective actions. So, Root Cause Analysis (RCA) is the systematic process of investigating the reasons behind defects or failures in a given system. In the case of textiles, RCA helps to determine if the materials used were substandard, if the processes were too harsh, or if the defects are caused by outside conditions which we will call the environment. When defects and failures are eliminated through the understanding of the causes of the defects, targeted solutions are provided and hence efficiency increases and wastage is decreased [5].

1.3 Traditional Methods for Defect Detection

Within the field of textile engineering, the characterization of basic sources of defects has traditionally been undertaken through methods such as visual inspection (optical microscopy) and mechanical evaluation.

(i) Optical Microscopy: Optical microscopy is the best-known class of methods for the examination of textile fibers and the evaluation of surface defects. However, such resolution limits, especially at higher magnifications, prevent it from finding finer structural details such as micro-cracks or nano-scale contamination. The technique cannot show defects under that diffraction limit of the light used, approximately 200 nm on average in most optical microscopes.

(ii) Mechanical Testing: Tensile testing and abrasion resistance tests probably are the most common analyses that are used for checking the mechanical properties of fibers. The above tests can indirectly suggest some defects by way of strength and durability evaluation, but they do not give direct visual evidence of the defects themselves. Purely relying on mechanical testing is meager enough to pinpoint or identify precisely the sources of defects and makes source determination quite elusive.

1.4 Limitations of Traditional Methods for Defect Detection

While these techniques are highly sensitive to large defects, they do not have any resolution level for the detection of micro-scale defects. This is why many scientists established the limitations of traditional techniques. They often fail to deliver the required level of resolution and analytical complexity necessary to identify the causes of defects at a microstructural level. For example, this limitation is severely critical in the cases of detecting anomalies at small scales, like micro-cracks, fiber dislocations, surface contamination, and poor bonding of fibre-matrix of composite textiles [3].

1.5 Introduction of Scanning Electron Microscopy Techniques

Advanced microscopy techniques have been developed to bridge the lacuna regarding traditional approaches to textile defect evaluation. The most outstanding tool mainly because it generates micrographs of the surface and the structures of textiles at unprecedented resolutions, is SEM. SEM has various advantages compared to optical microscopy among which include magnification and depth of field. The SEM relies on a focused beam of electrons to offer high-resolution images of the textile surfaces and cross-sections that expose defects invisible to the naked eye. A review has established the utility of SEM in analyzing the microstructural defects of both woven and nonwoven fabrics; fractures along a fiber; surface contamination and areas of incomplete bonding between fibers [6]. Unlike optical microscopy, which has disadvantages including light diffraction and lesser resolution, SEM uses a focused beam of electrons that interact with the surface of a material to produce secondary and backscattered electrons that may reveal the detailed topography and composition of the surface. This would consequently increase the applicability of scanning electron microscopy, particularly because it can function in more than one modality- namely, secondary electron, backscattered electron, and X-ray microanalysis. This makes it possible to gather data on both the surface morphology and chemical composition, essential for identifying root causes of defects. Moreover, SEM enables the morphological observation of textile fibers and surfaces at magnifications ranging from hundreds to hundreds of thousands; therefore, it becomes possible to visually observe such defect structures on a micro and nano level [7]. High magnification makes possible the study of sub-micron features, such as fiber fractures, voids, and contamination that are not easily visible by optical microscopy or even by visual inspection. Hence, SEM has very widely been used in the textile industry over the last decade due to its excellent ability to provide high-resolution images of both surfaces and cross-sections of textiles. Identification and defect analysis through microscopic observation are fast becoming an extremely valuable tool in SEM analysis. Analysis of such defects facilitates diagnosis of the defect and tracing back of the defect to certain stages of the manufacturing process. For example, fiber breakage might be associated with insufficient spinning tension whereas contamination could relate to low environmental controls of the processing course. Thus, the basic focus in this regard would be on the usage of SEM for extensive root-cause analysis of defects in textile samples.

Therefore, the main goals of this article are (i) General defects encountered and documented with different textile materials using SEM imaging techniques. (ii) Fundamentals of defects: correlation of SEM results with manufacturing parameters, material characteristics, and environmental variables. (iii) Suggestions to processing techniques with possible enhancements and corrective actions found from fundamental causes established by SEM examination. By doing so, the outcomes of this analysis will provide manufacturers in the textile industry with actionable

knowledge, which helps reduce defects, increases the efficiency of production processes, and generates higher quality textiles.

In simpler words, placing more emphasis on the root cause analysis of defects would provide practical advice to the textile industry that would help manufacturers solve their problems at the source rather than relying on rectification actions after production. The results will contribute to existing knowledge in the domain of textile defect analysis, showing just how essential SEM can be in identifying those micro-structural defects that the more conventional methodologies often miss. The inclusion of SEM in routine quality control may imply massive improvements in the quality of products entering the market, defect reduction, and observation of industry standards.

1.6 Limitations of SEM in Textile Analysis

Though scanning electron microscopy is useful as a characterization technique to inspect textile defects, there are limitations. An important limitation relates to the lack of preparation of samples in many cases when a thin conductive coating, like gold or platinum, needs to be deposited on non-conductive textile materials to prevent charging effects resulting from the exposure of the electron beam. The problem is, in some cases, this affects the surface properties of the textile; therefore, the defects cannot be observed in their natural form. Moreover, SEM is a surface-sensitive technique and sometimes may not provide a piece of information related to internal defects unless cross-section imaging is performed. However, this gap would be bridged by bringing together SEM with the utilization of secondary techniques such as TEM and AFM because most restraints would be overcome as a much better examination of defects in textiles would be conducted.

2. Methodology

The method describes a procedure to detect and classify the defects associated with the application of Scanning Electron Microscopy on textiles. It encompasses the following: selection of samples, sample preparation, imaging by SEM, classification of defect types, and root cause analysis. The main idea is to utilize SEM for the proper observation and defect diagnosis at the microscopic level and to develop the source of defects during manufacturing.

2.1 Sample Selection

In this regard, the first one was a sample selection of textiles that corresponded to certain manufacturing techniques and kinds of fibers. The choices were made so that both normal and defective portions would be included in the selection process. This was to enable a comprehensive examination of the possible defects existing in the textile industry.

2.2 Sample Preparation

Textile samples are carefully prepared for SEM analysis to ensure accurate imaging and minimize the introduction of artifacts during preparation.

- **Cleaning:** In some instances, the specimen requires cleaning following compressed air in a manner of attempting to remove the loosely bonded particles and debris. Where surface contamination exists, by way of oils or chemical residues, cleaning using solvents like ethanol or acetone is used followed by air drying in desiccators or ambient air. There is a need to avoid contamination while viewing in the SEM.
- **Cutting:** Cross-sections by microtome or sharp blade for fibers, yarns, woven, nonwoven, and composite textiles. This proved useful in preparing the samples for SEM study on characteristics of internal structure and surface texture.
- **Mounting:** Prepared samples are mounted on SEM stubs using carbon adhesive tape. Carbon tape has the advantage of reducing charging effects in non-conductive material and ensuring that the stability of the sample is met during imaging.
- **Coating:** For SEM, some specific conditions are required, especially for non-conductive materials, such as textiles that require special procedures for coating and mounting. In such a case, the non-conductive textiles undergo a thin layer of a conductive material, such as gold or platinum, to prevent the charging effects due to exposure to an electron beam. A sputter coater is used for this purpose. The coating is carried out at low pressure (~10 mTorr) with a gold/palladium target. The deposition time ranged between 30 and 90 seconds to obtain a deposition thickness of about 10 nm, thereby preserving the native surface properties of the textile while avoiding electron charging.

2.3 Scanning Electron Microscopy Process

The study utilizes a high-resolution scanning electron microscope with a magnification range of 15 to 300,000 times. For analysis of defects in BTRA, the used equipment is the JEOL JSM IT 200 LV of Japan SEM which is equipped with EDAX (USA) EDX detector as they offer good resolution sensitivity and appropriateness for defective samples. Typically, an accelerating voltage of 5 to 10 kV is sufficient to provide maximum resolution without damaging the fibers. The applied voltage is less in surface images and higher for deeper penetration in composite materials. The vacuum is excellent with a high level without admitting moisture and oxygen, which are highly devastating. Several imaging modes are applied to produce sufficient information concerning surface morphology and micro-structural flaws.

- **Secondary Electron (SE) Imaging:** Used to capture high-resolution surface details, ideal for identifying defects like surface contamination, pilling, and fiber roughness.
- **Backscattered Electron (BSE) Imaging:** Employed to analyze the composition of the textile materials, especially useful for detecting defects caused by chemical inconsistencies or impurities within composite fibers.

- Energy Dispersive X-ray Spectroscopy (EDS): This technique is coupled with SEM to identify elemental composition and locate chemical defects or contaminants. EDS is critical in analyzing defects caused by chemical residues or additives used during textile processing.

2.4 Imaging Procedure

- Magnification Range: Initial low-magnification (50x to 100x) images are taken to provide an overview of the textile structure. These are followed by higher magnification images for detailed defect analysis.
- Imaging Angle: Tilted views (45° to 70°) are used to capture three-dimensional aspects of the defects, particularly for fibers showing fracture or delaminating.
- Sample Size: Each textile sample is imaged at multiple locations to ensure representative results, especially for heterogeneous materials like composites.

3. Defect Classification and Analysis

The defects observed through SEM are classified based on their morphology, location, and probable causes. This classification helped in systematically identifying the root causes of defects and drawing correlations with specific manufacturing stages.

3.1 Qualitative Analysis

Defects are categorized into the following types based on SEM observations:

- Fiber Breakage/Fracture: A very clear-cut break, but sometimes with a thinning of the fiber on one side to suggest excessive mechanical stress in spinning, weaving, or chemical processing. It occurs in natural and synthetic fibers.
- Surface Contamination: Found to appear as rough layers on the surface or agglomerated particle deposits, contamination arises from either the finishing or dyeing stage. EDS has been applied to detect the chemical composition of the contaminants.
- Pilling and Fuzzing: Common in fabrics like wool and polyester, these surface defects are identified by the formation of small balls of fibers on the surface, often attributed to friction during wear or mechanical processing.
- Delamination and Debonding in Composites: It seemed in composite textiles where poor bonding between the fiber and the matrix resulted in real gaps or fiber pullout. Such defects are typically associated with inadequate resin penetration during composite processing.

3.2 Quantitative Analysis

- Defect Density: Based on the defects per unit area of the textile specimen, for example, defects/mm², defects were

calculated. These have been used to assess defects and estimate defect rates in different materials and under varying processing conditions.

- Crack Length and Width Measurement: The length and width of cracks in fractures are measured depending on in-build or external image analysis software, including ImageJ. The scale of fiber fractures is measured. It correlated measurements of crack size with the mechanical stress parameters of the manufacturing process.

4. Root Cause Analysis Framework

Once the defects are identified and classified, a root cause analysis framework is employed to trace each defect back to its origin in the manufacturing process.

4.1 Root Cause Hypotheses:

Based on the critical SEM observations, combined with the classification of defects, the following have been developed as hypotheses for the root causes of these defects: (i) Fiber Fracture: This is generally caused by an over-tension and uneven mechanical stress while spinning, weaving or chemical processing. (ii) Surface Contamination: It may be associated with the handling or exposure at pre and post-processing stages of finishing or packaging. (iii) Composite Delamination: This is caused by poor resin penetration and improper curing during fabrication of composites.

4.2 Process Data Correlation:

All the correlations done on manufacturing process data, including spinning tension, heat treatment temperatures, and chemical additives applied during the dyeing stage, matched defects that were found. Research proved a hypothesis of the root cause and identified various stages where potential improvements may be brought to the manufacturing processes.

4.3 Failure Mode and Effect Analysis (FMEA):

FMEA is used to prioritize the defects based on their severity, occurrence frequency, and detectability. This analysis provides a systematic approach for recommending preventive actions to minimize future defects.

5. Applications of SEM in Textile Defect Analysis

This section includes general conclusions from a few studies based on the analysis of SEM of textile defects using failure mode analysis. The result is categorized very systematically regarding the kind of defects identified in the test sample such as fracture in fibers, surface contamination, delamination of composite, and many more in the structure. Discussions of all these kinds of defects are based on their morphology using SEM, the number of defects occurring, and possible causes in the textile production process. Based on this, various previous studies used SEM for testing defects in textiles with a wide range of materials from natural fibers such as to natural materials like cotton and wool to synthetic polymers such as polyester and nylon. The following subsections will review some of the important studies that

applied SEM to analyze defects and provide insight into their underlying causes as well as possible mitigation.

5.1 Fiber Fractures and Breaks

Among the common defects in fiber materials, fiber breaks are relatively predominant, especially in high-performance fibers, as it is more prone to mechanical stress. A lot of work was performed with the aid of SEM for the investigation of the microstructure of broken fibers and the failure mechanism. For example, tensile fracture in polyester fibers was demonstrated using SEM studies that fractures were initiated from the irregular crystallization of fibers during spinning [8]. Like this, SEM imaging also found that natural fibers like cotton mostly undergo overstretching while spinning, thinning, and breakage of the fibers [9].

5.1.1 Natural Fibers (Cotton and Wool)

SEM imaging of cotton and wool fibers revealed several instances of fiber breakage, with the fractures characterized by sharp, irregular edges. The following observations were made:

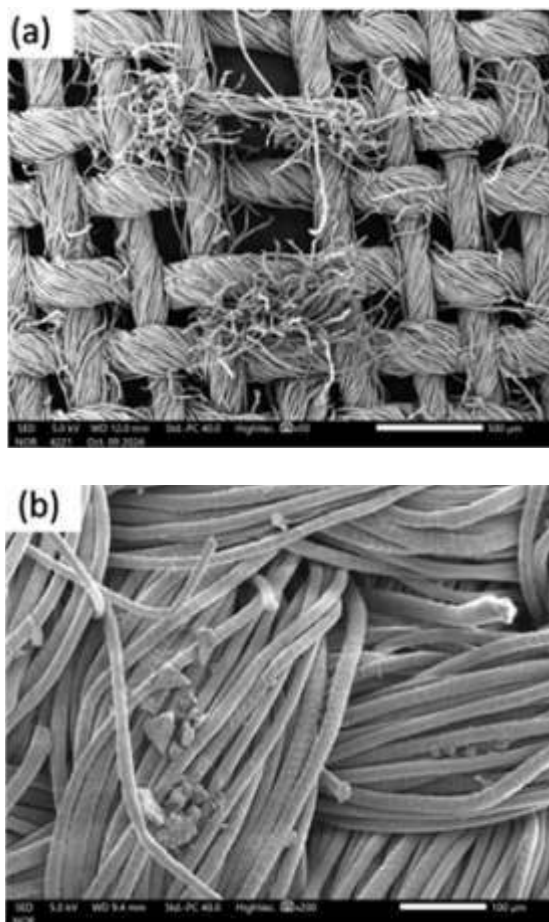


Fig. 1 SEM images of (a) Cotton showing stretched breaking and necking and (b) Wool fibers showing brittle breaking

- Cotton Fibers: The fractures occurred primarily at weak points in the fiber architecture and especially where

fibers had been overstretched in spinning. The fractures of all these fibers have a "necking" effect, which indicates high tensile stress. Broken fibers had rough surfaces, indicating a ductile fracture or localized chemical degradation.

- Wool Fibers: Fractures are rougher in wool samples and indicate the breakdown of fibers through splitting at breaking sites. Furthermore, the wool fibers have been evidenced of degradation as their fraying and thinning near breaking points are found. In wool samples, fractures occur with higher concentrations.

Root Cause: The main cause of fiber fractures for both natural fibers was established to be overstressing mechanically, owing to spinning itself. Firstly, twists and tension imparted to fibers in the process of spinning probably happened beyond tensile strength and broke fibers. Further on, at the weaving, the fault in control of tension continued piling up local stresses. The fibers derived from cotton and wool exhibited fractures characterized by brittleness, which are suggestive of mechanical overstress, a phenomenon typically associated with inadequate tension regulation throughout the spinning procedure. The cotton fibers manifested "necking" at their fracture locations, indicating that the applied tensile forces surpassed the fibers' ultimate tensile strength. This observation agrees with the findings since they pointed out that too high twist insertion during spinning may cause fibers to experience concentrated stress at certain points, thus breaking them down prematurely [10].

Mitigation Strategies: To minimize fiber fractures in natural textiles, careful optimization of the spinning parameters, such as reducing twists and controlling tension, is essential. Additionally, the introduction of intermediate relaxation treatments during the yarn formation process could help reduce internal stresses in the fibers. This approach could also be beneficial in reducing long-term fiber damage during weaving and finishing. Proper chemical handling also leads to the elimination of localized fiber damage.

5.1.2 Synthetic Fibers (Polyester and Nylon)

SEM analysis of synthetic fibers such as polyester and nylon provided insight into the different fracture mechanisms:

- Polyester Fibers: The polyester fibers showed brittle fractures with clean, straight breakage points. Some of the fibers had voids and micro-cracks near the breakage points which indicate the existence of internal defects at the time of polymer extrusion, which weakened the fibers.
- Nylon Fibers: The nylon fibers appeared more ductile by fracture characteristics; fracture points looked pulled out and stretched. Moreover, some of the nylon fibers also showed signs of thermal degradation: some had melted and thinned along the fracture boundaries of the fibers indicating high-temperature exposure during the processing or finishing operations.

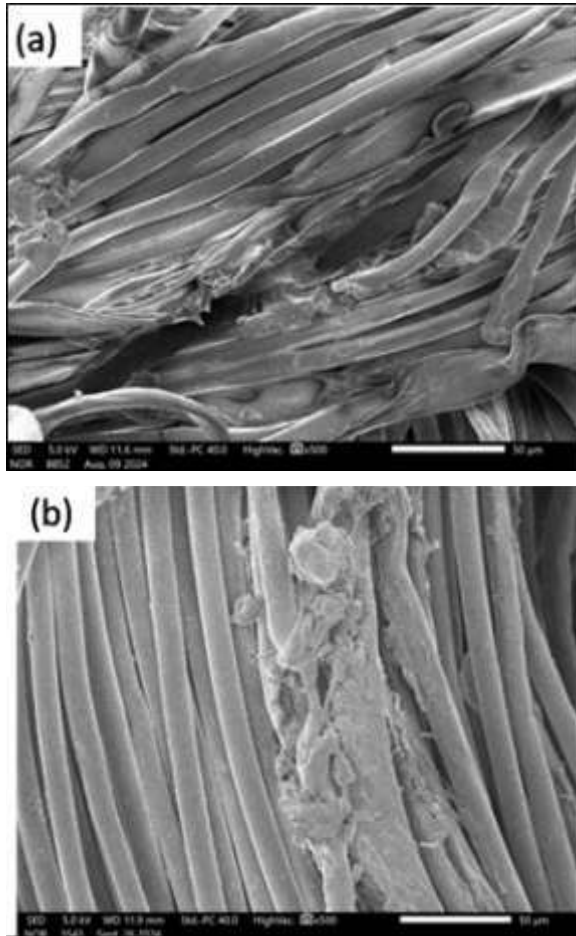


Fig. 2 SEM images of (a) Polyester and (b) Nylon fibers showing breakage and thermal damage respectively.

Root Cause: In contrast to natural fibers, synthetic fibers like polyester and nylon exhibit distinct fracture behaviors that reflect issues in polymer processing. Fiber fractures in synthetic materials are primarily linked to polymer processing defects, such as improper extrusion conditions and insufficient control of fiber cooling rates during the drawing process. High temperatures during manufacturing or finishing could also contribute to the thermal degradation observed in nylon fibers. Polyester fibers, for instance, showed brittle fractures with signs of microvoid formation, which indicates poor molecular orientation during the drawing process. It is also shown that improper drawing temperatures or excessive drawing speeds can lead to uneven molecular chain alignment, resulting in weaker fibers prone to brittle failure. Nylon, on the other hand, demonstrated more ductile fracture characteristics, with signs of thermal degradation, such as melted or thinned sections near the fracture points. This suggests that the fiber is exposed to high temperatures either during polymer extrusion or finishing, weakening the polymer chains. **Mitigation Strategies:** For synthetic fibers, optimizing the polymer extrusion and drawing process is critical. Ensuring consistent drawing temperatures and controlling the rate of fiber stretching can improve the molecular orientation of the fibers, resulting in improved tensile strength. For nylon, implementing lower

temperature limits during finishing could help prevent thermal degradation, especially in performance textiles where strength and durability are critical.

5.2 Surface Contamination and Impurities

Contamination on textile surfaces, such as dust, oils, or residues from processing chemicals, can significantly impact the appearance and performance of fabrics. SEM has proven useful in identifying these contaminants and tracing their origin. A study demonstrated the use of SEM in detecting silicone-based deposition on the surface of polyester [11]. SEM's ability to provide elemental composition data through Energy-Dispersive X-ray Spectroscopy (EDS) further enhances its capability to identify the chemical nature of contaminants, by SEM-EDS to detect the presence of metallic impurities in technical textiles. EDS analysis identified these particles as silicone-based residues, likely originating from the finishing agents used during the fabric treatment process.

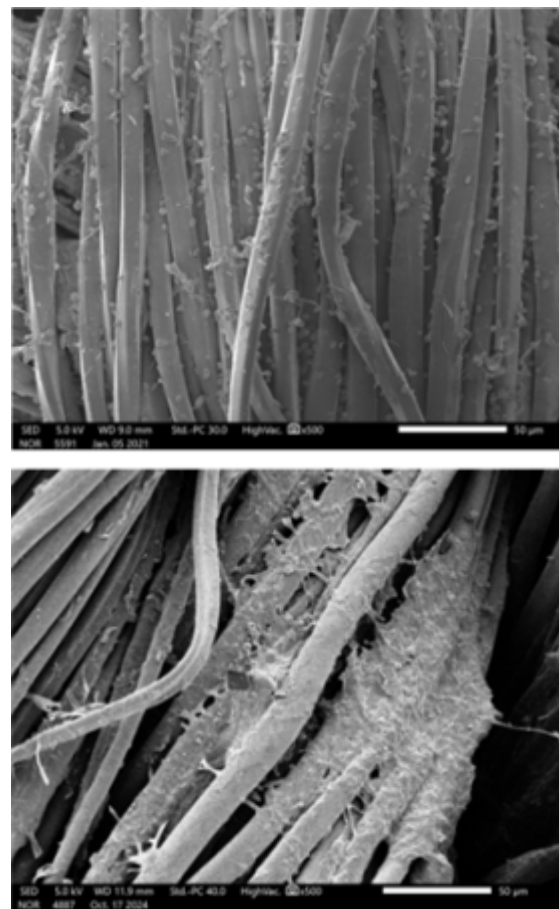


Fig. 3 SEM images of Polyester fibers showing surface contamination and impurities.

Root Cause: Surface contamination was traced back to the post-processing stages of textile manufacturing, specifically during finishing and handling. The silicone residues in polyester fabrics likely resulted from improper rinsing during the finishing process.

Mitigation Strategies: To reduce surface contamination,

especially silicone residues, it is essential to optimize the rinsing process during finishing. Increasing the duration and thoroughness of rinsing can help remove excess finishing agents. Additionally, using alternative, more easily removable agents may further reduce the likelihood of contamination.

5.3 Pilling and Fuzzing: Effects on Textile Wear and Performance

Pilling and fuzzing, the most apparent and damaging defects in textiles to be used as apparel or upholstery are generally found in wool and polyester fabrics [12]. They degrade the appearance and surface smoothness of fabrics and end up resulting in consumer dissatisfaction.

Cotton Fabrics: Pilling is due to the entrapment of free fiber ends at the surface of the fabric. Pills can be sized and it has been noticed that the number of pills differs highly with the density and lengths of fibers. In particular, in the case of cotton fabrics, pilling is distinctly present because of the intertwining of loose fibers existing at the surface. SEM images of the pills revealed that the pills contain an assemblage of short fibers, thus marking the failure mode that leads to the generation of defects under mechanical abrasion during wear.

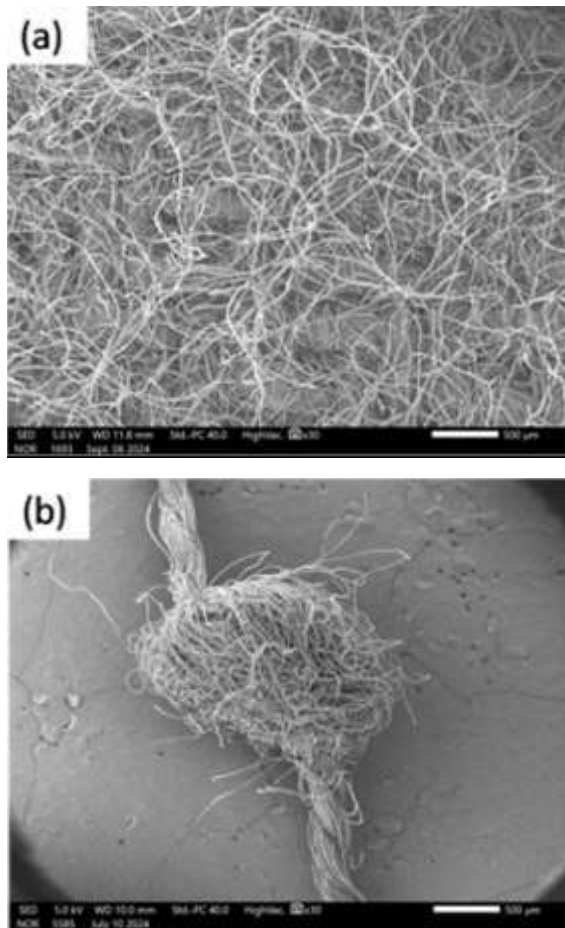


Fig. 4 SEM images of cotton showing (a) fuzzing on the surface of the fabric and (b) pill attached to the surface of the yarn

Root Cause: Pilling and fuzzing were primarily attributed to mechanical abrasion during use or processing. For cotton, the entanglement of fibers resulted from insufficient fiber cohesion during spinning.

Mitigation Strategies: Reducing the tendency of cotton fabrics to pill can be achieved by controlling fiber length and reducing the presence of short fibers during the spinning process. Additionally, chemical treatments that reduce fiber friction, such as enzyme treatments or the application of anti-pilling agents, could be applied to enhance the fabric's resistance to pilling.

Polyester Fabrics: Both pilling and fuzzing were demonstrated by polyester, and characterized by fine fibrils detaching from the main fibers to form small, round pills. The SEM images on polyester exhibit fine fibrils detaching from the main fibers. One of the primary reasons for pills formed upon the surface of polyesters was friction caused by wear or during washing. Polyester falls into the category of possessing a smooth surface with low friction making it even more susceptible to fibril formation.

Root Cause: In polyester fabrics, the low surface friction and elasticity of synthetic fibers made them particularly susceptible to pilling.

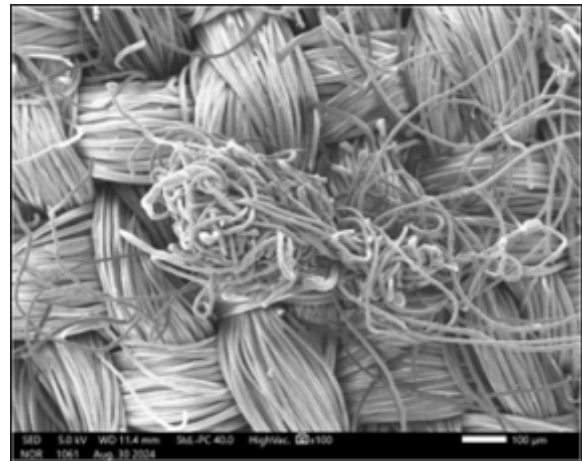


Fig. 5 SEM images of Polyester fibers showing pilling on the surface of the fabric.

Mitigation Strategies: In polyester, both pilling and fuzzing are decreased when the fibers undergo alterations to their surface properties by either a hot setting or an anti-pilling finish. Both treatments raise the possibility that fibrils may form because they increase surface friction and cohesion between the single fibers.

5.4 Defects in Composite Textiles: Delamination and Fiber-Matrix Debonding

For instance, in aerospace and medical applications the bonding at the interface between the fiber and the matrix is critical in maintaining the mechanical performance of the composite textile. Anomalies at the interface related to bonding can cause delamination or pullout of the fibers, those elements that compromise the integrity of the structure.

SEM has been considered as a method to observe bonding defects and their impact on performance. For example, SEM studied delamination in fiber-reinforced polymer composites and thus proved that the defects originated due to an improper resin filling at the production of fiber bundles [13].

Delamination in FR composites was one of the most significant defects observed using SEM. The following features were noted:

- **Cross-Sectional Imaging:** SEM cross-sections of the composite revealed clear gaps between the polyester fibers and the polymer matrix, indicating poor bonding between the fibers and matrix.
- **Fiber Pull-Out:** In some areas, polyester fibers were observed to have completely separated from the matrix, with SEM images showing individual fibers protruding from the surface.

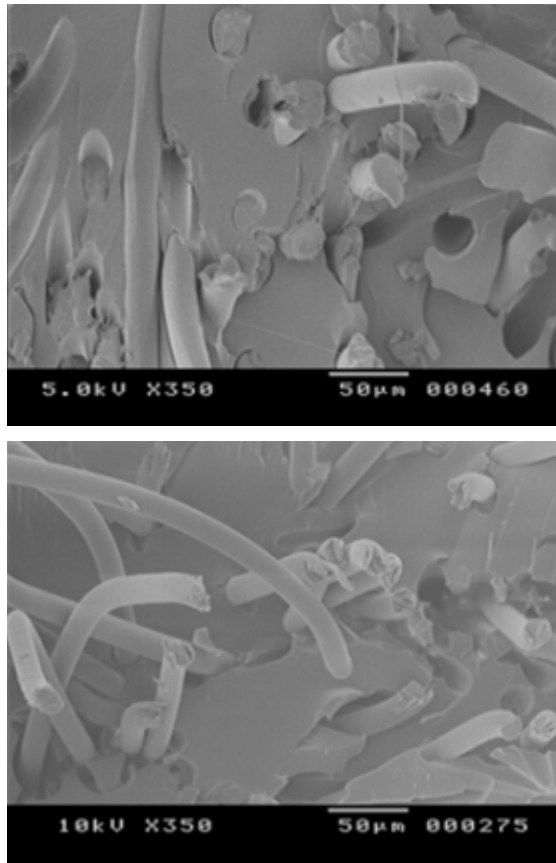


Fig. 6 SEM images of Polyester fibers reinforced epoxy matrix composite showing delamination and fiber-matrix debonding

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Root Cause: The following are the reasons for FRC specimen delamination: poor infiltration of resin in making composite, poor curing conditions, and uneven distribution of fibers. Poor application of heat during the curing process may have left the resin cross-linked incompletely. Stress concentrations caused by fiber misalignment weakened the adhesive fiber-matrix interface. Polyester fibers have badly been wetted by the polymer matrix, which has resulted in poor fiber-matrix adhesion; hence, delamination and further fiber pullouts are caused when subjected to load.

Mitigation Strategies: There should also be a resin infusion process that would prevent delamination. Optimum pressure and vacuum conditions can obtain the proper wetting of fibers by the matrix such that the creation of voids may be minimized. Coupling agents or surface treatment of carbon fibers can increase adhesion between the matrix and fibers such that it may reduce delamination. If the fiber alignment in the lay-up procedure is correct, then fiber-matrix debonding can prevent delamination. This can be achieved by using pre-impregnated fibers or through automated fiber placement techniques to minimize the likelihood of misalignment. Optimizing the curing temperature and time also ensures the attainment of full curing of the matrix and a strong bonding with the fibers.

6. Conclusions

The defects in textiles are discussed based on SEM-based root cause analysis, which reveals the mechanisms of various types of defects such as fiber fracture, surface contamination, Pilling and fuzzing, and composite delamination. Knowing the causes might be of benefit to the manufacturer by directed improvement in their processes that should lead to better performance and durability of the textiles. Very helpful to explore the roots of these defects, and analysis findings support suggestions to prevent the same possible faults in the improvements of the process. Analysis, therefore, in its most extensive sense opens up the prospect that SEM can function as a powerful tool in analysis and defect remediation in the textile industry to pave the way for better quality overall quality, durability, performance, and efficient production of textiles can be developed most.

Competing interests

The author(s) declared no potential conflicts of interest concerning this article's authorship, and/or publication.

Acknowledgments

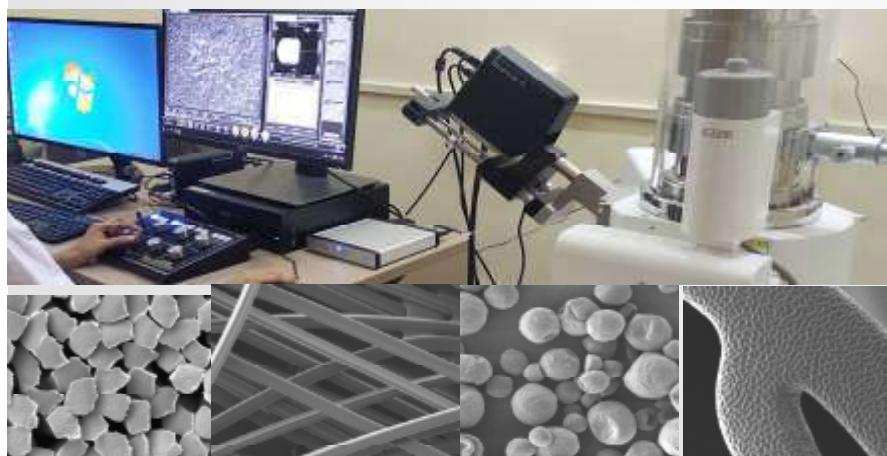
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Rheology of UHMWPE/ solvent systems: A literature review

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Abstract

The paper reviews the current available literature on the rheological behaviour of Ultra High Molecular Weight Polyethylene (UHMWPE)/ solvent systems. The rheological behaviour of UHMWPE /solvent systems was found to depend on the selection of solvent, shear rate, concentration of polymer, temperature and shear history. The influence of solubility on the rheological characteristics was discussed. The implication of the reports on gel spinning was also discussed.

Keywords:

steady shear viscosity, dynamic shear viscosity, viscoelastic properties, miscibility, gel spinning.

Citation

Sandeep Padmakar Jape - "Rheology of UHMWPE/ solvent systems: A literature review", *BTRA Scan* - Vol. LIII No.4, October 2024, Page no. 14-25, DOI: 10.70225/921685ptselh

1.0 Introduction:

Ultra-High Molecular Weight Polyethylene (UHMWPE) exhibits a number of exceptional properties, including chemical inertness, lubricity, impact resistance and abrasion resistance.[1]. As a result it has been receiving increasing attention in regards to its application in variety of fields which include biomedical[2, 3] and military[4].

In medical applications, it has been used as a bearing material in the manufacture of artificial joints. Its use in orthopaedics has been the subject of several reviews[5-7].

The extremely high molecular weight and poor melt flow property makes processing of UHMWPE extremely challenging[8]. For example, in the manufacturing process of artificial knee joints, UHMWPE powder is initially shaped into primary articles with either square or cylindrical geometries using compression moulding or plunger extrusion techniques. Subsequently, these articles are transformed into artificial knee joints through a turning process[8, 9].

The extrusion of UHMWPE products, including pipes, sheets, and bars, necessitates the use of specially engineered extruders that incorporate a significant quantity of organic compounds to serve as lubricants during the process[8].

In case of UHMWPE fibres, gel-spinning method has been employed as a means of processing UHMWPE due to the

fact that UHMWPE cannot undergo melt-spinning like traditional polymers without degrading prior to achieving flow [4]. As per Tam and Bhatnagar [4], gel-spinning processes process can be classified into two main systems: single solvent system and dual solvent system. In the single solvent gel-spinning process, a solvent like decalin is employed to disentangle UHMWPE polymer enabling its spinning through a spinneret with conventional melt-spinning apparatus. The fibre solution is then conveyed through an evaporation chamber, where the solvent is removed to form a gel fibre. During the following drawing process, the remaining solvent may be evaporated, enhancing the tensile strength.

An additional variation of this process involves the formation of gel fibres through the quenching of solution fibres within a liquid bath subsequent to their extrusion through the spinneret and an air gap. The gel fibre is then drawn in an oven to evaporate the solvent and align the polymer molecules, thus producing strong fibres[4].

In the dual solvent system, a low molecular weight solvent functions as the initial solvent, promoting the disentanglement of UHMWPE chains. After the extrusion, the solution fibre is quenched in a liquid bath, to form a gel fibre, which may be stretched. The solvent used in spinning is extracted utilising a second solvent with a low flash point. The initial solvent is thus replaced, and the fibre is drawn in several stages to optimise its mechanical properties.[4]. The manufacture of UHMWPE fibres via the gel spinning technique has been the subject of several studies.[10-18].

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The high performance of UHMWPE fibre is attributable to a combination of high strength (around 3.5 GPa) and low density, with a typical value of 970–980 kg/m³ [19]. The fibre properties depend on a variety of factors which include, molecular weight [12], gel concentration [13], L/D of extruder [10], spinneret hole size [14], winding speed [11, 15], spinning temperature [16], cooling bath temperature [17], & draw ratio [18].

In gel spinning, the role of the solvent is to disentangle the polymer solution while keeping a minimum number of entanglements for proper spinning of the solution. The peak draw ratio that can be attained, largely depends upon the concentration of the polymer solution. Therefore, the solvent type used in spinning and their concentrations are critical factors [20].

Among the solvents paraffin oil and decalin have been extensively used in industry [21] for gel spinning. There are also several reports where paraffin oil [11, 13-16, 22] & decalin [17, 18, 23] has been used in gel spinning UHMWPE. Other solvents used in gel spinning of UHMWPE include, kerosene [24]; mineral oil, decalin/ 1-dodecanol, lauric acid, stearic acid, peanut oil, olive oil [25]; Sunflower oil, palm oil, orange oil [26]; Polyalphaolefin Oil [27]; Polybutene [21]; camphene [28].

The rheology of polymer solutions can also have a significant impact on spinning processes. Rheological studies can be conducted using various techniques, including steady shear, dynamic shear, and transient tests. While steady shear testing provides insight into the material's viscosity under constant stressor strain, dynamic measurements offer information about the material's viscoelastic properties. Transient testing can be employed to examine the deformation behaviour of the material over time.

As mentioned previously, there are several reviews which deal with medical applications of UHMWPE [5-7]. There has also been a report on rheology of UHMWPE blends and composites [29], where there was a limited attention on rheology of UHMWPE solutions. This study reviews the existing literature on the rheology of UHMWPE/solvent systems, to facilitate understanding of UHMWPE flow behaviour during gel spinning.

2. Gel structure

In regards to gel spinning, it is important to note that UHMWPE does not form gels in conventional sense. In UHMWPE, the junction points of the gel networks are composed of crystallites unlike conventional type of gel network, where completely disordered chains are covalently linked together [30].

The gelation process of UHMWPE in solvents like paraffin oil and decalin can be explained by liquid-liquid phase separation, which is influenced by concentration fluctuations. These fluctuations occur as a result of the

polymer's crystallization during the cooling of the solution [20]. Gels formed by this mechanism are also thermally reversible [30]. Similar behaviour is noted for other systems such as isotactic polystyrene [31-33], & poly(vinylchloride) [34-37].

Chung and Zachariades [38] indicate that a UHMWPE solution in paraffin oil, with concentrations ranging from 2% to 8% w/w, form a three-dimensional molecular network with secondary bonds at the junction sites.

This can result in the formation of crystalline regions and the development of physical entangled structures with varying lifetimes. Trapped entanglements between crystallites have an apparent infinite lifetime, just like the crystallites. Non trapped entanglements have a transitory lifetime [38].

The parameters employed during the preparation process significantly influence the uniformity of the resultant pseudogels. Non-uniform, gel-like structure appears under non-isothermal conditions. In such cases, the gel is made up of single crystals and fibrils in a shish kebab-like structure, reaching several millimetres in length [39]. In contrast isothermal, quiescent environments, produce a more uniform gel consisting of aggregates of single crystals, and large spherulitic crystals [39].

Moreover, as per the model proposed by Pakshomov et al [40], during the solvent extraction process that leads to the formation of xerogel, the crystal morphology of UHMWPE evolves from a cracked crystal to layered structures composed of multiple coplanar lamellar crystallites. The morphology facilitates the formation of crystalline phase with a high degree of orientation in the process of fabricating high tenacity fibres. The existence of the crystalline cross linked structure in gels can play a significant effect in the flow behaviour of UHMWPE as will be seen in later sections.

3. Solution Preparation

Alekseev [12] demonstrated that the dissolution process of UHMWPE, occurs in two distinct phases: the initial swelling of the polymer followed by the dissolution of the swollen gel particles. When UHMWPE solutions are formulated in petrolatum, the swelling phase occurs at temperatures ranging from 110 to 120°C, while the dissolution phase takes place between 135 and 140°C. Alekseev noted that to achieve a uniform solution, it is essential to gradually heat a suspension of UHMWPE powder in the solvent while maintaining vigorous stirring. If the polymer is subjected to hot petrolatum or heated too rapidly, the swelling process becomes uneven, leading to the formation of aggregates that cannot be dissolved afterward.

Alekseev emphasized the significance of the design of the dissolving apparatus. He indicated that use of typical blade and spiral mixers, along with extruders can have undesirable aspects, including uneven dissolution of particles, dominance of the Weissenberg effect, and limitations in handling higher concentrations of polymers. To address

these concerns, Alekseev suggested the adoption of twin screw extruders and specially designed self-cleaning mixers.

There have also been patents [41] reported which have attempted to address the issues. Development of gel spinning equipment have also been the subject of several patents and has been reviewed elsewhere [42]. The patents involve the use of a solution preparation vessel, mixer with a pump to keep the solution homogenous, and a single crew extruder [43]; twin screw extruder with gear pump [44]; helical intensive mixer, in addition to stirring tanks to create a homogeneous solution before introducing it in extruder fitted with a gear pump [45]; stirring tank, helical mixer, extruder with gear pump and a solvent extraction & recovery system [46]. The challenge of creating a homogeneous solution with flow behaviour suited for gel spinning remain, in this regard rheology studies can play an important role in optimizing gel spinning process as well as design of equipment's.

4. Rheology of UHMWPE/ solvent systems

As stated in the introduction, the proper selection of solvent plays a critical role in gel spinning process. Due to differences in the solubility, it can have remarkably different effect on the rheology of UHMWPE solutions and thus have been the subject of several studies [20, 21, 28, 38, 47-56].

4.1 Steady shear rheology

Steady shear rheology which involves applying a unidirectional deformation under constant shear rate over time [57] is crucial for understanding the flow of UHMWPE solutions, under different shear rates. This is particularly important for processing techniques such as extrusion, where the material is subjected to varying shear conditions. The rheological properties, including viscosity and shear stress, are critical for optimizing these processes.

4.1.1 Steady shear viscosity

Jen and coworkers [28] determined the influence of solvent upon viscosity for 2 % UHMWPE solutions, using a Brookfield viscometer. The shear rate covered ranged from approximately $1-80\text{ s}^{-1}$. They found that the samples exhibited shear thinning characteristics and the solution viscosity was in order of decalin > paraffin oil > camphene; which as per the authors, suggested that camphene may be suitable for spinning as compared the other two solvents.

Chiu and Wang [47] studied the influence of temperature, gel concentration, and shear rate upon viscosity of UHMWPE/ decalin solutions in the shear rate range of approximately $0-10\text{ s}^{-1}$. For 6% UHMWPE solution, in the temperature range between $100-160\text{ }^\circ\text{C}$, they found that viscosity drops with increase in shear rate, especially at high shear rates. At $100\text{ }^\circ\text{C}$, a linear decline of viscosity with rise in shear rate was noted which was attributed to the fact that the temperature was below the gel point where phase separation had already occurred. At temperatures above $120\text{ }^\circ\text{C}$, UHMWPE solutions exhibited shear thinning behaviour for shear rates above 1 s^{-1} . At shear rates below $<1\text{ s}^{-1}$, viscosity was not

dependent on the applied shear. It was suggested that the samples undergo shear stress induced orientation, at low shear rates and are fully oriented above 1 s^{-1} which results in the decrease in viscosity. As the critical value of shear rate did not depend on temperature, it was suggested that the formation of the oriented structure was also independent of temperature.

In regards to effect of UHMWPE concentration (2, 3, 4, & 7%) on viscosity at specific temperature such as at $140\text{ }^\circ\text{C}$, and $160\text{ }^\circ\text{C}$, an increase in shear viscosity with concentration was noted at both temperatures. At $140\text{ }^\circ\text{C}$ a linear decline in viscosity with increase in shear rate was noted for 2% solution. This was suggested to be indicative of limited structural change in the course of the rearrangement process, as a result of low concentration. For higher concentrations such as at 6 %, viscosity did not depend on the shear rate below the critical value of 1 s^{-1} and decreased rapidly above it. Similar behaviour was noted for all concentrations at $160\text{ }^\circ\text{C}$; linear behaviour with shear rate was not observed in any samples.

Jian et al [48] a Brookfield viscometer to study the shear viscosity of UHMWPE / decalin solutions at various temperatures. The experiments were conducted for varying concentrations of UHMWPE at temperatures between $80-190\text{ }^\circ\text{C}$. They determined that, a particular temperature, the shear viscosities of gels exhibited a consistent increase with rising concentration above $80\text{ }^\circ\text{C}$. Additionally, it was observed that shear viscosity exhibited a modest increase with increasing temperature, particularly at temperatures below $110\text{ }^\circ\text{C}$. At temperatures exceeding $110\text{ }^\circ\text{C}$, the shear viscosities exhibited a pronounced increase with rising temperatures, attaining a maximum within the range of $120\text{ }^\circ\text{C}$ to $140\text{ }^\circ\text{C}$. The maximum solution viscosity temperature (T_{maxv}) rose from approximately $120\text{ }^\circ\text{C}$ to $140\text{ }^\circ\text{C}$ with the increase in solution concentrations from 7 to 18 kg/m^3 . Subsequently, the gel shear viscosities decreased notably when the temperatures exceeded their respective T_{maxv} .

It was suggested that at low temperatures (below $110\text{ }^\circ\text{C}$) only a fraction of the crystals were melted. In the solutions, low interpenetrating mobility resulted in a gel network with loose entanglements. As result a slight increase in shear viscosity was seen as temperatures rose.

They suggested that, in the solutions at temperatures higher than $120\text{ }^\circ\text{C}$ but not above $140\text{ }^\circ\text{C}$, a sizable amount crystals melt, giving UHMWPE chains sufficient interpenetrating mobility to the formation of a stable interconnected gel network. As a consequence, the gel solutions demonstrated maximum shear viscosity at temperatures between $120\text{ }^\circ\text{C}$ and $140\text{ }^\circ\text{C}$.

Furthermore, the reduction in shear viscosity above T_{maxv} was attributed to the total melting of crystals, subsequent partial solvation of UHMWPE molecules, in addition to potential thermal degradation. From the spinning experiments conducted using the same solutions, it was noted that

temperatures lower than 120 °C and concentrations lower than 4kg/m³ were found to be unsuitable for stable spinning. An optimum spinning processing window was suggested based on the viscosity data and spinning experiments. The viscosity window for spinning corresponding to the temperature range 120 to 180°C was 1000 to 1,000,000 cP

Yang and Chen [49] used Brookfield viscometer to study the shear viscosity of UHMWPE / decalin solutions (0.5-3%) solutions in the temperature range 125-165°C in the shear rate range of approximately 0.1 to 100s⁻¹. Additionally, the impact of introducing 1% aluminium stearate on the shear viscosity of UHMWPE/decalin solutions was investigated. They found that the solutions demonstrated a shear thinning response across the range of shear rates that were measured. In addition they noted that for 2% solution, viscosity could be described with a power law equation $\eta = m\dot{\gamma}^{n-1}$ where m and n are the consistency and flow index respectively. n did not vary with temperature indicating that the non newtonian behavior did not vary much with temperature. The flow index decreased with increase in concentration, tending to level off at concentrations higher than 1.5%. indicating that solution became more non-Newtonian at higher concentrations.

The effect of temperature on viscosity could be characterized by the Arrhenius–Frenkel–Eyring (AFE) equation $m = m_0 \exp(\Delta E/RT)$, where ΔE & R are the activation energy of flow, and gas content respectively. The consistency, designated as m , was defined as the viscosity at a shear rate of 1s⁻¹. A master curve was generated for all concentrations using the relation $\eta' = \eta m_s / m_T$, where m_s & m_T are the m at the reference temperature and temperature T respectively. From the parallel lines noted for master curves for all concentration, it was surmised that the activation was not strongly dependent on the concentration.

The concentration dependence of viscosity could also be fitted with a equation $m = kc^a \exp(\Delta E/RT)$, where c is the concentration; a & k are constants. A master curve was generated for both UHMWPE solutions with and without aluminium stearate by plotting m versus the shifted reciprocal of temperature T' obtained using the relation $1/T' = 1/T + (R\alpha/\Delta E)\ln(c)$. It was noted that the two master curves intersected at $T' = 300K$, and the addition of 1% sodium stearate lowered the viscosity of low concentration solutions, while increased the viscosity in high concentrated solutions. The activation energy was observed to be greater in the solution containing stearate than in those in which stearate was absent which was attributed to sodium stearate promoting greater polymer-polymer interaction while reducing polymer-solvent interaction.

Ohta and co-workers [50] conducted steady shear viscosity using cone and plate in the shear rate range from 0.5-100s⁻¹ for 3% & 5% UHMWPE/paraffin wax solution at 160°C. They found the solutions exhibited shear thinning behaviour over the entire range. At high shear rate, they also noted an abnormal brief increase in viscosity which was followed by the solution becoming expelled from the cone & plate gap

resulting in a decrease in the shear stress. The nonlinear rheological behaviour was attributed to the accumulated strain during steady shear mode.

Thus from the available literature it can be noted that the viscosity of UHMWPE solutions depend on the selection of solvents. Shear thinning behaviour is seen in UHMWPE solutions in decalin, paraffin wax, paraffin oil & camphene. In case of one study UHMWPE/ decalin solutions [47], shear viscosity was found to be independent of shear rate at low shear rate. This behaviour was found to depend on temperature and UHMWPE content. Additionally, it is important to highlight that the melting of the crystalline macrostructure, along with the resulting entanglements, significantly influenced the viscosity of UHMWPE/decalin solutions at a particular temperature. The shear viscosity of UHMWPE/decalin became non Newtonian with increase in concentration, in addition, the activation energy was found to be independent of UHMWPE concentration [49].

4.1.2. Evaluation of gel point using rotational testing

The viscoelastic nature of UHMWPE/ solvent systems solution undergo significant changes throughout the gel formation process during cooling [47]. Initially, the system behaves like a liquid until it reaches the gel point, when it transitions to a gel. As the gel cools below the gel point, phase separation occurs, removing the excess solvent. [47].

Chiu and Wang [47] determined the gel point of for UHMWPE/ decalin solutions (2-7%) during cooling using a constant stress of 100Pa and controlled cooling of 2°C/min. The authors identified two regions; a high temperature shear thinning region where the viscosity gradually decreased with decrease in temperature followed by a low temperature region where an abrupt increase in the viscosity was noted with decrease in temperature. The drop in viscosity with the reduction in temperature in high temperature region was linked to possible orientation effect due to applied stress. The gel point determined through this process displayed a linear increase with concentration, i.e. it changed from 102 to 108°C as the concentration changed from 2-7%. This was attributed to the increase in entanglement density with the increase in polymer concentration.

However in another experiments reported by the same authors [51] for UHMWPE/ decalin solutions (concentrations: 2-7%), the viscosity displayed a continuous increase with decrease in temperature. They determined that the gel point rises from 90.3 to 99.3°C as the UHMWPE content changes from 2 to 7%. The gel point was constant shear stress of 100 Pa., the cooling rate was not specified. The increase in the gel point with UHMWPE content was cross verified by assessing the gel melting temperature through Differential Scanning Calorimetry (DSC). Although the values were lower, a similar trend was noted.

Jen et al. [28] using Brookfield viscometer found the gel points of UHMWPE with decalin, paraffin oil and camphene solvents to be 76.6°C, 102.5°C and 82.2°C, respectively and suggested that the high gelation temperature made paraffin oil less desirable for processing.

The gel point determined using steady shear in the reported studies were found to significantly depend on the UHMWPE content in solution. Although gel points determined depended on the mode of measurement for e.g. rheometer or DSC, similar trends were obtained in terms of variation of gel point with polymer concentration. Higher gel points observed for UHMWPE in paraffin as compared to decalin and camphene. In regards to the differences noted shear viscosity trend with temperature in the high temperature region for UHMWPE/ Decalin, in [47]&[51]. It may be due to difference in thermal and shear history leading to structural changes. The extent of entanglements in solutions can be significantly influenced by both the thermal as well the shear history, thereby influencing the flow behaviour during the cooling as will be detailed in Section 4.3.

4.1.1.3 Effect on shear stress

There has been limited study on the effect on shear stress of UHMWPE solutions. Chiu and Wang [47] studied the influence of temperature, shear rate and UHMWPE content, and on the shear stress of UHMWPE/ decalin solutions in rotation mode using parallel plate geometry. The shear rate covered was between 0-10 s^{-1} . In regards to relationship between shear stress and shear rate for different temperature (100-160°C), for 6 % UHMWPE solutions, shear stress was found to gradually increase with shear rate at 100°C. At 120°C, and above a peak in the shear stress versus shear rate curve was noted at $1s^{-1}$ which was linked to process of formation of orientated polymer chains. The shear stress decreased thereafter with increase in shear rate above $1s^{-1}$ which was suggested to be due to lowered resistance to flow as the chains were fully oriented.

In regards to dependence of shear stress upon shear rate for different UHMWPE concentration (2, 4, 6, 7%) and at different temperatures (140°C, 160°C); the shear stress also was found to increase with increase in UHMWPE concentration at both 140°C and 160°C. which was considered to be an indication that shear stress needed for deformation increased with concentration. For all samples tested at 160°C, a critical value of the shear stress with noted with rise in shear rate for all compositions. For samples tested 140°C, 2% UHMWPE/ decalin solution exhibited a gradual increase in shear stress corresponding to the rise in shear rate. For 4-7% UHMWPE/ decalin solutions, a critical value of shear stress with respect to shear rate was observed. The value of the shear rate corresponding to the critical shear stress at both temperatures ranged between $1-10s^{-1}$ and followed no particular trend.

Thus studying shear stress as function of shear rate can be a good probe of development of oriented structure or structural development in UHMWPE, it can be used to predict instability in the flow behaviour as was noted in steady shear experiments conducted by Ohta and coworkers [50] mentioned previously which have implications for gel spinning.

4.2. Capillary Rheology

Capillary rheology can be an important tool for measuring

the flow behaviour of materials at shear rates beyond the range of the rotational rheometer, prior to testing their applicability at a commercial scale.[58].

Zhang et. al [52] compared the rheological behaviour of dilute solutions of 5% UHMWPE in decalin and paraffin oil using capillary rheology at 150, 170 & 185 °C. The shear rate covered ranged from approximately $1-2000s^{-1}$. They found that that UHMWPE/decalin solutions exhibited higher viscosity than UHMWPE / paraffin oil solutions especially temperature below 170°C and low shear rates, approximately below $100s^{-1}$ at 170°C. At high temperatures, the trend is reversed. Both solutions exhibited shear thinning behaviour. In addition, UHMWPE/decalin solution displayed higher activation energy than UHMWPE/paraffin oil, where the activation energy was calculated using Arrhenius relation. The authors suggested that the higher activation energy resulted in lower entanglement density at 185°C and that was responsible for the lower viscosity for UHMWPE/ decalin as compared to UHMWPE/paraffin oil.

They also noted that the activation energy of the solutions depended on both shear stress and shear rate which was different than other polymers such as polyethylene terephthalate.

Moreover, they observed a discontinuity in η vs. $\dot{\gamma}^{1/2}$ curve for 5% UHMWPE/ decalin solution at approximately $70s^{-1}$ shear rate, which was deemed indicative of a significant change in the nature of the entanglements within the UHMWPE solution.

Another study [53] investigated the influence of addition of 1% aluminium stearate upon the rheological behaviour exhibited by 5% UHMWPE/paraffin oil solutions at 170°C. The study was conducted using a specially designed instrument containing a 3 litre vessel, which was attached to a one hole spinneret of different geometries by means of a tube.

It was found that both the 5% UHMWPE solutions i.e. with and without inclusion of aluminium stearate exhibited shear thinning behaviour within the approximate shear rate interval of $1-1000s^{-1}$; the incorporation of aluminium stearate resulted in a reduction in shear viscosity. The impact of the geometric dimensions of spinneret orifices, including length-to-diameter ratio (L/D) and entrance angle of the capillary was also investigated for a 5% UHMWPE/paraffin oil solution with aluminium stearate. The length-to-diameter ratio (L/D) ranged from 2 to 15, while the entrance angle spanned a range of 6 to 60. From the pressure versus mass flow curves, for different L/D or entrance angle at 170°C, it was determined that increasing L/D ratio or reducing the entrance angle of capillary enhances viscous flow resistance. An analysis of the curves of the first normal stress difference ($\sigma_{11}-\sigma_{22}$) in relation to shear rate (ranging approximately between 5 and $20s^{-1}$), revealed an increase in $\sigma_{11}-\sigma_{22}$ as the shear rate increased. The values of pressure recorded at the entry and exit of the capillary were used to determine the Bagley-end correction 'e,' which was subsequently evaluated

in relation to shear rate. The authors noted that the value of 'e' increases with shear rate (range approximately 5-20s⁻¹). The authors remarked that as the Bagley-end correction 'e' and first normal stress difference are indicative of the elastic behaviour, the results reveal that the elastic contribution to spinning solutions in flow is quite pronounced, even at shear rates lower than 20 s⁻¹.

Ohta et al [50] studied the steady shear viscosity of UHMWPE/ paraffin wax solutions (3, 5%) using a capillary type viscometer at 160°C. Three spinnerets with capillary lengths of 10, 20 and 40 mm. and with equal diameters of 1 mm but different L/D i.e. 10, 20 and 40 were carried out. The Bagley plot [59] was utilized to correct the effects of capillary length, while the Rabinowitsch equation [60] was utilized to adjust the shear rate. The shear rate covered ranged was approximately 10-1000s⁻¹. They found that the viscosity increased with UHMWPE content; in addition, the solutions exhibited shear thinning throughout the measuring range.

Fang et al [21] determined the shear viscosity of 2 % UHMWPE / PB solution at 150°C within the approximate shear rate range of 0-1000s⁻¹. The solution displayed significant shear thinning effects, which could be described by a power law relation.

From the capillary rheology studies, UHMWPE solutions exhibit similar shear thinning behaviour as was noted in the steady state shear studies using Brookfield viscometers and rotational rheometers. The shear viscosity UHMWPE/ decalin solution exhibited higher solution viscosity as compared to UHMWPE/ paraffin oil at low temperatures, as was noted in Brookfield viscometry studies. However, at higher temperatures, this trend was reversed indicating that temperature dependence of shear viscosity must also be taken in to account during selection of solvents for spinning. The spinneret geometry was found to affect the flow behaviour of UHMWPE solutions. The high degree of entanglements resulting from the large molecular weight results in significant elastic effects even at low shear rates [53] which have implication in gel spinning.

4.3. Dynamic shear rheology

Dynamic shear rheology can be used to measure the viscoelastic properties of UHMWPE solutions under oscillatory shear conditions and is an important tool in characterizing its stability during gel spinning.

4.3.1 Complex viscosity of dilute solutions

Complex viscosity ($|\eta^*|$), obtained via oscillation testing, is a vital parameter that characterizes the flow behaviour of UHMWPE solutions, much like steady shear viscosity. Shi et al [20] determined the complex viscosity of 1% w/v UHMWPE solutions in decalin as well as paraffin oil while cooling it between 150 and 70°C at a frequency of 1 rad/s. They noted that, at temperatures less than 120°C, the viscosity of the solution was much greater for decalin than for paraffin.

Lee et al [54] studied the dynamic rheological properties of UHMWPE / decalin solutions. The complex viscosity for 2%, 6%, 10% UHMWPE/ decalin solutions were determined at three temperatures (110, 130, & 150°C) from frequency sweeps using 12% strain. They found the solutions exhibit a shear thinning behaviour for the entire frequency range. The viscosity-frequency curves followed a power-law behaviour given by $\eta = A \dot{\gamma}^n$ where A , n are a constant & the power law index respectively. The power-law behaviour was noted irrespective of the temperature and UHMWPE content, with the Newtonian plateau absent at low frequencies. Viscosity increased significantly with concentration, with a tenfold increase from 2 to 6%, and then from 6 to 10% at low frequencies at 110°C. The relationship between viscosity and temperature was represented by the Arrhenius equation $\eta = Ae^{E/RT}$. In the equation, A , E , R , and T represent a constant, the activation energy for viscous flow, the gas constant, and the absolute temperature, respectively. Linear Arrhenius plots was obtained for the 3 concentrations which was taken as an indication of absence of chain scission during testing. The activation energy increased with gel concentration which was taken as an indication that the temperature dependence of viscosity increased with gel concentration. A master curves of viscosity function versus frequency was generated for all three concentrations from the Arrhenius equation using the relation $a_T = \eta/\eta'_0 = e^{E/R(1/T - 1/T_0)}$ where T_0 is the reference temperature, a_T is the shift factor. The curves could be superimposed on a single curve with some deviation noted at high frequencies for the 10% UHMWPE/ decalin solution.

A heating/cooling temperature sweep of 2% UHMWPE solution from 110°C to 150°C was also performed at a rate of 2°C/min and at a fixed frequency of 1 rad/s. It was determined the viscosity initially decreased during heating from 100-130°C, which linked to lowering of the viscosity of the "medium" containing dispersed crystals; it kept constant from 130 to 150 °C which was attributed to formation of extensive physical crosslinks due to meeting of crystals. During cooling step the viscosity remained unchanged from 150 to 120°C, where its viscosity was similar to heating step. It subsequently increased dramatically after 120°C as a consequence of crystallization along with phase separation. For higher concentration (6%, 10%) the temperature hysteresis increased, i.e. the viscosity on cooling was lesser than on heating in the melt state which was attributed to persistence of oriented structures formed during heating.

Ohta and coworkers [50] determined the complex viscosity for 1, 3, 5 % UHMWPE/ paraffin wax solutions at 160°C in the frequency range 0.634 to 10.5 rad/sec. The viscosity increased with polymer concentration. They also noted that the samples exhibited shear thinning behaviour over the measuring range with a small kink in data which shifted to lower frequency with increase in UHMWPE concentration. The power law index (n) of viscosity was determined from a linear fit of the dynamic viscosity as a function of shear rate. The decrease in n values with concentration was considered indicative that the solution exhibits greater non-Newtonian behaviour. The 3 % and 5% viscosity results were combined

with results from steady shear and creep measurements using cone plate geometry, and the viscosity results from the capillary rheology studies. The viscosity data for the two concentrations from the measurements could be plotted on single curves corresponding to each concentration. It was suggested that as the shear viscosity data obtained under the steady shear almost coincided those obtained by the dynamic measurements, it indicates validity of the Cox Merz rule [61] in this system.

Thus complex viscosity, as in case of steady shear viscosity, was found to depend on polymer concentration, temperature and frequency of measurement and selection of solvent. As was noted in the steady shear experiments, the UHMWPE / decalin viscosity was found to higher than the UHMWPE/paraffin oil solution at temperatures less than 120°C. The complex viscosity dependence on frequency of UHMWPE / decalin, paraffin systems could be fitted with a power law relation. The activation energy of UHMWPE / decalin [54] was found to depend on the concentration of UHMWPE unlike report in [49] for UHMWPE / decalin system, where from the Arrhenius plots it was surmised that the activation energy was independent of polymer concentration. However, it should be pointed out that the study was performed for lower concentration range of UHMWPE(0.5-3%), in addition, activation energy values were not presented. A master curve could be generated for the viscosity function with frequency for the UHMWPE / decalin system [54]. The physical crosslinks formed by melting of crystals play a key role in the temperature dependence of complex viscosity.

4.3.2 Evaluation of gel point using oscillation testing

There have been some reports on evaluation of gel points of UHMWPE solutions using oscillation testing. Fang and co-workers [55] for UHMWPE/polybutene (PB) solutions (2,4&6%) determined the gel point using oscillation testing. The complex viscosity was noted as a function of temperature during cooling and was utilized to evaluate the gel point. The gel point generally increased with polymer concentration, the specific values were not presented. The results were cross verified by noting the melting behaviour of gel using DSC which showed an increase in melting temperature from 114.5, 116, 117.5 °C for 2, 4 & 6 % solutions respectively. For a particular concentration(4%), it was observed that the gel point exhibited an increase alongside the molecular weight of the solvent,, the authors stated that a similar trend was noted for the other concentrations. In another paper, the authors [21] cross verified the gel point obtained from oscillation testing during cooling from 150 to 100°C for 2% UHMWPE/ PB solution with the crystallization temperature obtained from DSC.

Thus as in the case of rotational testing, gel point was found to be dependence on the concentration of UHMWPE was confirmed. Moreover, the molecular weight of solvent was also found to influence the gel point, thus it is an additional factor to consider when selecting solvents for spinning.

4.3.3 Viscoelastic property of UHMWPE solutions using oscillation testing

The measurement viscoelastic parameters, such as storage and loss modulus, can play an important aid in optimizing the spinning parameters as the fibre formation process is significant influenced by the viscoelastic properties of the solution.

Fang et al[21] conducted an oscillation frequency sweep of a 2% UHMWPE/PB solution. The experiments were carried out at a fixed strain of 1% and 5%, keeping the temperature at 150°C. The found that the both storage and loss modulus increased with frequency. It was observed that at frequencies lower than 1 Hz, the loss modulus (G'') is greater than the storage modulus (G'), indicating a dominance of viscous characteristics in the solution. They asserted that the UHMWPE solution should be suitable for spinning in this frequency region by curtailing elasticity, thus allowing for the development of high-strength fibers. Furthermore, they indicated that in the high-frequency region (greater than 1 Hz), where the storage modulus exceeds the loss modulus, elastic effects prevail. Consequently, spinning in this domain may lead to significant die swell, potentially resulting in fibers with increased defects. In regards to variation with % strain, G' , G'' and viscosity were higher for sweeps conducted at 1 %, than 5% strain .

In another paper, Fang et al [55] investigated the viscoelasticity of the 2% UHMWPE/ Polybutene(PB) and compared it with UHMWPE/paraffin oil solution using frequency sweeps. They reported that, at a concentration of 2% UHMWPE and using solvents of comparable molecular weight, the spin dope containing PB showed enhanced G' , G'' , and $|\eta^*|$, at 150°C. This suggests that, under equivalent extrusion conditions, the UHMWPE/PB spin dope is more capable of yielding continuous filaments, and that further decreasing the concentration may improve the strength of the resulting fibre. For UHMWPE/paraffin, (G'), was higher than the G'' over the entire frequency range which would suggest that elastic nature was dominant in the frequency range. For UHMWPE/ Polybutene(PB) similar behaviour could be seen; In addition, a crossover point could be seen at the beginning of the frequency sweep. In the both cases the modulus increased with increase in frequency.

Chiu and Wang [47] studied the viscoelastic properties UHMWPE/decalin system. In the experiment, the viscoelastic properties of UHMWPE / decalin system at UHMWPE concentrations of 2%, 4%, 6%, and 7%, conducted at temperatures of 120°C, 140°C, and 160°C. Strain sweeps were performed from 1 to 100% and frequency at 2 Hz.

During strain sweep of 6% UHMWPE/ decalin system at different temperature, it was observed G' exhibits a plateau region at low strain levels. Subsequently, a significant decline was noted as strain increased, particularly at a critical point of approximately 45%.

They proposed that the molecular chains which orient themselves with increase in strain rates at low strain rates, form physical crosslink interaction force at the critical strain value. When the strain is increased beyond this point, the polymer chains will be drawn out of the entangled area and start to slide. As a result, the crystalline region starts to break down, which causes G' to drop dramatically.

Furthermore, it was noted that the G' values achieved during strain sweeps at 140°C were greater than those obtained at 120°C and 160°C. They attributed it to the gel forming a shish kebab structure at the temperature due to the stress-induced crystallization. G' at 120°C was lower than at 160°C. The authors proposed that the incomplete formation of crystals at 120°C resulted in a low G' . Moreover, they suggested that at 160°C, which is higher than the crystals' melting point of 147°C, the crystal domains are destroyed, resulting in a lower G' value.

Furthermore, during the strain sweep conducted at various UHMWPE concentrations at 140°C, it was noted that as the content of UHMWPE increased, the critical strain value shifted to higher ranges. Additionally, the storage modulus (G') exhibited an increase with rising UHMWPE content. The authors attributed the increase in the critical point to presence of a greater number of entangled crystals at higher concentrations which cannot be easily pulled out from the entangled region thus contribute to increase in the critical point.

Chung & Zachariades [38] conducted dynamic frequency and temperature sweep experiments for 2-5% UHMWPE/paraffin oil system (referred as pseudogels). During the frequency sweep on 4% UHMWPE / paraffin oil gels at 25°C, it was noted that the G' and G'' were frequency dependent. G' exhibited a decline in the low frequency range, subsequently reaching a plateau in the intermediate frequency range, before experiencing a rapid increase in the high frequency range. G'' exhibited similar trend with respect to frequency. In addition the modulus also varied as the number of consecutive runs .i.e. the G' and G'' exhibited lower values at from second run onwards, with loss modulus exhibiting lower shear sensitivity. This shear history dependence of the viscoelastic properties was less pronounced at 0°C which was linked to low mobility of the chains. The effect of entanglements with short life times (from section 2) was linked to the initial decrease in G' at low frequency and overall decrease in G' in subsequent scans. The plateau region was associated with the presence of crystals and the confined entanglements in among them, which collectively exist in a state of pseudo-equilibrium. This state depended on the gel concentration, temperature, frequency, and the shear history. The authors reported that similar behaviour was noted for the other concentrations, data was not shown.

Moreover, it was observed that the dynamic shear modulus of 3% UHMWPE/paraffin oil gels exhibited a hysteresis response when subjected to thermal cycling from -20°C to 100°C. It was observed that during the heating cycle ranging from -20 to 110°C, the storage modulus (G') showed a

significant decrease as the temperature increased to -10°C. The authors indicated that this finding is contrary to the expected behaviour of cross-linked gel systems, which typically exhibit a constant G' . The observed behaviour was linked to a rise in the mobility of temporary entanglements. As the temperature beyond 60°C, G' further decreases sharply due to deformation of crystals resulting in reduction of trapped entanglements. The authors assert that this process results in a reduction of the overall level of entanglements by the conclusion of the heating cycle.

During cooling G' initially exhibited similar value as the heating cycle. Upon further cooling, G' values exhibited a rapid decline, ultimately reaching a minimum and increasing thereafter reaching a modulus value much lower than what was noted at the beginning of the heating cycle at -20°C.

The initial decrease was once again associated with the disentanglement effect observed at elevated temperatures, where the crystals were deformable. The authors noted that this behaviour is contrary to most polymer systems where storage modulus exhibits higher values during cooling due to decreased chain mobility, it was suggested that the disentanglement effect offset the expected increase in G' with cooling. As the temperature was lowered below 40°C, it was suggested that the reduced deformability of the crystals, would lead to the entrapment of a greater portion of entanglements thus contribute to resisting the decline of storage modulus. In addition, it was proposed that the reduction of temperature to -20°C led to a significant decrease in chain mobility, which subsequently resulted in an increase in the G' . The final G' value measured during the cooling phase did not attain the initial value noted during the heating phase. This discrepancy was attributed to the permanent loss of some confined entanglements throughout the thermal cycle, leading to an irreversible reduction in G' .

They also observed that the hysteresis behaviour of the G'' was less pronounced. Additionally, the researchers reported that during the temperature sweep ranging from -20°C to 100°C, there was a notable increase in the storage modulus (G') with increasing concentration. This increase was associated with a higher density of entanglements and an elevated level of crystallinity in the more concentrated solutions. In addition consistent decrease in the G' could be seen with increase in temperature which may due to increased mobility of the chains as mentioned above.

Qiao et al [56] studied viscoelastic properties of UHMWPE swollen by petrolatum in 1, 4, 10% concentrations. Dynamic time sweeps were performed for 60 min at 150°C with 10% strain. They found that $G' > G''$ storage modulus was higher than the loss modulus without any change over time which was taken to be an indication of indicating the stability of the petrolatum. G' decreased with increasing solvent content which was considered to be an indication that the elasticity and strength of UHMWPE was reduced.

On carrying dynamic temperature sweeps in the range 140-180 °C, the authors reported that the modulus has a weak

dependence on temperature, the data was not presented. The authors concluded that UHMWPE flow properties have a weak dependence on temperature.

For the three concentrations, dynamic strain sweeps were performed across a strain range of 0.1% to 100%, with the frequency held constant at 10 rad/s. They found that with the increase in strain, G' , after initially remaining constant, decreases after a critical value which taken to be an indication of breakdown of the macrostructure at the non-linear strain. G' also decreased with the rise in petrolatum content which was attributed to the increase in the degree of disentanglement with petrolatum content thus promoting reduced elasticity.

Furthermore, the storage modulus decrement was determined using G'_0 i.e. the plateau storage modulus in the linear strain region and G'_1 i.e. the decreased storage modulus at a certain nonlinear strain using the relation, $(G'_0 - G'_1)/G'_0$. As the nonlinear strain increased and the petrolatum content decreased, there was a corresponding rise in the values of storage modulus decrement. It has been suggested that reducing the quantity of petrolatum or increasing the proportion of UHMWPE in samples could result in diminished chain flexibility. This decrease in flexibility may subsequently increase the degree of macrostructural damage, leading to elevated values of storage modulus decrement.

The authors conducted dynamic frequency sweeps ranging from 100 rad/s to 0.1 rad/s at a temperature of 150°C for the three concentrations. It was noted that both G' and G'' decrease with the increase in petrolatum content as was noted in dynamic time and strain sweeps. Both G' and G'' showed an increase with increasing frequency, with G'' showing less of a frequency dependence at high UHMWPE content. At lower frequencies, G'' was found to be greater than G' ; however, as frequency continues to increase, a crossover point is observed between G' and G'' . The crossover point ($G' = G''$) which was denoted as the gel point signified the transition from viscous to elastic behaviour. The disentanglement time, denoted as t_d , and derived from the inverse of crossover frequency, ω_c , was associated with the relaxation time of a segment situated within two entanglement points. The data revealed that as the levels of petrolatum increased, there was a corresponding decrease in relaxation time and a lower crossover point modulus. Consequently, it was concluded that the addition of petrolatum caused improved chain disentanglements, enabling the segments to relax more quickly. It was suggested that the process led to the formation of more loosely held chain networks, which in turn resulted in a decrease in the elastic modulus.

Ohta and coworkers[50] studied the frequency dependence of 1,3,&5 % UHMWPE paraffin wax dilute solutions at 160°C. The frequency range was tested was between 0.634 to 10.5 rad/sec with 5% strain. They found that G' and G'' increase with frequency, and also increased with UHMWPE content. While the authors did not elaborate on the trend, it is

evident that the 1% and 3% solution curves showed a higher G'' compared to the G' at the low values of frequency, with a crossover point emerging as frequency increased. This crossover point was observed to move to lower values of frequency with the rise in UHMWPE content. For the 5% solution, the crossover point is positioned very close to the onset of the frequency sweep, making it difficult to determine accurately.

In addition, the authors noted the linear Han plots of $\log G''$ versus G' shifted with increase concentration. As the Han plot generally do not show and concentration or temperature dependence in miscible or compatible systems; however they show deviation from linear behaviour due to polydispersity, it was suggested that the shift in Han plot with concentration was due to wider polydispersity by considering paraffin as low molecular tails of polyethylene. In addition they noted that the plots exhibited a slope greater than $\frac{1}{2}$ which was also taken as evidence of effect of polydispersity as was reported by Han & Kim[62].

From the results, it can be noted that the viscoelastic properties such G' and G'' of the UHMWPE solutions depend selection of solvent and the concentration of polymers. It can also depend on the, strain rate, frequency, as well as the temperature of measurement. While in regards to frequency, G' in particular tended to continuous increase with frequency at higher temperatures when the sample are in melt state as was noted for the UHMWPE/ PB [21, 55], petrolatum[56], and paraffin wax systems[50]. At lower temperatures, the entanglements confined by crystals can cause G' values to remain constant for a particular frequency range, as was noted for the UHMWPE/ paraffin oil system at 0 and 25°C [38]. The viscoelastic properties are also shear history dependent due to the effect of trapped entanglements between crystalline regions. The dependence of the modulus on the shear history can be less pronounced lower temperatures. G' was more sensitive to the shear history than the G'' . In regards to effect strain rate it can be noted that for UHMWPE /decalin [47], paraffin wax [50] & petrolatum systems [56], the G' significantly reduces with the rise in strain above a critical value due to breakdown of the entanglement network.

It can be noted that the G'' was higher than G' at low frequency for many systems, this trend was revised at higher frequencies, with crossover point exhibiting a dependence on concentration of UHMWPE.

In regards to solvents, it can be noted that the UHMWPE/ PB exhibited higher G' and G'' as compared to UHMWPE / paraffin oil which may be the result of higher degree of entanglements. It is also important to note that during temperature sweeps while G' of UHMWPE/ petrolatum system shows weak dependence with temperature, G' of UHMWPE/decalin gels decrease with increase in temperature[38]. The difference may be due to differences in degree of disentanglement by the solvent. In the UHMWPE/ petrolatum system, chains being more tightly held similar to a covalently bonded gel network system.

4.4. Creep recovery tests

The creep recovery test is an important tool to measure response to applied stress over time. Ohta and coworkers [50] carried out creep recovery experiments for 3 & 5 % UHMWPE / paraffin wax at 160°C. The applied creep stress was varied from 0.5 to 30 Pa.

While the steady shear viscosity was obtained from the linear fit from the strain time curves. The creep compliance $J_e(\sigma)$ was calculated from the relation: $J_e(\sigma) = S_r / \sigma$ where S_r is the recoverable strain and σ is creep stress.

It was noted that steady shear viscosity showed shear thinning behaviour in the shear rate window of 10^{-4} to 10^{-2} s^{-1} i.e. the Newtonian plateau was missing even at low shear rates. The shear viscosity also increased with UHMWPE concentration.

The authors also determined that steady shear viscosity was related to the concentration through the relation $\eta(\dot{\gamma}) = aC^\alpha$ where α , C are the power law index and the concentration respectively. Using the shear viscosity values of the 3 & 5% solution, α was determined versus shear rate. Upon plotting α against the shear rate, it was determined that the value of α lies between 3.4 and 3.5, as the shear rate tends toward zero.

They also studied the creep stress dependence on $J_e(\sigma)$. The creep compliance for each concentration was almost constant for the creep stress range with the 3% UHMWPE./ paraffin wax solution displayed higher values of $J_e(\sigma)$ than the 5 % solution.

By interpolation of $\sigma = 0$, the zero shear creep compliance J_e^0 of 3 % & 5% UHMWPE/ paraffin wax solution was determined to be 10^{-11} Pa^{-1} and $3 \times 10^{-2} \text{ Pa}^{-1}$ respectively. After using the relation of the concentration dependence of creep compliance $J_e^0 = J_e^{00} \phi^{-m}$. The J_e^{00} , the creep compliance of non-dense system of the UHMWPE, was determined to be 10^{-5} Pa^{-1} which was close to an earlier report value of 10^{-5} Pa^{-1} for monodisperse high molecular weight polystyrene [63]. The m value was determined to be 2.4 ± 0.2 . It was suggested that the large values and recoverable strain of UHMWPE solution was a result of extremely large molecular weight between entanglement points (M_e).

5. Conclusions

From the available literature, it appears that among the commercially utilized solvents in gel spinning i.e., decalin and paraffin oil, decalin exhibits higher solvent viscosity at low temperatures below 170°C which suggests that UHMWPE is more readily disentangled by paraffin oil. However, as UHMWPE solutions in decalin have a greater temperature dependence than those in paraffin oil, the trend is reversed at higher temperatures.

In experiment conducted by [21], it was observed that during the crystallization of UHMWPE gels containing decalin, solvent was expelled from the gels. Conversely, the gels with paraffin oil exhibited no substantial solvent loss, while those with PB experienced a moderate loss of solvent. It was attributed that to the fact that paraffin oil exhibits greatest

affinity with polyethylene due to their similar alkyl main chains although the chain lengths differ [20]. The authors also noted that high level of interaction would make the process of extraction of solvent more difficult during gel spinning process.

The gel point observed during cooling under both rotation and oscillations testing were found to depend on the concentration of UHMWPE. The type of solvent used for the disentanglement process also affected the gel point. Gel points were higher in case of paraffin oil than decalin and camphene.

A broad viscosity window in the range of 1000 to 1000000 cP corresponding to the 120°C to 180°C was reported to be suitable for gel spinning for the UHMWPE/ decalin system.

It was determined that both the steady shear and dynamic shear viscosity are dependent upon the shear rate and the frequency at which measurements are taken. In regards to UHMWPE/PE-Wax [50] system both steady shear and complex viscosity appears to coincide with which would suggest that Cox-Merz rule may be valid for the system.

In general, shear thinning behaviour was noted in both steady state and dynamic shear for UHMWPE systems especially at high shear rate and could be modelled using power law equation [49, 50, 54]. The temperature dependence of viscosity could be modelled with Arrhenius equation [49, 52, 54]. The activation energy was found to depend on the concentration of UHMWPE except in [49].

Aside from the findings presented in [47] regarding UHMWPE/decalin solutions, the majority of cases exhibited a lack of a Newtonian plateau at low shear rates. The lack of a Newtonian plateau at low shear rates would render the startup process for gel processing highly challenging. [54]. The presence physical crosslink between crystals was found to greatly affect the viscosity of UHMWPE at different temperatures. The concentration dependence of shear viscosity could be described by power law type equation for UHMWPE / decalin [49] and UHMWPE / paraffin wax system [50]

Capillary rheology studies have shown that changes in spinneret geometry, including enhancing the length-to-diameter ratio (L/D) or lowering the entrance angle of the capillary, promotes to a greater resistance to viscous flow. The viscoelastic properties such as storage and loss modulus of UHMWPE / solvent systems determined under oscillation tests depended on the type of solvent, polymer content; shear history; temperature and frequency of measurement. The presence of entanglements between the crystalline regions also played a significant role in the viscoelastic behaviour of UHMWPE in terms of frequency and temperature.

6. Implications for gel spinning:

From the reports it can be surmised that the use of capillary rheology studies remains a useful tool in optimizing the processing parameters due to the fact that the shear rates that are measured in capillary rheology are closer to the shear

rates observed in spinning. Miscibility of solvent with the polymer remains important parameter for determining the level of disentanglement as well as the ease of extraction of solvent from fibres. Measurement of viscoelastic properties such as storage and loss modulus through parallel plate rheology also play a guide in diagnosing issues such as non-formation of continuous filaments. In this regard, there are reports where the dynamic rheological properties have been

used to predict the melt spinnability of polymers[64], and PAN/ CNT systems[65] using experimental spinning data. However, there have been no reports which validate the predications made in viscoelastic property studies with experimental spinning behaviour for UHMWPE. It may also be due to the complex rheological behaviours which occur during spinning and remains a challenge for the future.

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A Comprehensive Review of High Performance Synthetic Fibers for Composite Materials & its Application

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Abstract

Synthetic fibers, engineered through chemical synthesis, differ significantly from natural fibers derived from living organisms like plants and animals. These fibers are a product of extensive research aimed at mimicking the properties of natural fibers, involving processes such as extruding materials through spinnerets to form fibers. The widespread application of synthetic fibers spans various fields of fiber and textile technology, reflecting their integral role in modern material science. However, their production and use also contribute to environmental issues, such as micro plastic pollution from laundry machines. This review explores the evolution and impact of synthetic fibers, particularly focusing on their reinforcement capabilities in polymer matrix composites (PMCs). These composites are favoured for their lightweight properties coupled with enhanced stiffness, modulus, and strength, making them ideal for numerous industrial applications.

This review emphasize the significance of synthetic fibers in advancing composite material technologies and highlights the ongoing challenges and future research directions aimed at mitigating environmental impacts while enhancing material properties.

Keywords:

Synthetic Fibre, Fibre Reinforced Composites, Properties, Application

Citation

Omkar Sadalkar, Shital Palaskar, "A Comprehensive Review of High Performance Synthetic Fibers for Composite Materials & its Application", *BTRA Scan*-Vol. LIII No. 4, October, 2024, Page no. 26 to 31, DOI: 10.70225/466534mweqma

1.0 Introduction

Synthetic fibers (SFs) are becoming increasingly significant in fiber-reinforced composite structures worldwide. Due to their lightweight nature and distinct properties, SFs are highly valued for their exceptional characteristics [1, 2, 3]. They are extensively used in developing innovative composite materials for the automotive and aerospace sectors, prized for their outstanding strength and stability [4, 5].

The exploration and application of synthetic fibers are more crucial than ever, as their role in composite reinforcement has revolutionized various industries, including sports equipment and aerospace. Fiber-reinforced composites, for example, provide innovative solutions to previously unsolvable challenges, showcasing a compelling blend of materials science and engineering [6]. This introduction highlights the importance of these fibers, emphasizing their role in advancing composite materials that meet the stringent demands of modern, high-tech applications. Reinforcements in polymer matrix composites (PMCs) are indispensable for bearing structural loads, requiring compatibility with the

matrix, the ability to form chemical bonds or adhere effectively, superior properties to the matrix, optimal orientation, and appropriate shaping [7, 8].

This review article seeks to provide an in-depth exploration of high-performance fibers, examining their various types, properties, and applications within composite materials. It will start with a look at the range of synthetic fibers utilized for composite reinforcement, analyzing their main characteristics and the benefits they offer over natural fibers. Following this, the article will delve into the different manufacturing techniques for composites, emphasizing how these processes affect the final product's performance. Additionally, recent research advancements and emerging trends in high-performance composite applications will be discussed, offering valuable perspectives on the continuous innovations within this field.

2. Types of Synthetic Fibre:

Designed for high-performance applications, synthetic fibers come in a range of materials, each with special qualities and applications. High performance synthetic fibres (HPSF) are designed to satisfy particular needs in a

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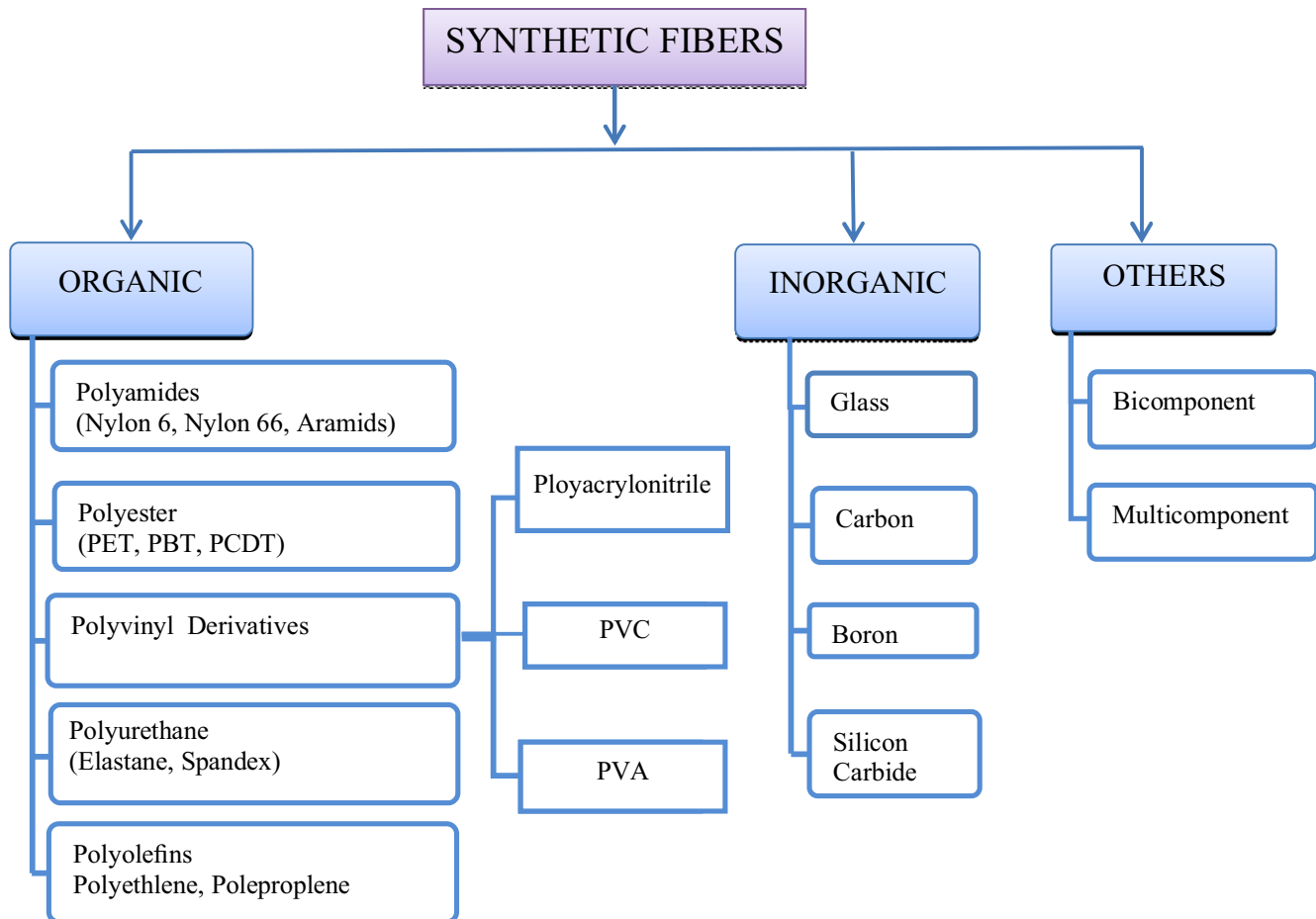


Fig 1. Classification of synthetic fibers.

range of industries because of their remarkable qualities, including conductivity, elongation, modulus, tensile strength, and environmental friendliness. For both traditional and high-tech applications, glass, carbon, aramides, polyolefins, and ceramic fibers are frequently utilized to reinforce composite materials [9,10,11].

3. Fibre properties & their application in composites:

3.1 Polyamides:

The research and manufacturing of textile composites heavily relies on polyamides, such as Nylon 6, Nylon 66, and Aramid, because of their special qualities and adaptability. These substances are used in many different applications, improving composites' durability and mechanical performance. High mechanical strength and flexibility are characteristics of polyamides. They continue to function at high temperatures, which is essential for applications where heat exposure is present. Because of their chemical resistance, polyamides last longer in a variety of settings [12,13]. Although polyamides have several benefits in textile composites, issues including production hurdles and cost concerns may prevent them from being widely used in some industries, including the military, sportswear, automotive, and aerospace [14].

3.2 Polyesters:

Polyester fibers are appropriate for a variety of uses in textile composites due to their diverse range of characteristics. Because of their great tensile strength, polyester fibers are perfect for structural uses in composite materials. Stretchable fabrics benefit from the latent crimping qualities of some polyester composites, which are enhanced by substantial thermal shrinkage. The structural integrity of the composite is improved by the mixture of polyesters with different inherent viscosities, which enables customized mechanical properties. In line with material science's sustainability objectives, several polyester composites are made to decompose naturally [15]. Polyesters are used in composite materials because of their high strength and low weight, which are essential for the automotive and aerospace sectors. Because of its affordability and mechanical strength, polyester composites are being utilized more and more in geotextile applications [16,17].

3.3 Polyurethane(Elastane,Spandex):

Polyurethane fibers, like Elastane and Spandex, are valued for their elasticity and are predominantly used in the fashion industry, particularly in the manufacture of stretchable clothing. These fibers provide comfort and flexibility, enhancing the functionality of everyday garments [18].

3.4 Polyolefins (Polyethylene, Polypropylene):

Polyolefins, including polyethylene and polypropylene. Polyethylene fibers, particularly ultra-high molecular weight polyethylene, are valued for their lightweight and abrasion-resistant properties. These fibers are used extensively due to their high strength and minimal moisture absorption, which are beneficial in both consumer products and industrial applications. Polypropylenes are known for their chemical resistance and are used in a myriad of products from packaging to textiles. These fibers are appreciated for their lightweight and durable nature, which makes them suitable for a wide range of applications [19]

3.5 Glass fibre:

Glass fibers offer a number of benefits that enhance concrete's functionality, particularly in construction applications. They improve the material's longevity, mechanical strength, and resistance to environmental effects when added to concrete formulations. Increased fiber content, however, may cause issues with flowability and workability.

Concrete's mechanical properties and durability are greatly improved by glass fiber reinforcement. It increases tensile strength to [20] in certain mixes and compressive strength [21] with the proper fiber content. Additionally, glass fibers improve the flexural strength of concrete, increasing its ability to withstand bending stresses. Strong alkali resistance, reduced water absorption for longer lifespan, and chemical resistance against a range of substances, including Portland cement byproducts, are all advantages of durability [22]. However, there are workability problems; higher fiber doses might result in increased water consumption and decreased flowability, which calls for the use of plasticizers. Both of these factors need to be managed to maintain optimal strength. Therefore, even though glass fibers significantly improve concrete's strength and durability, it is important to carefully analyze how they affect workability for optimal outcomes [23]. Glass reinforced polymer composites (GRPCs) are employed in a wide range of sectors due to its low cost, strength, and portability. GRPC is utilized in roof sheets, windows, sunshades, bathtubs, and other house and furniture storage racks. However, GRPC is widely used in aviation and aerospace for engine cowlings, luggage, containers, and gadget enclosures because other materials often better meet core aircraft needs. Because of its durability and low weight, GRPC is perfect for boat building, while contemporary resins help with water absorption problems. GRPC is widely used in the automotive industry for body frames, door boards, windshields, and other parts, frequently as an affordable substitute for metal assembly [24].

3.6 Carbon Fibre:

Carbon fiber is a desirable material in many high performance applications due to its exceptional qualities. Carbon fiber composites frequently outperform

conventional materials due to their remarkable mechanical qualities, which include high modulus and tensile strength. Modifications like acid treatment can improve their overall mechanical performance and impact resistance [25]. Carbon fibers are appropriate for high-temperature applications because of their stability in terms of thermal and electrical conductivity at high temperatures. They also have outstanding electrical conductivity, which is useful for applications that need shielding from electromagnetic radiation [26]. In order to lessen dependency on petroleum-based supplies while maintaining desired mechanical qualities, research on bio-based carbon fibers made from lignin and cellulose is progressing [27]. Since carbon fibers are an excellent material with high strength, stiffness, and low weight, the aerospace industry is using them extensively. They came to the conclusion that carbon fibers could offer corrective solutions for a wide range of issues related to the strengthening and degradation of infrastructure [28]. Application of carbon fiber composite material to make printing press and printer rollers, corrosion-resistant materials, and shielding materials [29]. The composite material that has the biggest influence on high speed, safety issues, and fuel efficiency is employed in the manufacture of cars. They came to the conclusion that carbon fibers are lighter than traditional materials and may be more advantageous for production that is more economical [30].

3.7 Silica Carbide Fibre (SiC):

SiC fibers' strong performance across a range of parameters is demonstrated by their mechanical characteristics. The ultimate tensile strength and elastic modulus of SiC fibers are both average [31]. They have outstanding thermal stability, retaining their mechanical integrity even under oxidizing and mechanical stress circumstances at temperatures as high as 1200 degrees Celsius. They can also be used for extended periods of time in hot conditions due to their improved creep resistance [32]. SiC fibers often exhibit superior tensile strength and toughness in comparison to other ceramic fibers. They are also resistant to heat shock, but other ceramic fibers might be more prone to losing strength when exposed to heat [33]. Due to their exceptional strength and thermal stability, silica carbide fibers are well suited for high-temperature applications, including turbines for power production, spacecraft, burners, nuclear applications, and airplane engines. When it comes to resilience to environmental conditions and longevity, these fibers provide notable benefits [34].

3.8 Bio Component Fibres:

Fibers that have two different raw material components in one fiber are known as bi-component fibers. Typically, a melt spinning machine is used to prepare bio component fibers. The most prevalent forms of bi-component fibers are segmented-pie (orange), islands in the sea (I/S), side-by-side (S/S), and core-sheath (C/S). The advantages of both components such as strength, hydrophilicity, affordability, etc. are combined in bi-component fibers. C/S bi-component

fibers are frequently utilized as bonding/binding fibers in the nonwoven sector. While micro denier fibers are produced by a very sophisticated extrusion known as a segmented pie fibers are usually used to create self-crimping yarns. Bi-component fiber is anticipated to have significant growth in the upcoming years. In order to address issues and satisfy consumer demands, this kind of fiber is anticipated to advance as a material in a wide range of end-use industries, such as textiles, automotive, home decor, and hygiene [35].

3.9 Multicomponent:

To get particular qualities, such high tenacity or fine denier, multicomponent fibers combine several types of polymers. These fibers are employed in sophisticated applications where certain qualities are essential, such as textiles and filtration [36].

4. Different Manufacturing Techniques for Composites:

4.1 Hand Lay-up:

The Hand Lay-up method is one of the oldest and simplest techniques used in the manufacturing of composites. It involves manually placing the fiber reinforcements into a mold and then applying the resin to saturate these fibers. This method is highly favored for its low cost and the ease of implementation, particularly suitable for large or complex shapes.

4.2 Vacuum Bagging:

Following the initial lay-up, Vacuum Bagging enhances the consolidation and adhesion of the composite materials by applying a vacuum to remove air trapped within the lay-up. This process not only improves the structural integrity but also reduces voids and ensures a more uniform thickness and resin distribution throughout the composite material.

4.3 Resin Transfer Molding:

Resin Transfer Molding (RTM) involves injecting resin under pressure into a fiber preform contained within a mold. This method allows for greater precision in controlling the fiber-to-resin ratio and can be used to produce complex geometric shapes with improved surface finishes and structural properties.

4.4 Low-Temperature Prepreg:

Low-Temperature Prepreg techniques involve the use of pre-impregnated fibers (prepregs), where the fibers are pre-coated with a partially cured resin. This allows for easier handling and more accurate placement. The curing process occurs at lower temperatures compared to traditional prepreg methods, making it suitable for applications sensitive to high temperatures.

4.5 Spray Lay-up:

Spray Lay-up is similar to hand lay-up but involves spraying the resin and chopped fibers simultaneously onto the mold. This technique is faster than hand lay-up and allows for a

quicker build-up of the composite material, making it ideal for large-scale productions or items requiring a thicker composite structure [37].

5. Application in Composite Materials:

5.1 Aerospace:

Synthetic fibers like glass and carbon are essential to the aerospace industry since they are used to make airplane parts. These fibers are included into important parts, such as the engine and fuselage, because of their exceptional strength-to-weight ratio and heat resistance. For instance, to ensure longevity, carbon fibers are woven in a linear sequence inside the fuselage, whereas aramid and Dyneema textiles reinforce the structure in places like the wheels, engine, and flaps. Synthetic fibre reinforced polymer (SFRP) composites provide lightweight, heat-resistant solutions with remarkable mechanical, tri-biological, and electrical qualities, which makes them ideal for long-lasting aviation materials [38,39].

5.2 Automotive:

Synthetic fibers are quite advantageous to the automobile sector, especially when it comes to lightweighting and reinforcing vehicles. Carbon fibers are used in a variety of auto parts to increase performance and fuel economy by reducing weight and increasing durability. The employment of these fibers in both non-structural and structural elements demonstrates their adaptability and improves the general functionality and safety of automobiles [40].

5.3 Medical Device:

Synthetic fibers are utilized in the medical field for a number of purposes, including as implants and prostheses, where their resilience to biological conditions and compatible with human tissue are essential. These materials aid in the creation of strong, lightweight medical equipment that can comfortably and functionally serve patients even under the most demanding conditions. Composite materials have become increasingly adaptable in healthcare due to advancements in synthetic compounds, surgical procedures, and sterilization practices. These days, several implants and devices are used in modern medical applications. Composites are widely employed in artificial hearts, pacemakers, dental implants, cardiovascular transplants, cardiac valves, eyeglass lenses, sutures, skeletal and joint replacements, biosensors, and more. In the end, these materials improve people's standards of life by supporting healing, repairing defects, and replacing or restoring the function of injured or deteriorating tissues or organs [41].

5.4 Sports Equipment:

Because there is a great need for materials that are both lightweight and durable, composite materials are widely used in sports equipment. Commonly seen in products like hockey sticks, bicycles, and tennis rackets, synthetic fibers like carbon and glass offer greater strength without

sacrificing weight. The performance characteristics of sporting equipment are greatly improved by this application, giving players better handling and better results overall [42].

5.5 Geo-textile:

Another use that demonstrates the adaptability and usefulness of synthetic fibers is in geo-textiles. These materials are essential for improving soil stability, erosion prevention, and earth structure strengthening in civil engineering projects. Synthetic fibers are perfect for these applications where longevity is crucial because of their resilience to chemicals and durability [43].

6. Challenges & Limitation:

The environment and the industries involved are impacted by the numerous obstacles and restrictions associated with the development and use of synthetic fibers. Environmental effect, cost, production complexity, and recycling challenges are important considerations. Due to their production and extraction from non-renewable resources like gas and petroleum, synthetic fibers like polyester and nylon contribute to environmental deterioration, especially greenhouse gas emissions that hasten climate change. The production method also uses a lot of water, particularly when dyeing, which causes freshwater to be depleted and untreated effluent to contaminate the area. Since many synthetic fibers are based on petrochemicals, their production costs are correlated with the price of petroleum as well. Because of this connection, the price of synthetic fiber is susceptible to changes in the oil market, which presents manufacturers with financial difficulties. Technological and financial challenges impede the recycling of synthetic fibers. Sorting is made more difficult by the wide range of synthetic materials since

different fiber types require different recycling techniques. Because recycled fibers are typically of a lower standard than virgin fibers, their attractiveness is limited, and the textile industry's transition to a circular economy is slowed down. Technical and financial limitations prevent fiber-to-fiber recycling from being widely adopted at this time; more research and funding are needed to make it reality [44].

7. Conclusion:

By exploring the diverse realm of synthetic fibers, we have brought attention to their vital contributions to the development of composite materials for a range of industrial uses. The importance of synthetic fibers has already been extensively studied, demonstrating their unmatched benefits in their durability, toughness, lightweight, and flexibility in a variety of applications, including medical devices, sports equipment, and aerospace and automotive. The various uses of these fibers as well as their synthesis methods highlight the creative approaches being used to improve their qualities and practicality in modern innovation and daily life.

Future research and development efforts must be focused on minimizing the negative effects of synthetic fibers while optimizing their positive effects, given the persistent concerns around them. Future breakthroughs are made possible by the encouraging trajectory of innovations, especially in the fields of nanotechnology environmentally friendly manufacturing, and smart composite materials. With the promise of ground-breaking capabilities that will further influence the field of engineering and material science, synthetic fibers have enormous potential to contribute to technological advancement and sustainability as we investigate and enhance these materials.

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- Dynamic Triaxial test : ASTM D5311
- Soil Cone Penetration : IS:2720 (Part 5), BS 1377
- California Bearing Ratio (CBR) :IS: 2720 (Part XVI), BS 1377; 1924; EN 13286-47/ ASTM D 1883; AASTHO T 193
- Three gang consolidation : IS: 2720 (Part-XV), IS: 12287, BS: 1377, ASTM D2435
- Triaxial - UU CU CD : IS:2720 (Part 11 & 12), ASTM D 3999, ASTM D 5311
- Light Weight Deflection : ASTM D 2583

- Swell test : IS:2720 (Part 40 & 41)
- Unconfined Compression test : IS:2720 (Part 10), AASHTO T208
- Standard Penetration Test (SPT) : IS:2131, IS:9640
- Dynamic Cone Penetration Test : IS:4968 (Part - 1)
- Static Cone Penetrometer : IS:4968 (Part-3)
- Plate Bearing Test : IS: 1888
- In-Situ Vane shear : IS 4434
- Field CBR Test : IS:2720 (Part 31)
- Vane Shear : IS:2720 (Part 30)
- Soil to soil friction by Direct Shear : IS: 2720 (Part XIII),ASTM D3080; BS 1377
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- Viscosity Std tar viscometer : IS 1206, IP 72, BS-2000, (Part 72)
- Ductility test on asphalt : IS 1208, ASTM D113, AASHTO T 51
- Softening point : IS 1205, BS 4692
- Asphalt Mixture Theoretical Density : ASTM D2041-03
- Physical properties of Aggregates
- Flash point : IS:1209, IS:1448 (Part 21), ASTM D 93, BS 2839, ISO 2719,
- Penetration test on asphalt : IS 1448 (Part 60), IS 1203, ASTM D 5, IP 49, ISO 2137
- Marshall mix design for asphalt mixtures : ASTM D6926-15, 6927-20

- Wheel rut test : EN 12697-33
- Core drilling for asphalt pavements : EN 12504-1
- Dynamic Shear Rheometer :IS 15462:2019
- Asphalt Institute MS-2
- Asphalt pavement field density - Pavetracker (NDT) : ASTM D 7113; AASHTO - T343
- Light Weight Deflection (LWD) : ASTM D 2583
- Rotational Viscometer IS 154621:2019
- Tensile Strength Ratio - AASHTO-T283
- Gyrotory compactor test
- Binder extraction from bituminous : ASTM D 2172, AASHTO T-58, T-164, EN
- Four Point Bending Test - ASTM 8237



Installation Damage test on geosynthetics

BTRA's geosynthetics installation damage test aligns with ISO standards, such as ISO 10722, which defines the procedure for assessing mechanical damage under simulated installation conditions. This standard evaluates how geosynthetic materials, including geotextiles, geogrids, and geomembranes, withstand installation stresses caused by coarse materials and equipment load.

This test helps quantify the durability and effectiveness of geosynthetic materials after installation.

By adhering to ISO 10722, BTRA ensures that the test results are globally relevant and can guide engineers in selecting durable geosynthetics that meet international standards for various civil engineering applications, including roads, embankments, and landfill covers.



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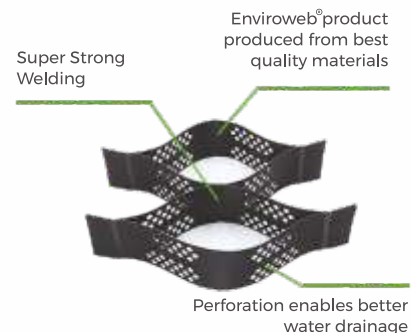
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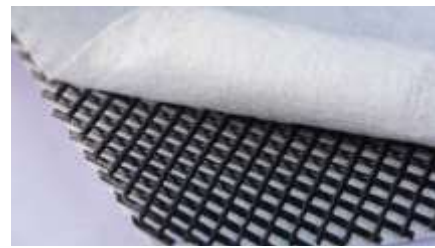
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