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Short Communications

Effect of n-hexane on the physical and mechanical characteristics of the liquid paraffin-based UHMWPE gel spun fibre

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The current research examined the impact of n-hexane on the liquid paraffin (LP) extraction and the resulting change in the physico-mechanical properties of LP-containing UHMWPE gel-spun fibres of different fineness. In response to the DSC data, each fibre sample showed increased crystallinity and melting point after LP removal, whereas it decreased relatively with fineness. SEM images revealed that the fibre's cross-section showed a distorted polygon shape, with significant areal changes despite minimal changes in the motif after LP removal. It was also observed that the fibre contracted more in the cross direction than in the longitudinal direction. Regarding shrinkage, the fibre mass loss was also observed and directly correlated with the denier and oil content, which caused improvement in fineness. Improvements in fineness and breaking strength resulted in increased average tenacity by up to 50% and breaking elongation by up to twice the original value. DMA analysis revealed that, at higher temperatures, fibre samples with LP exhibited greater extension compared to LP-extracted samples. The current study found that stretching of the gel fibre with intermediate LP removal may be beneficial for achieving high draw ratios.

Keywords: Creep, DSC, Liquid paraffin, N-hexane, SEM, UHMWPE

Ultra-high-molecular-weight polyethylene (UHMWPE) has currently gained popularity because of its excellent physico-chemical and tribological characteristics[1]. Moreover, it is suitable for numerous industrial uses due to its low density, low solubility, and exceptional mechanical characteristics, including high Young's modulus and tensile strength, particularly in drawn films[2].

Since UHMWPE polymer comprises significantly high molecular weight chains, it is not as easy to process in the melt phase as conventional polymers like LDPE and HDPE[3]. Thus, it must be converted into gel form by preparing a uniform solution using a suitable solvent. Researchers have explored the dissolution of UHMWPE using a wide range of solvents, including liquid paraffin, decalin, dodecane, p/o-xylene, 1,2,4-trichlorobenzene, mineral oil, naphthenic oil, tetralin, dioctyl phthalate, butyl benzyl phthalate, dibutyl sebacate, trichlorobenzene, 1,2-dichloroethane, stearic acid, cyclopentane, hexadecane, diphenyl ether, and kerosene, etc[3-5]. However, decalin and paraffin oil are the most commonly accepted solvents for preparing UHMWPE gel solutions[6].

In the present research, the liquid paraffin was used to prepare the UHMWPE polymer gel solution. As liquid paraffin is a non-volatile solvent, it must be extracted using a secondary volatile solvent once the UHMWPE gel spinning process is completed[7]. The solvent is usually extracted using n-hexane, which is also used in the food industry for extracting various vegetable oils[8]. Several researcher shave investigated the extraction dynamics of the solvent from UHMWPE gel fibre, wherein n-hexane is predominantly used[9-13]. In all previous studies, the solvent was extracted just before the stretching of the gel fibre, and interestingly, the characteristics of fibre stretching before solvent extraction have not been studied. Moreover, the drawing behavior of UHMWPE polymer with solvent is an interesting parameter to study since the solvent increases the plasticizing properties of the polymer. In this context, the effect of n-hexane on changes in the physical and mechanical behavior of the solvent containing UHMWPE fibre after removal of the solvent has not been studied yet.

Therefore, based on the aforementioned research gaps, the present research used n-hexane to investigate the changes in the physical and mechanical behaviour of the UHMWPE fibre, followed by solvent removal. In this research,liquid paraffin was used as a primary solvent to convert the UHMWPE powder into a gel of 10% polymer concentration and subsequently into gel-spun fibre, and the widely used secondary s olvent, n-hexane,was chosen for the primary solvent removal[8-9]. Initially, the gel fibres of variable

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thickness were stretched by a factor of 25-33 times before the remaining liquid paraffin was extracted. In this research, 5 different gel-spun fibresof distinct denier were prepared. Thereafter, changes in physical and mechanical properties were studied using DSC (differential scanning calorimetry), SEM (scanning electron microscope), a UTM (universal tensile testing machine), and DMA (Dynamic mechanical analysis). The characteristics of the fibres were investigated, including crystallinity, melting point,and morphological structure, along with longitudinal and cross-sectional shrinkage, andchanges in mechanical properties such as denier, tenacity, elongation, shrinkage, and creep behavior.

Experimental

Materials

The UHMWPE powder of molecular weight 3.5 million grams/mole was purchased from Mitsui Chemicals Inc. Pvt. Ltd. The primary solvent, paraffin oil witha density of 0.86 g/cc, was purchased from the local market. As an antioxidant, pentaerythritol, tetrakis (3,5-di-tert-butyl-4-hydroxy-hydrocinnamate) from Sigma-Aldrich Chemicals Pvt. Ltd. was used. The n-hexane of 99% concentration was procured from Loba Chemie Pvt. Ltd.

For processing the UHMWPE gel solution, a twin-screw extruder, model SO 35737 - Omega 25 from STEER Engineering Pvt. Ltd., was used.

Preparation of UHMWPE Fibre Sample

The conversion of the UHMWPE powder into gel solution and extrusion of the gel fibre were performed simultaneously by a twin-screw extruder from Steer World, Bangalore, India. In the present study,a higher polymer concentration was used, wherein the polymer-to-solvent ratio of 10:90 was chosen, and 0.1% antioxidant. In this case, the UHMWPE gel solution was prepared with liquid paraffin from which the monofilament was extruded using a 1 mm diameter single circular nozzle. Following extrusion, the gel fibre was coagulated using normal water at room temperature and subsequently drawn at a temperature of 110-130°C by a factor of approximately 25 to 30 times, i.e., a draw ratio of 25-33. By keeping the aforesaid draw ratio, the five distinct fibre samples with different fineness values (in terms of denier) designated as A, B, C, D, and E (before n-hexane treatment) were produced, as listed in Table 1. For the reproducibility of the experimental results, three replications was taken for each fineness of UHMWPE fibre.

Characterization of UHMWPE Fibre

The fibre samples were analysed for mechanical and physical properties such as DSC, morphology, shrinkage, fineness, tensile, and creep. The detailed test methods for the aforesaid properties are described below.

DSC Analysis of Fibre

The crystal and melting behaviours of UHMWPE samples were investigated by differential scanning calorimetry (DSC) using a DSC-6000 differential scanning calorimeter made by PerkinElmer. UHMWPE samples were cut into small pieces, and each sample weighed about 5 mg; they were placed in a standard aluminium pan and tested under a nitrogen atmosphere, with a supply of 20.0 ml/min. Based on the various investigations, the present samples were first heated from room temperature to 210°C at a heating rate of 10°C/min and then cooled to room temperature at a cooling rate of 10°C/min[1,10,14]. The degree of crystallinity (X_c) of fibres was calculated using Equation (1), where ΔH_f is the enthalpy of the fibre sample, and ΔH is the enthalpy of 100% crystallized polyethylene, which is 293 J/g[15].

$$\text{Degree of Crystallinity, } X_c \text{ in \%} = \frac{\Delta H_f}{\Delta H} \times 100 \quad \ldots (1)$$

Morphological Analysis of Fibre

The scanning electron microscope JEOL-JSM-IT200 was used to analyse the longitudinal and cross-sectional shape of the present fibres. In this instance, fibres were zoomed up to 200x for cross-sectional and 500x for longitudinal views. Furthermore, the change in morphological shape was also studied after the treatment of the sample with n-hexane under 5 min of sonication at room temperature. In this case, the time of treatment was found after investigating the solvent removal process of the fibre with different times

Table 1 — Details of the UHMWPE fibre Samples

S. code	Draw ratio	Fineness (denier)
A	25	157.8
B	26.3	144.6
C	27.2	125.4
D	27.7	121.2
E	32.9	69.0

under sonication. It was found that 5 min under sonication is the minimum or average time to extract up to 100% solvent from the sample.

Shrinkage Analysis of the Fibre

The dimensional changes or shrinkage behaviour of the present 5 distinctive fibres on treatment with n-hexane were investigated.The shrinkage behaviour of the UHMWPE fibres with solvent (liquid paraffin) was analysed on treatment with n-hexane under sonication for 5 min. The lengthwise shrinkage of the sample was visualized by the naked eye, and the crosswise was determined through the scanning electron microscope diameter measurement. The shrinkage percentage of the UHMWPE fibres, both lengthwise and crosswise, was calculated by using Equations 2 and 3.

$$\text{Lengthwise shrinkage (\%)} = \frac{\text{initial length of fibre} - \text{final length of fibre}}{\text{initial length of fibre}} \times 100 \quad \ldots (2)$$

$$\text{Cross wise shrinkage (\%)} = \frac{\text{initial crosswise area of fibre} - \text{final crosswise area of fibre}}{\text{initial crosswise area of fibre}} \times 100 \quad \ldots (3)$$

Fineness Analysis of Fibre

The fineness or denier of the fibre is measured by the manual method, i.e., measuring 1 meter in length and the corresponding mass of the fibre, which was then projected for mass in grams for a 9000-meter length. The formula is given below.

If 1-meter length of the UHMWPE fibre ≈ m gram … (4)

Then, for the denier conversion will be,

9000-meter length of the UHMWPE fibre ≈ m × 9000 gram ≈ x denier … (5)

Tensile Analysis of Fibre

ASTM D2256 was chosen to perform the tensile analysis, which includes the measurement of tenacity and elongation of UHMWPE fibre, with a rate of extension maintained at 300 m/min.

Creep Analysis of Fibre

In this experiment, the creep behaviour of the fibres was analysed using DMA near the UHMWPE polymer's softening temperature, i.e., 110°C. Each fibre was exposed to a constant static load of 0.5 N with a preload of 0.1 N over 20 min (1200 sec). The temperature was kept at 110°C throughout the experiment.

Results and Discussion

The present UHMWPE fibre of 5 distinct fineness coded as A, B, C, D, and E (Table 1) was treated with secondary solvent n-hexane for the removal of the primary solvent, LP. Since the untreated gel-spun fibre samples comprise LP up to 55%, the impact of n-hexane on different physical and mechanical characteristics was significant, which are described below.

DSC Analysis of Fibre

Figure 1 shows the DSC spectra for the present UHMWPE samples during heating and subsequent cooling, concerning both before (untreated) and after (treated) solvent extraction. The melting and crystallization parameters are listed in Table 2. Since the solvent, i.e., LP, is an amorphous-dominated part, therefore, it induces an overall reduction of average crystallinity of the samples even after successful hot drawing. On the other hand, after the removal of the LP by n-hexane, the fibre exhibited a notable increase in crystallinity, as shown in Fig. 1 and Table 2. Figure 1

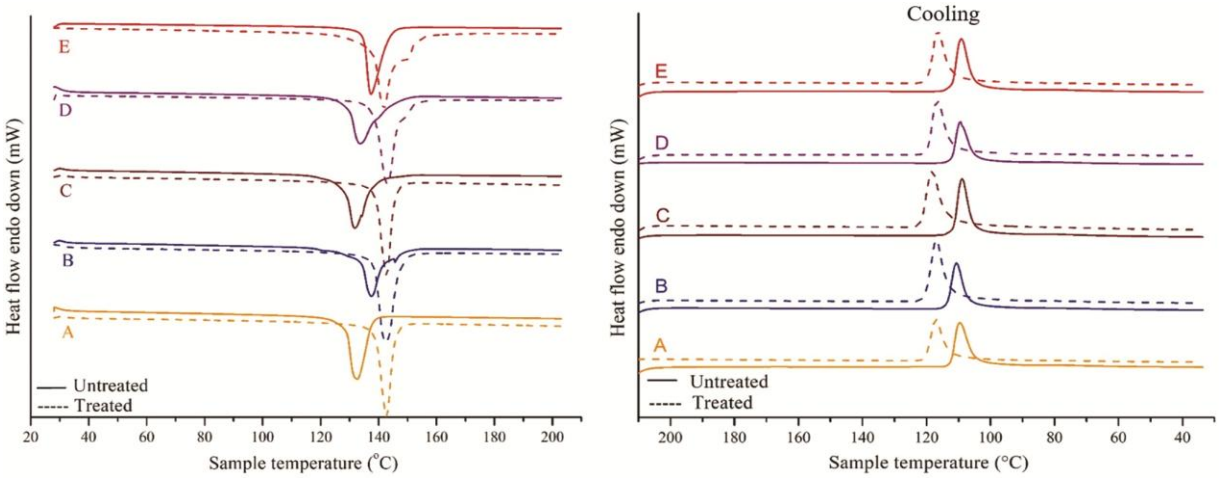

Fig. 1 — DSC heating and cooling curve of various UHMWPE samples

Table 2 — Extracted value of crystallinity and melting point

S. Code	Crystallinity (%)						Melting point (°C)		
	Heating			Cooling					
	Untreated	Treated	Change, %	Untreated	Treated	Change, %	Untreated	Treated	Change, %
A	41	74.2	81.0	26.4	46.5	76.1	128.3	137.7	7.3
B	41.9	74.9	78.8	28.6	50.7	77.3	129.3	138	6.7
C	43	76.3	77.4	29.7	51.1	72.1	130.7	139.2	6.5
D	46	77.4	68.3	30.1	51.7	71.8	133.7	139.9	4.6
E	47.8	80.1	67.6	31.2	51.9	66.3	135	140.8	4.3

clarifies that the crystal peak significantly increases after solvent extraction for both heating and cooling DSC curves, which can also be referred to from earlier research[16]. This is because the solvent extraction process removes the solvents that are within the oriented chain of the UHMWPE fibre, retaining the gaps between them. In this process, less ordered amorphous domains were reduced, and the increased proximity between chains allowed for more efficient packing of the chains, resulting in a higher proportion of crystalline domains. Additionally, the residual stresses from the molecular chain are allowed to relax, which permits better crystal formation and growth.

The peak of each respective sample decreases after heating, i.e., in the case of cooling, DSC curves showed a lower peak than the heating peak. This is owing to the rearrangement or relaxation of drawn molecular chains, as well as more molecular diffusion from the crystalline to the amorphous phase, resulting in a decrease in the alignment of chains along the fibre axis and forming a lower number of crystals[10]. Moreover, the draw ratio also has a significant impact on crystallinity, and in the present case, with the increase in draw ratio, the crystallinity and melting point increase marginally. As the crystalline region increases, the compactness within the fibre structure increases, resulting in a higher melting point, which happens with the different samples. Moreover, considering solvent removal of the specimens, the change in the degree of crystallinity and melting point between the heating and coolingcurves is noticeably influenced by the draw ratio. In this context, the change in the degree of crystallinity and melting point decreases with the draw ratio. The aforesaid phenomenon can be explained by the fact that a higher denier consists of more solvent and more spaces between the intermolecular chains. This means a greater change in intermolecular packing, corresponding to a greater change in crystallinity and melting point than lower fineness samples. Sample E, showed higher crystallinity and melting point as it is comparatively highly drawn. Similarly, low crystallinity and melting point were observed in the case of sample A because of the relatively low draw ratio among all specimens.

Morphological Analysis of Fibre

SEM analysed images, considering longitudinal and cross-sectional views, are shown in Fig. 2 and Fig. 3, respectively. It was seen that the treated samples longitudinally, seem like rod-like structures and are free from oil stains. The traces of oil (solvent) were seen in untreated samples, which were noticed from a cross-sectional view. The cross-sectional shape of the UHMWPE fibre looks like a randomly distorted polygon shape, and its number of sides depends on the number of hot drawings.It is because the solvent in the UHMWPE fibre makes it softer with resilient, and each time it is stretched, it is compressed by contact with hot rollers. The treated samples are free from solvents and are more compact and crazing than the untreated samples; thus, visible differences can be easily noted.The shrinkage, both width-wise and cross-wise, can be seen from the longitudinal and cross-sectional views in Fig. 2 and Fig. 3, respectively. Details of shrinkage are described in the following section.

Shrinkage Analysis of Fibre

Shrinkage is common in thermoplastic materials, particularly when they come into contact with dry heating and boiling water or liquids[17]. However, in the present case, the shrinkage is due to the removal of solvents from the UHMWPE fibre samples, and the graph is plotted as shown in Fig. 4. It is observed that there is significantly higher shrinkage in the cross direction and lower in the longitudinal direction. This significant variation in shrinkage can be attributed to the orientation of the molecular chain, which follows the length of the fibre and is more resistant to shrinkage than the cross-direction. In Fig. 4, it is also noticeable that when the denier increases, the shrinkage percentage increases since the solvent content is higher for thicker fibres, and vice versa, as

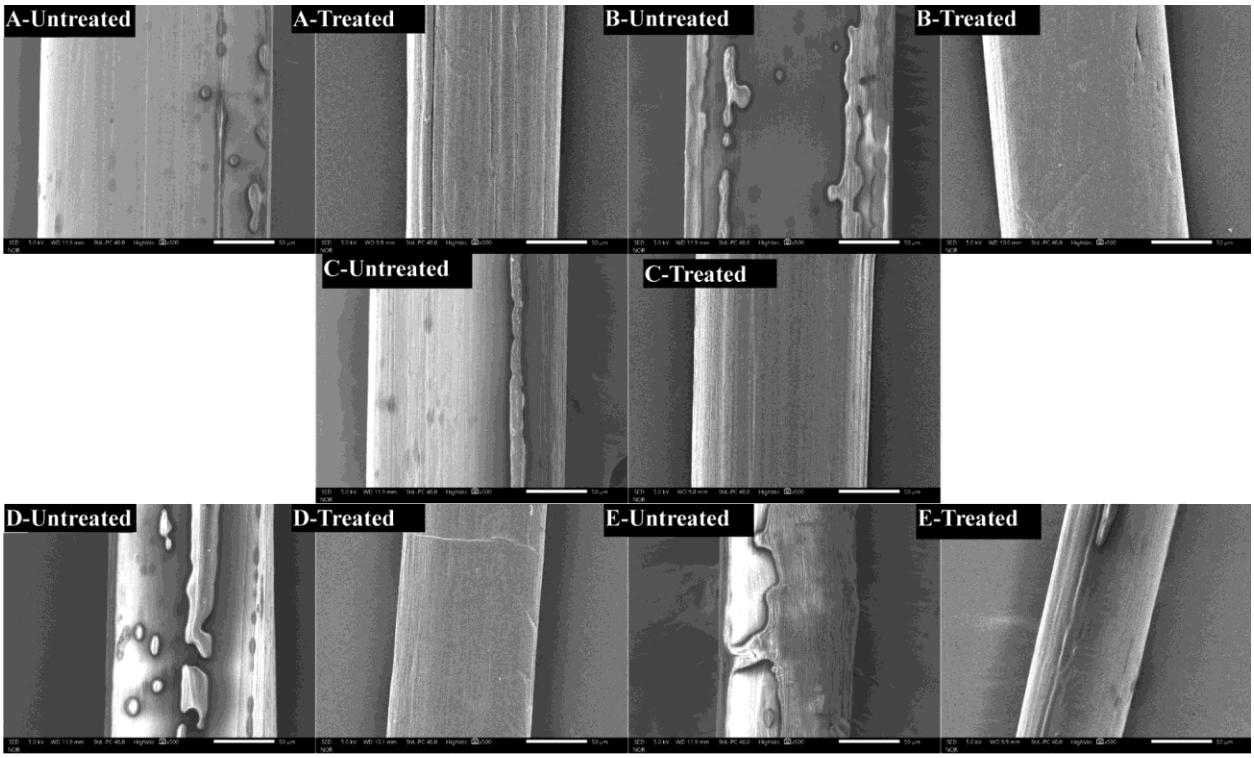

Fig. 2 — Comparative longitudinal morphological changes of UHMWPE fibres before and after n-hexane treatment (A-E): untreated vs treated pairs revealing structural changes following solvent extraction

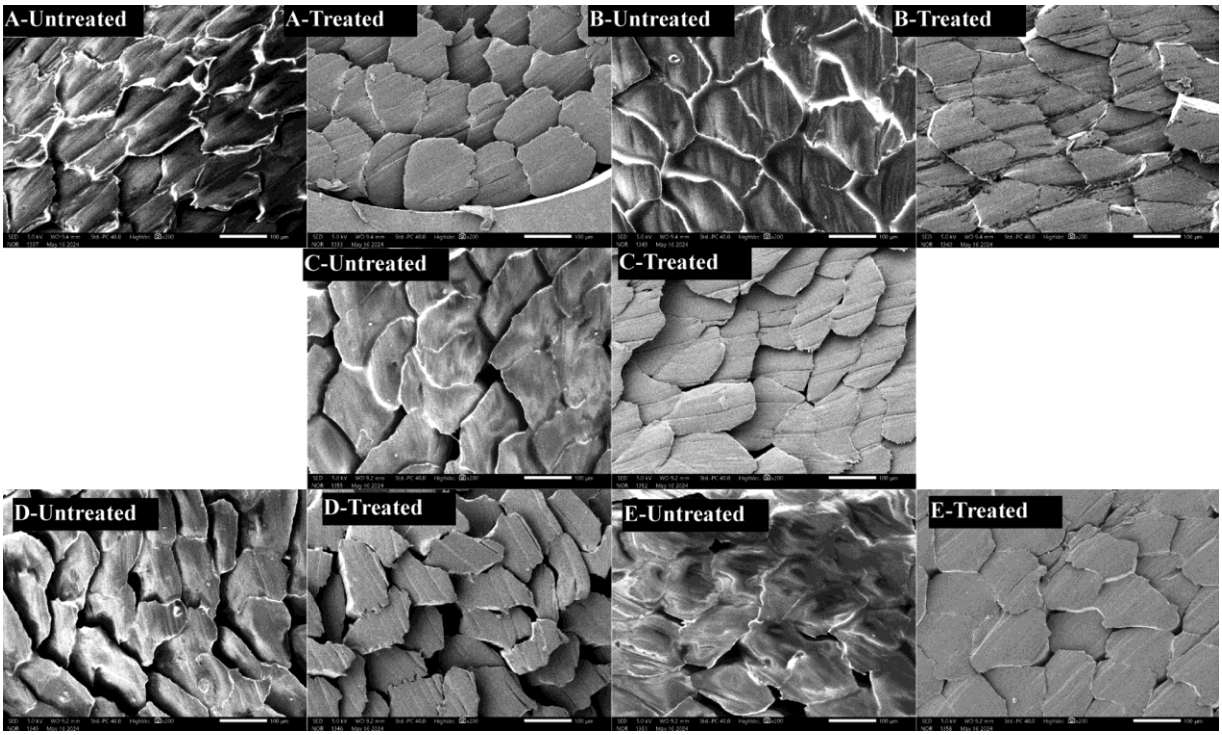

Fig. 3 — Comparative cross-sectional morphological changes of UHMWPE fibres before and after n-hexane treatment (A-E): untreated vs treated pairs revealing structural changes following solvent extraction

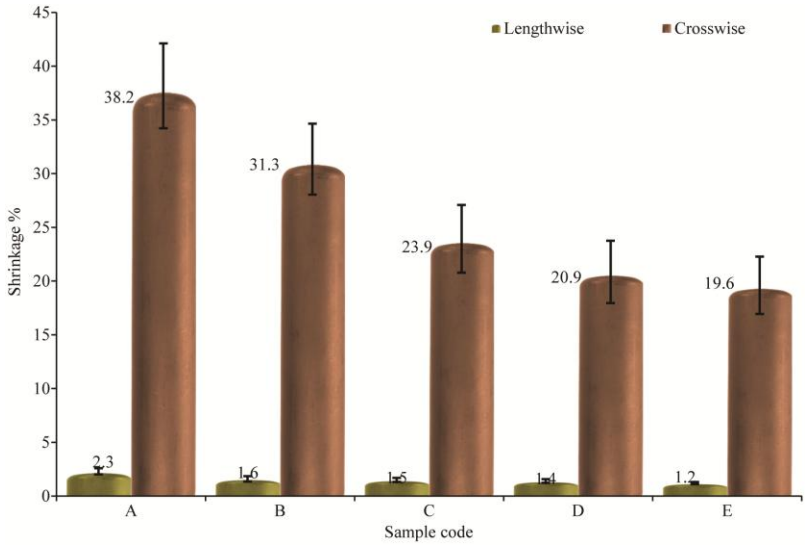

Fig. 4 — Effect of n-hexane on the shrinkage of the UHMWPE fibre
(*Each error bar shows the standard deviation of the mean)

Table 3 — Change in mass and corresponding denier of the fibre with n-hexane treatment

S. Code	Mass, g		Mass loss, %	Fineness (denier)		Change in Fineness, %
				Untreated	Treated	
	Untreated	Treated				
A	0.01753	0.00793	54.8	157.8	71.4	-54.8
B	0.01607	0.00811	49.5	144.6	73.0	-49.5
C	0.01393	0.00760	45.5	125.4	68.4	-45.5
D	0.01347	0.00737	45.3	121.2	66.3	-45.3
E	0.00767	0.00468	39.0	69	42.1	-39.0

* The mass of each fibre is measured in grams per meter.

described before. The lower denier or thickness fibre contains less solvent and is comparably slightly more compact, causing less n-hexane penetration than the higher denier fibre. This causes higher shrinkage in Sample A than in Sample E for both crosswise and lengthwise. The shrinkage behaviour of the UHMWPE fibre may lead to the conclusion that heat-setting is necessary to reduce the level of shrinkage after the removal of the primary solvent.

Fineness Analysis of Fibre

The fineness, or denier, of the fibre is an important property that can directly influence its strength, orientation, and end application. Since the constant length,the denier is directly influenced by the mass of the fibre,and thus the denier was changed with the mass change, as given in Table 3. Furthermore, in the present case, the mass is reduced for a minor change in length, resulting in a significant reduction in the denier values.

Furthermore, for descriptive and comparative analysis between n-hexane-treated and untreated fibre samples, the curve is plotted between different denier UHMWPE fibres and the change in denier, as depicted in Fig. 5. Table 3 and Fig. 5 show that mass loss and corresponding denier loss are directly proportional to the thickness or denier of the UHMWPE fibre, followed by solvent removal.The present analysis indicates a maximum loss of 54.8% and a minimum of 39.0%. In this context, the higher the thickness of the untreated sample,the higher the solvent content and the greater the penetration of n-hexane compared to the lower thickness. The penetration of n-hexane decreases with the number of drawing or stretching operations, following the increasing orientation of the molecular chain and the compactness or crystallinity (Table 2) of the fibre surface. The aforementioned causes more loss in mass for a higher-thickness fibre than for a lower one. Figure 5 also clarifies that the change in denier after n-hexane treatment is minimal for sample E, as it has the lowest fineness among all samples and contain slow solvent or LP content.

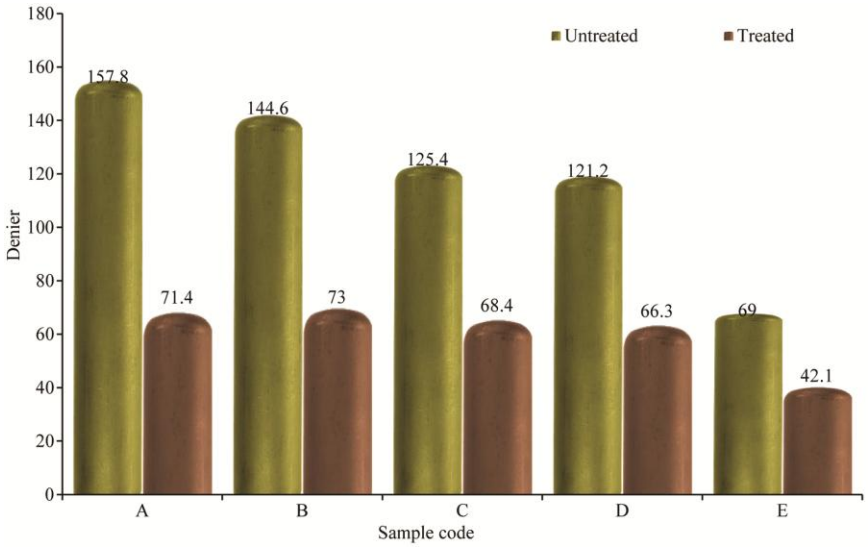

Fig. 5 — Effect of n-hexane on the denier of the UHMWPE fibre

Tensile Analysis of Fibre

Tenacity along with breaking elongation, is the primary property of all textile fibres, and in the case of UHMWPE fibre, it is the most essential part as it is widely used in ballistic-related applications where the strength plays a vital role[18]. Considering the secondary solvent, n-hexane, the tenacity of treated and untreated UHMWPE fibres was analysed, and the graph is shown in Fig. 6.

Figure 6 shows that there is a statistically significant difference in the tenacity of each untreated (with solvent) and treated (without solvent) sample. The change in mass and corresponding denier (Fig. 5) following solvent removal causes a change in tenacity, i.e., grams per denier. However, there is a low marginal sequential difference between each sample, from sample A to sample E. The lowest tenacity values were produced by sample A, due to its higher denier, lower orientation, and crystallinity. Similarly, sample E, characterized by lower denier and higher orientation and crystallinity, resulted in the highest tenacity values. Since Sample A is a less oriented structure and has more space to align molecular chains with each other, it causes more shrinkage (Fig. 4) and the highest change in tenacity, followed by solvent (LP) removal. The change in tenacity decreases with a decrease in linear density (denier). However, although Sample E has the lowest linear density, it shows the highest tenacity value and the significant change in tenacity due to the greatest change in crystallinity (Table 2). The maximum tenacity with the lower denier occurs as the draw ratio marginally increases (25 to 33) from higher to lower

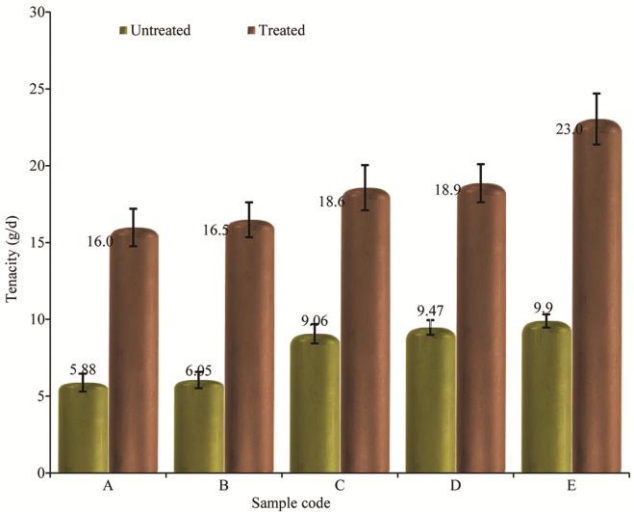

Fig. 6 — Effect of n-hexane on the tenacity of the UHMWPE fibre (*Each error bar shows the standard deviation of the mean)

denier, and expected comparative increase in crystallinity and orientation too. Samples C and D showed similar trends in tenacity, as they both have similar or marginally different deniers.

The elongational behaviour of the fibre samples (Fig. 7) exhibits asimilar statistically significant pattern to the change in tenacity between each untreated and treated sample. The breaking elongation (BE) percentage increases with an increase in linear density due to the higher number of molecular chains in the high-linear-density structure. In this context, there is a higher chance that the remaining unoriented molecular chains will straighten due to cohesiveness during the application of force. The change in breaking elongation between each untreated and

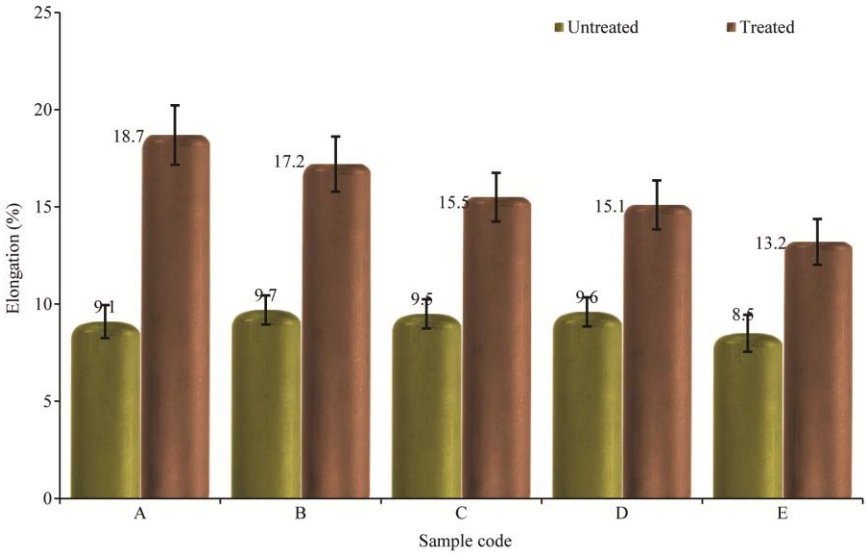

Fig. 7 — Effect of n-hexane on the breaking elongation of the UHMWPE fibre
(*Each error bar shows the standard deviation of the mean)

treated sample increased with linear density due to contraction in width (Fig. 4) and the corresponding increase in the contact points among the molecular chains after solvent removal. This phenomenon occurred more frequently in higher linear density samples than in lower density samples. As with tenacity, samples C and D indicated similar changes in BE, wherein the reasons are stated previously.

Creep Analysis of Fibre

UHMWPE is a highly thermosensitive polymer, which means that its physio-mechanical properties vary with temperature and loading conditions, especially around or above its softening point[19-20]. Thus, creep characteristics are an important consideration that is investigated for the present different fibre specimens to determine the timing of hot stretching, i.e., before or after the removal of the solvent. The timing of hot stretching of gel fibre affects the final denier and tensile strength, which was investigated in the present research. In general, the solvent (LP) is added to the UHMWPE polymer to increase the plasticizing activity and to make a spinnable gel form, which is very difficult to obtain without a plasticizer or solvent. In the gel form of the polymer, the mobility of the molecule is maximum, allowing for the greatest stretch. The details of stretchability before and after solvent removal are described below.

The creep behaviour of the present fibre samples is analysed based on the change in length as strain % with time for 20 min or 1200 sec at a temperature of 110°C, as shown in Fig. 8. In this work, the deformation was kept adequate so that the constant load experiments may be interpreted as constant actual stress tests. From Fig. 8, it can be seen that in all cases other than tertiary creep, primary and secondary creep are induced by the specimens due to the time and temperature limitations. In addition, the curves have all grown exponentially, with the exception of the treated samples, which appear to exhibit linear growth, particularly from the midsection to the end of the curve.

On the other hand, the samples containing solvent (untreated) were stretched more than the samples without solvent. In this context, it has been well reported by earlier researchers that during the removal process of the solvent, molecular chainsconvert to large folded lamellae[12]. Since the untreated samples shrink up to 2.3% lengthwise (from Fig. 4) in which molecular chain folded longitudinally and that folded structure creates more extension at initial phases in all cases (Fig. 8). The unfolded time or expansion time of the treated samples is quite faster than the actual extension of the untreated samples which causes higher strain% in the initial phases of all samples. Additionally, when the folded structure of the molecule is completely unfolded, the strain rate of treated samples decreases, unlike untreated samples. It can also be seen that the strain rate increases with the decrease in denier, and this is because the heat transfer is greater for the smaller area of the low denier fibre and vice versa. For the aforesaid reasons, sample E being the lowest denier, extends up to 146% while sample A only achieves up to 6.5%.

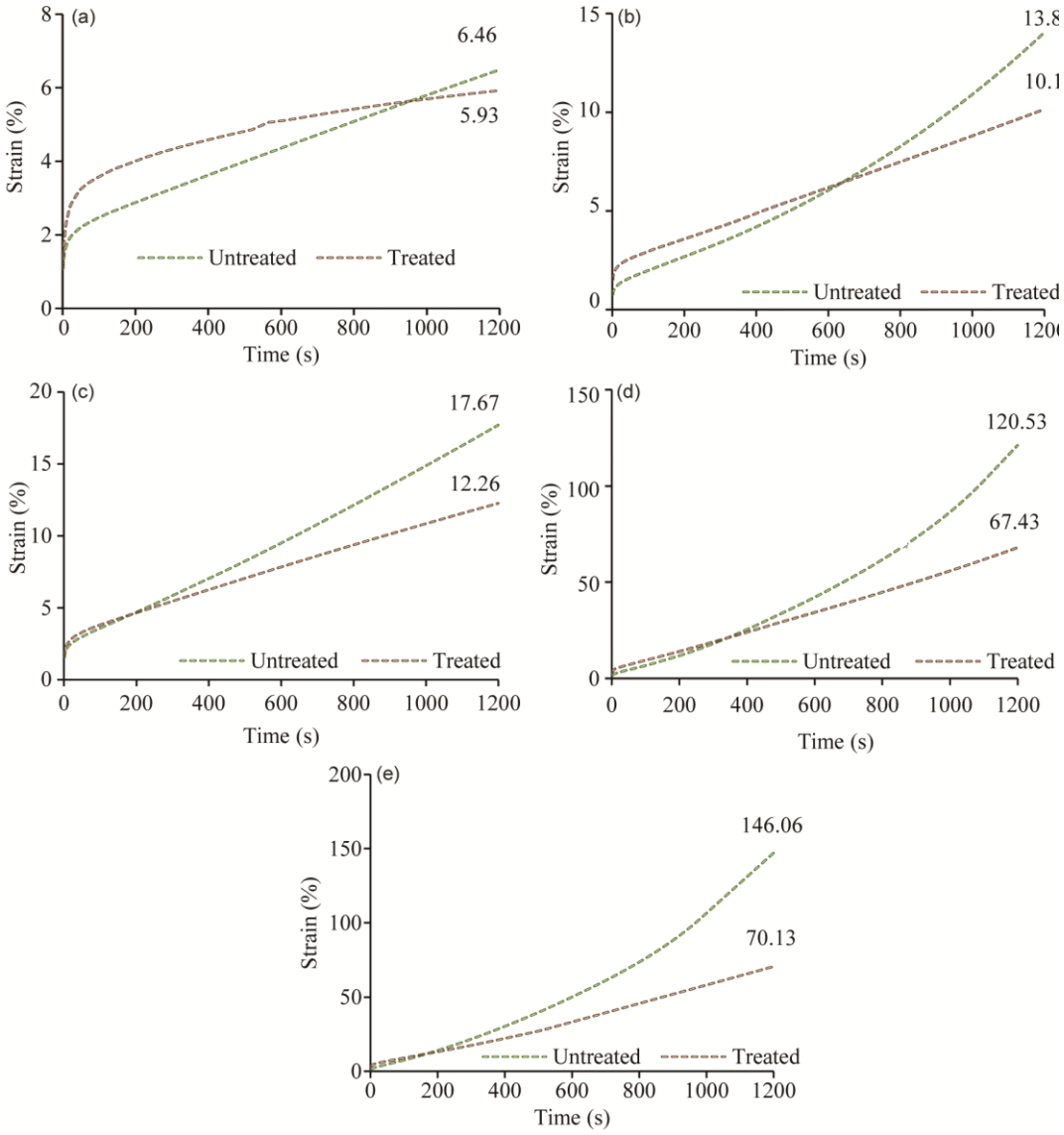

Fig. 8 — Creep behaviour of the present untreated and treated samples based on % of strain with time for a constant temperature of 110°C

From the above discussions, it is observed that the fibre can be stretched more in the presenceof solvents than without; however, the elongation curve (Fig. 7) indicates that the treated samples exhibited greater extension in a specific condition due to enhanced intermolecular connections. In another case, the more entangled points of the polymer chains may limit the maximum extension of the fibre. Therefore, partial solvent removal may lead to a greater extension or draw ratio than complete solvent removal.

Conclusion

The role of a secondary solvent is essential, specifically when the primary solvent is non-volatile. In the present study, the liquid paraffin was successfully removed using n-hexane, and the effect on UHMWPE fibres of different deniers was successfully evaluated. The DSC spectra revealed that the degree of crystallinity and melting point increased after solvent removal, and both parameters decreased with the denier. Morphological analysis revealed that after n-hexane treatment, the fibres exhibited increased compactness and a distorted polygonal cross-sectional shape, which was marginally altered. As the fibre shrank after solvent removal, the fibre mass and corresponding denier decreased. Itwas found that denier loss increased with the initial thickness of the fibre. The tenacity and elongation of the fibre increased upon the removal of solvent, which

was inversely proportional to the initial denier of the fibre. In conclusion, the creep analysis of the current study at high temperatures revealed that UHMWPE fibre can be stretched at high temperatures before solvent removal. However, intermediate or partial solvent removal may be a more effective option to enhance stretchability, especially when fine denier is the predominant requirement.

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