

Ultra-high molecular weight polypropylene (UHMWPP): Synthesis and fiber processing

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ABSTRACT

Ultra-high molecular weight polyolefin (UHMWPO) has enormous potential applications due to its excellent mechanical properties such as tensile strength, flexural modulus, toughness and outstanding chemical resistance. But the processing of polyolefin, in particular UHMWPO fibers, cannot be processed by conventional methods due to its very high melt viscosity. In this work, we synthesized isotactic ultra-high molecular weight polypropylene (UHMWPP) resin and studied the processability of UHMWPP fibers using gel spinning and investigated physicochemical properties. UHMWPP gel was made at various concentrations in decalin solvent at 150°C to produce consistent spinning dope solutions. The 7 wt.% concentration of UHMWPP was deemed best for fiber creation, compared to 3 wt.% and 5 wt.%. A rheological time sweep was done to ensure the gel's stability at 170°C before the spinning process. The UHMWPP's gelation and fiber formation were studied by tweaking the gel concentration and adjusting the processing temperature. The resulting UHMWPP monofilament had a measure of 220-250 denier. The hot stretched fibers were analyzed with scanning electron microscopy (SEM) to understand the surface morphology of the fibers. The crystal morphology of UHMWPP fibers was measured with wide-angle X-ray diffractometry (WAXS) and DSC. The X-ray patterns of hot stretched UHMWPP fibers showed crystalline peaks compared to those without stretched fibers. **Polyolefins J (2024) 11: 21-28**

Keywords: Polyolefin; UHMWPP fibers; gel-spinning process; fiber stretching.

INTRODUCTION

The demand for high-strength polymer synthetic fibers is increasing day by day for many engineering applications [1]. Such types of synthetic fibers are made from thermoplastic polymers (like PP, PE, etc.) using the appropriate processing methods (dry spinning, wet spinning, melt spinning) [2-4]. In particular, the UHMW polyolefin fibers are replacing conventional materials useful in the defense sector, aerospace, automotive and composite applications such as ceramics and metals mainly due to their lightweight, high heat and oxidative

resistance, chemical inertness, high dimensional stability, corrosion resistance and ability to maintain mechanical properties over a wide temperature range [5]. In addition, UHMW polymer powder is challenging to process directly into fibers because of its high viscosity, high melting point, and highly entangled molecular structures [6]. However, the first high-performance polymer was commercially developed by DSM (Netherlands) using ultra-high molecular weight polyethylene (UHMWPE) polymer and prepared very high strength UHMWPE

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fibre using a gel-spinning technique [7, 8].

Several research groups have worked on the processes and studied their impact on the structure-property correlation of UHMWPE fibers. The different processing techniques involved include gel-spinning, hydrostatic extrusion, solid-state extrusion, high drawing, hot drawing, swell drawing, etc. [9, 10]. Recently, Yang et al. prepared the blended fibers of ultrahigh molecular weight polyethylene (UHMWPE) and high-density polyethylene (HDPE) using a solution blending and gel spinning process [11]. Moreover, the gel spinning process required solvents such as paraffin, decalin, n-dodecane, p-xylene, 1,2,4-trichlorobenzene, and kerosene to dissolve ultrahigh molecular weight polyolefin [12,13]. Rajput et al. worked on natural oils such as sunflower, palm and orange oil (terpene) as extrusion solvents for UHMWPE fiber preparation [14]. The gel-spinning technology process mainly consists of typically 10 wt.% UHMW polymer and 90 wt.% solvent mixed either with decalin or mineral oils, which are cost-effective. UHMW polymer gels are extruded with a screw-type mixer, and fibers are shaped through a spinneret and subsequently cooled, and drawing process is followed to prepare high strength fibers.

Recent studies by multiple teams have showcased advancements in UHMWPE fiber through various methods. For instance, Prajesh Nayak et al. elevated the performance of electrospun UHMWPE fibers using post-processing treatment, achieving a tensile strength of 11.14 ± 3.65 GPa at a stretching temperature of 130°C [15]. Another important study by Yang et al. reported UHMWPE fibers grafted with polypyrrole (PPy) after undergoing a plasma treatment in a mixed oxygen and nitrogen environment. This resulted in impressive interfacial shear strength of 15.75 MPa, and a 357% enhancement compared to the original UHMWPE fiber [16]. Similarly, Han's team reported the creation of UHMWPE/epoxy composites boasting superior adhesive properties, achieved by forming an interface membrane on the UHMWPE fiber surface [17]. Furthermore, a recent study introduced a ballistic composite material that offers a unique microstructure, setting it apart from traditional UHMWPE UD fabric composites [18]. UHMWPE fibers are known for their strength and lightness. Ongoing research explores into how their microstructure affects ballistic performance, with factors like molecular alignment and crystallinity crucially influencing the fiber's response to impacts [19]. Integrating UHMWPE fibers with materials such as graphene or carbon nanotubes in composites can offer enhanced performance, as highlighted by Marques

and colleagues [20]. In the composite materials development based on high molecular weight fiber, the functionalization of UHMWPE fibers' surface is of utmost importance. This modification guarantees an enhanced bond between the UHMWPE fibers and the matrix of the composite reported by Jiang et al. [21].

The wide acceptance and diverse applications of UHMWPE fibers, contrasted with the developmental nature of UHMWPP fibers, is a consequence of several factors related to material properties, processing challenges, etc. The development and commercialization of UHMWPE fibers (often recognized under brand names like Dyneme or Spectra) began earlier than UHMWPP. This gave industries more time to explore, understand, and implement UHMWPE fibers in various applications. Another bigger challenge associated with UHMWPP fiber is the structure of polypropylene (with its methyl group) introduces complexities in fiber formation. Achieving a highly oriented structure, crucial for high strength, is more challenging due to this structural difference. However, the higher thermal properties such as higher melting temperature may benefit where higher thermal resistance requires. Given this, development of UHMWPP with low cost and comparable properties to UHMWPE is challenge at the current stage.

In this paper, the synthesis of ultra-high molecular weight PP (UHMWPP) performed and UHMWPP fiber processing using gel spinning technique, process parameters, post treatment of fibers and their characterizations have been studied. Due to superior mechanical properties such as low weight, high-strength, modulus, and good impact resistance in addition to their environmental and chemical stability, UHMWPP fibers can find a wide variety of applications that include bulletproof jackets, helmets, high-performance ropes, high-performance fabrics, and reinforcements for composites [22].

EXPERIMENTAL

Materials

Irganox 1010 antioxidant (Sigma-Aldrich), Decalin (LR grade, SD Fine chemicals), and n-hexane (AR grade, SD Fine chemicals) solvents were used without any further purification. The magnesium alkoxide precursor was prepared as per reported process [23]. Ziegler-Natta pre-catalyst was prepared with MgCl_2 supported 3,3,3',3'-tetramethyl- 2,2',3,3'-tetrahydro-1,1'-spirobiindane-5,5',6,6'-tetracarboxylate (STC) as an

internal donor as given in our given research work [24]. The ultra-high molecular weight PP (UHMWPP) was prepared by using propylene polymerization in slurry process. 2 L n-hexane was added in a preheated 4 L SS reactor under nitrogen atmosphere, the pre-catalyst to co-catalyst molar ratio kept 250, whereas co-catalyst to external donor (D-donor) at 50 molar ratio. We performed the polymerization reaction at 30 and 50°C temperatures in the absent of chain terminating agent and set 7 kg/cm² propylene pressure for 2 h.

Method

UHMWPP-1 polymer resin was dissolved in a 2 L stirrer tank batch reactor containing 1.5 L decalin as a solvent and stirred for 1 h at 150°C to obtain homogenous spinning dope solutions with three different concentrations such as 3%, 5%, and 7% (by weight). Irganox 1010, as a primary antioxidant (0.2% (by weight)) was used in all batches. After 1 h heating, the solution was cooled to room temperature and transferred in a beaker for the spinning process. The same gel preparation conditions were maintained for all different concentrations of the samples.

Thermal analysis was performed using a Mettler-TA instrument and Mettler-Toledo-TA89E system software. The particle size distribution (PSD) analysis of UHMWPP polymer resin was done in dry dispersion mode using a Cilas particle size analyzer (Model: 1190). The weight of the specimen was 5 mg in an aluminum pan. The second heating results were considered in the analysis. The xylene soluble content was measured according to ASTM D-5492 method by dissolving polypropylene samples in boiling xylene and the weight percentage of soluble portion was reported as xylene soluble (XS). Polymer resin bulk density was measured according to ASTM D792. The molecular weight and molecular weight distribution of resin were determined by Polymer a Char high temperature gel permeation chromatography (HT-GPC) instrument equipped with PL GEL Olexis 13-micron, 300x7.5 mm columns, an infrared and viscometric detector and operated at 135°C temperature in 1,2,3-trichlorobenzene as a eluting solvent. Calibration was done with polystyrene (molecular weight from 600 to 2.4 million g/gmol). Rheological time sweep analysis of prepared UHMWPP gel has been carried out on a TA rheometer instrument at 170°C temperature for one hour.

Gel Spinning Process

A four-zone screw extruder was used for spinning the UHMWPP suspension. A schematic diagram is shown

in Figure 1; it shows that the dissolved UHMWPP-1 gel passed through the zone with adequate heating. After extrusion, the preheated spinning solution was passed through a conical spinning hole die. The extruded UHMWPP-1 solution was crystallized by cooling, thus resulting in the formation of fibers. The solution was then extruded through 0.5 mm die orifices with an aspect ratio of 3:1. The optimized zone temperature maintained at Z1 : 150°C, Z2 : 155°C, Z3 : 165°C, Z4 : 165°C and Z5 : 170°C.

Solvent extraction

For gel spun UHMWPP fibers, solvent extraction is a key process because it affects the durability of fibers. To get good quality fibers, the trapped decalin solvent needs to be extracted using a nonpolar solvent like n-hexane. This extraction process was carried out by dipping the bobbins in 1 L pot containing the solvent. After solvent extraction, the UHMWPP fibers were sent for the hot stretching process.

Fiber hot stretching process

Hot stretching was performed using heated godet rolls. As shown in Figure 2, the prepared UHMWPP fiber is processed using the hot stretching unit with five hot godets assemblies. The godet speed is adjusted so that the fibers cannot break down during the hot stretching process. The fiber extrusion speed was set to 5 RPM. The UHMWPP fibers extrusion freely extruded into ~20 cm air gap and quenched into water bath and simultaneously passed through stretching roller and quenching water bath. The stretching speed of each roller was set up such that the fiber would not break during spinning. After successful spinning, the fiber was collected on single head winder for further processing and hot stretching. The fiber drawing was done with different draw ratios for each run such as 1:2, 1:5, and

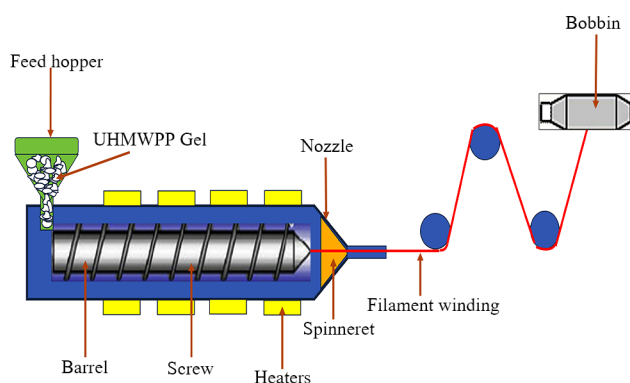


Figure 1. Schematic diagram of UHMWPP fiber spinning process.

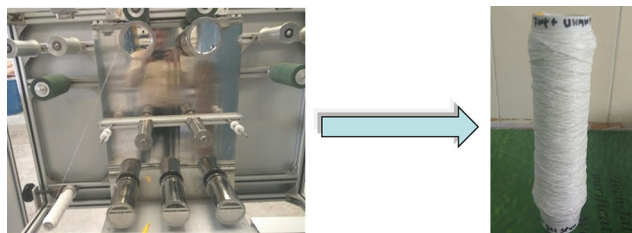


Figure 2. UHMWPP fiber hot drawing and winding process.

1:8 and 1:10. The draw ratio is defined as the ratio of the collection roller speed to the feed roller speed. All the godets were heated to 150 °C temperature to prevent the fibers from melting during the process.

The prepared fiber diameter was measured by an optical microscope (Carl Zeiss) instrument, and fiber morphology analysis was performed through a high resolution scanning electron microscopy (HR-SEM) instrument from NovaNanoSEM 650 (ThermoFisher, USA) using a BSE detector in low vacuum at ambient temperature. X-ray diffraction (XRD) studies have been conducted for prepared UHMWPP fiber samples on Bruker XRD (Model:D8) for our fiber samples by using small-angle and wide-angle X-ray scattering (SAXS and WAXS). Thermal analysis of prepared UHMWPP resin, as such fiber and stretched fiber samples was performed at heating rate of 10°C/min between 30°C to 200°C temperature range. Mechanical analysis of stretched fibers was performed on a Zwick Roell Z005 universal testing machine at ambient temperature. The fibers were attached using hydraulic pressure tester clips and stretched uniaxially with a constant speed of 1 mm/min.

RESULTS AND DISCUSSION

The lab synthesized isotactic ultra-high molecular weight PP (UHMWPP) produced at elevated temperature with 1.0-1.1 million molecular weight and broad molecular weight distribution [25] is given in Table 1. The morphology of the synthesized UHMWPP resin is found to be spherical. The average particle size distribution (D_{mean}) of resin particles was observed ~550

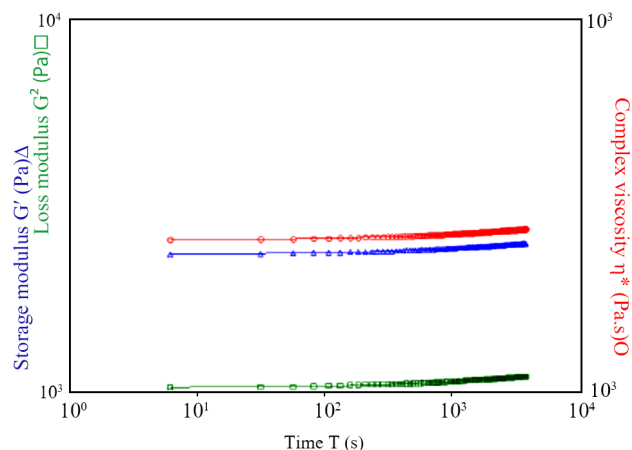


Figure 3. Rheological time sweep graph of prepared UHMWPP gel.

μm with narrow particle size distribution. The thermal characteristics were studied by the differential scanning calorimetry (DSC) analysis and the melting temperature (T_m) was found in 159-162°C range, whereas the onset crystallization temperature (T_c) was resulted between 121-122°C. The isotacticity was determined by the xylene soluble method [24] and observed more than 95% isotactic UHMWPP polymers with 0.3 g/cm³ bulk (tapped) density.

The UHMWPP gel was prepared to understand the stability of these gels, specifically in elevated thermal conditions. The chosen solvent for creating UHMWPP gel samples was decalin, primarily due to its well-known effectiveness in certain polymer-based reactions and formations. The rheological time sweep analysis subjected the prepared gel samples to a rheological time sweep analysis. This analytical technique, tailored to gauge the flow and deformation characteristics of materials, provides an unparalleled insight into the behavior of polymer gels over time. The primary aim was to understand the stability of the UHMWPP gel when exposed to a temperature of 170°C, spanning an observation period of one hour. As illustrated in Figure 3, the gel showed impressive toughness. The graphical representation offers a clear insight that there was minimal to no variation in the properties of the gel, implying that it maintained its structural integrity and did not undergo any unfavorable phase changes or

Table 1. Productivity, xylene-soluble percentage, bulk density, and HT-GPC results of polypropylene products.

Sample name	Temperature (°C)	Productivity (kg PP/g cat)	Xylene soluble (%)	Bulk density (g/cm ³)	Tapped density (g/cm ³)	M_w ($\times 10^5$) ^(a) (g/mol)	MWD ^(a) (M_w/M_n)
UHMWPP-1	30	0.4	4.2	0.27	0.30	11.1	12.8
UHMWPP-2	50	0.6	4.5	0.28	0.31	10.9	11.1

^(a)Molecular weight determined by HT-GPC (polystyrene standard).

decompositions at the said temperature. The UHMWPP gel's ability to remain stable in decalin solvent at 170°C exhibits that it won't break down easily. This means it's suitable for both the fiber spinning and the hot stretching processes.

Comparing the thermal analysis of the UHMWPP resin polymer with the stretched UHMWPP fibers labeled as S1-1:2 and S4-1:10 (Figure 4) and the as-spun UHMWPP fiber, showed that the fibers with a higher draw ratio have an increased crystallization temperature compared to both the UHMWPP resin and the as-spun fibers. The main reason for the increase in crystallization temperature is molecular orientation, during the fiber drawing process, the polymer chains (or molecules) are aligned in the direction of the fiber axis. This alignment or orientation tends to increase the order in the polymer system also during orientation a more efficient and ordered packing of the polymer chains took place, which often results in a higher crystallization temperature. Another reason for increased in the crystallization was chain entanglement. The raw UHMWPP resin consists of polymer chains that are randomly coiled and entangled. These entanglements can act as physical crosslinks, hindering the movement of individual chains, and thus making it harder for them to adopt a regular, crystalline arrangement. As a result, the crystallization temperature might be lower for the resin. When the polymer is drawn into fibers and further hot stretched, some of these entanglements are reduced, allowing a more efficient crystalline packing and, hence, a higher crystallization temperature. Moreover, the drawing and stretching processes can lead to some level of chain scission or a change in the molecular weight distribution. Changes in molecular weight distribution can also affect the crystallization behavior of the polymer.

The structural evaluation and crystallization of the

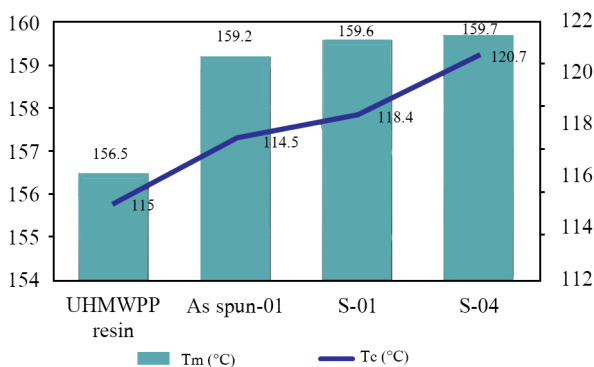


Figure 4. Thermal analysis of UHMWPP resin, as spun fiber and stretched fibers.

hot stretched UHMWPP fibers were evaluated by XRD characterization. Tian et al. examined ultra-high molecular weight polyethylene (UHMWPE) pre-stretched fibers in situ using small angle x-ray scattering (SAXS) and wide angle X-ray diffraction (WAXD) during the ultra-high stretching process [26]. The transition from shish-kebab to fibrillar crystals during ultra-high hot stretching of UHMWPE fibers was studied. The crystal morphology of UHMWPP fibers was measured by using wide angle X-ray scattering method. The XRD analysis of the UHMWPP fibers with different stretching ratios is shown in Figure 5. Cycle S1-S4 corresponds to the stretching ratios of 1:2, 1:5, 1:8 and 1:10 times. As shown in XRD peaks, as the stretching ratio increases, UHMWPP fibers' crystallinity shows more crystalline in nature compared to the first stretching ratio.

UHMWPP in resin form generally exhibits a monoclinic crystalline phase, known as the alpha (α) phase. The XRD pattern would predominantly display peaks corresponding to this phase, reflecting a relatively lower degree of molecular orientation. Spinning UHMWPP fibers from a polymer such as UHMWPP creates a level of molecular alignment due to drawing and extrusion processes. This will reflect in its XRD pattern, where the peaks corresponding to the polymer chain's axis will become more defined, indicating a higher level of orientation compared to its bulk form. Hot-stretched UHMWPP fiber similar to UHMWPE [27], if UHMWPP is hot-stretched, this process would further enhance the orientation of the polymer chains, leading to a more significant degree of crystalline orientation and possibly even crystallinity. In the XRD pattern, the peaks corresponding to the chain axis would become sharper and more intense. Some peaks might diminish, shift, or even split, depending on the changes

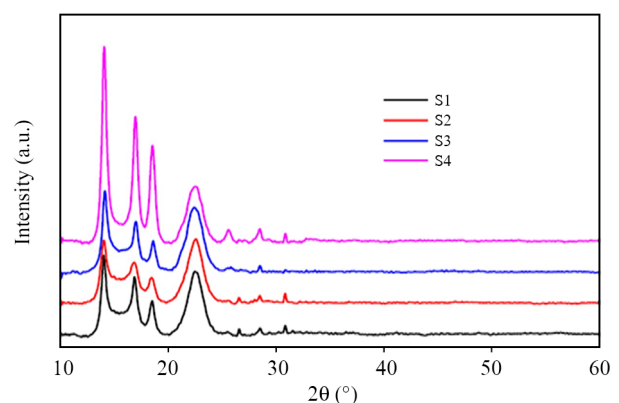


Figure 5. XRD characterization of UHMWPP hot stretch fibers and Shish-kebab structure.

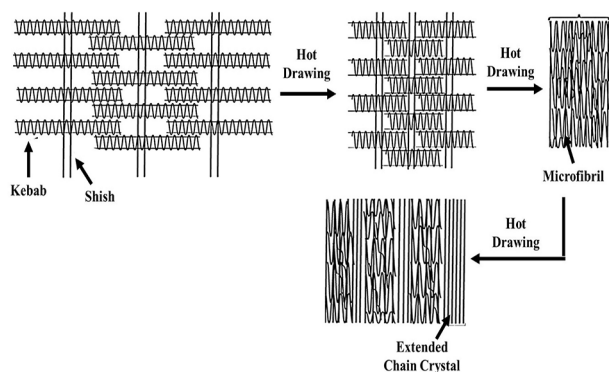


Figure 6. The structural evaluation of shish-kebab to microfibril formation of UHMWPP fibers [29].

in crystalline phases and the orientation of crystallites [28].

The crystalline nature of the fibers and the microfibril formation present in the as-spun fibers with respect to the stretched fiber samples of different drawing ratios can be compared to a shish kebab-like structure as shown in Figure 6 [29]. The tensile properties were performed at ambient conditions. Figure 7 shows the stress-strain curves obtained from the tensile testing of the UHMWPP fiber samples. The results exhibit that the Young's modulus (MPa) increases as the stretching ratio of the fiber increase from 1:2 to 1:10. The Young's modulus (MPa) found 247 MPa at 1:2 stretch ratio whereas at 1:10 its stretched ratio increases to 748 MPa (Table 2, Figure 7). When fibers undergo hot stretching, several changes occur at the molecular level that can increase the Young's modulus. As fibers are hot stretched, the polymer chains tend to align more in the direction of the stretching. This orientation of the polymer chains helps them bear loads more effectively, thereby increasing the fiber's stiffness or modulus. Another reason is that during the hot stretching crystalline regions of the fiber increases. These crystalline regions are stiffer and

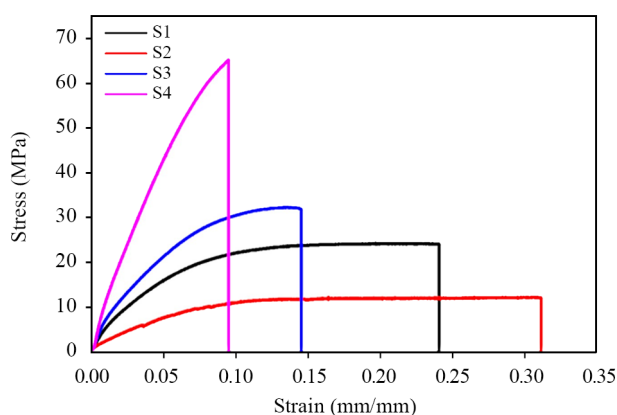


Figure 7. Tensile strength of UHMWPP fibers: S1 corresponds stretching ratio of 1:2, S2-1:5, S3-1:8 and S4-1:10.

Table 2. Properties of UHMWPP fibers.

Sample code	Young's modulus (MPa)	Diameter (μm)	Stretching ratio
S1	247	275	1:2
S2	118	230	1:5
S3	318	320	1:8
S4	784	245	1:10

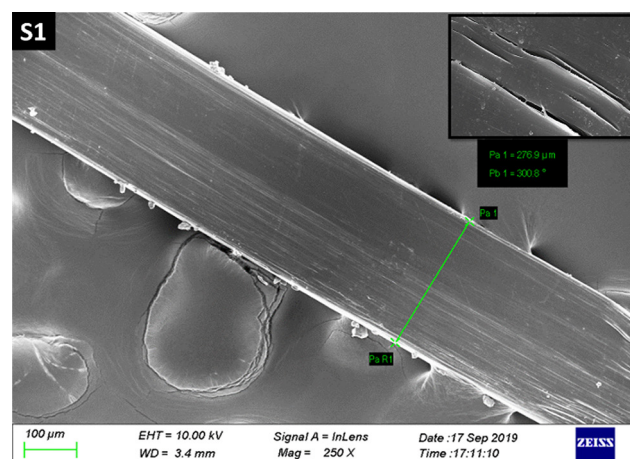


Figure 8. FESEM analysis of UHMWPP fiber (corresponds to stretching ratio of 1:2).

stronger than the amorphous regions of the polymer resulted as higher modulus. During hot stretching, the increased temperature and applied force can help eliminate or reduce chain entanglements, which is also a key factor in increasing fiber strength [26,30].

The fiber morphology/topography and its physical appearance were characterized using FESEM. The representative SEM micrograph of S1 fiber sample corresponds to the stretching ratio 1:2 as shown in Figure 8. The fiber appeared smooth at lower magnifications and rough surface was observed at higher magnifications (inset image shown in Figure 8). For the prepared UHMWPP monofilament, the fiber diameter was observed to be 220-250 denier. However, it was found that the surface morphology of the fibers is homogeneous and cylindrical.

CONCLUSION

Ultra-high molecular weight PP (UHMWPP) was synthesized at lab scale, and the morphology of synthesized resin was found to be spherical in shape. The isotacticity analysis resulted more than 95% isotactic UHMWPP and 0.3 g/cc bulk (Tapped) density. The average particle size distribution (D_{mean}) of resin was $\sim 550 \mu\text{m}$ with a narrow particle size

distribution. Furthermore, gel spinning is well known for processing UHMWPE fiber, but the gel spinning process of ultra-high molecular weight polypropylene (UHMWPP) fibers is difficult due to the presence of methyl pendant group of propylene monomer, which itself increases the entanglement density. We prepared UHMWPP gel with different concentrations in decalin solvent at 150°C to obtain homogenous spinning dope solutions. The concentration of 7 wt.% of UHMWPP is more optimal for fiber formation compared to 3% and 5% wt. The rheological time sweep analysis was performed to confirm stability of gel at 170°C before gel spinning process. The gelation and fiber formation of UHMWPP has been investigated by optimizing the gel concentration and varying processing temperature. Fiber hot stretching also carried out to improve mechanical strength. A 220-250 denier property was observed for UHMWPP monofilament observed. FESEM analysis of the prepared fiber showed a homogeneous cylindrical surface morphology. Crystal morphology of UHMWPP fibers was measured with wide angle X-ray scattering (WAXS) technique. UHMWPP fibers showed high crystalline nature compared to the first stretching ratio. This highly crystalline nature of the fibers and the microfibril formation as in the as-spun fibers as the drawing ratio increases can be compared to a shish kebab-like structure.

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CONFLICTS OF INTEREST

The authors declare that they have no conflicts of interest.

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