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(54) **Title:** A COMPACT POLYMERIC GEL AND A PROCESS FOR PREPARAING THE SAME

(57) **Abstract:** The present disclosure provides a compact polymeric gel comprising disentangled ultra-high molecular weight polyethylene (DUHMWPE), turpentine, at least one nucleator, at least one filler, at least one antioxidant, and optionally, at least one lubricant. The present disclosure further provides a process for the preparation of compact polymeric gel. The compact polymeric gel of the present disclosure is used to prepare disentangled ultra-high molecular weight polyethylene (DUHMWPE) filaments. Further, a process for the production of DUHMWPE filaments is provided. The DUHMWPE filaments thus obtained exhibit high tensile strength, and modulus which make them suitable for use in defense sector, and medical sector.



A COMPACT POLYMERIC GEL AND A PROCESS FOR PREPARING THE SAME

FIELD

The present disclosure relates to a compact polymeric gel and its use in preparing DUHMWPE filaments.

DEFINITIONS

As used in the present disclosure, the following terms are generally intended to have the meaning as set forth below, except to the extent that the context in which they are used indicate otherwise.

Ultra-High Molecular Weight Polyethylene (UHMWPE): The term ultra-high molecular weight polyethylene (UHMWPE) used hereinafter in the specification refers to a polymer of ethylene having long chains and viscosity average molecular weight of 2 million Dalton and above.

Dis-Entangled Ultra-High Molecular Weight Polyethylene (DUHMWPE): The term dis-entangled ultra-high molecular weight polyethylene (DUHMWPE) used hereinafter in the specification refers to a polymer of ethylene having viscosity average molecular weight of 2 million Dalton and above, wherein the polyethylene chains have substantially low entanglement. DUHMWPE used in the context of the present disclosure refers to a homopolymer or copolymer of ethylene having molecular weight in the range of 2 million Dalton to 20 million Dalton, heat of fusion in the range of 180 J/g to 250 J/g and bulk density in the range of 0.05 g/cm³ to 0.08 g/cm³ and initial elastic modulus value of 1.0 MPa, preferable < 1.0 MPa of the melt when measured by dynamic rheometry (strain: 0.5%, frequency: 10 rad/s, temperature: 180 °C).

Denier: Denier is a unit for the measurement of the linear mass density of filaments. It is defined as the mass in grams per 9000 meters the filaments.

% Filament Elongation: Specified as the percentage increase in the length of the filament when stretched as compared to the initial length of the filament, before the breaking of the filament.

Draw Ratio: Draw Ratio is expressed as the ratio of cross sectional area of the undrawn material to that of the drawn material.

Tow: A tow is an untwisted bundle of manufactured filaments.

BACKGROUND

- 5 Ultra-high molecular weight polyethylene finds numerous applications due to properties such as high abrasion resistance, high impact strength, and low coefficient of friction.

Although, UHMWPE shows good physical and mechanical properties, the melt viscosity average of the UHMWPE is high, resulting in poor homogeneity of the processed product obtained therefrom. This problem is due to high entanglement in the polymeric chains, which
10 causes restricted mobility of the polymer chains in the melt during processing.

Disentangled ultra-high molecular weight polyethylene (DUHMWPE) shows significantly low entanglement, possesses good mechanical properties, and therefore, can be processed easily.

Conventionally, DUHMWPE filaments are prepared from a compact polymeric gel of
15 DUHMWPE by the gel spinning method. The conventional gel spinning method relies on large amounts of fluid media for dissolution and extraction, leading to a slow, costly and environmentally unfriendly process. However, gel spinning has proven to be a major breakthrough in processing ultra-high molecular weight polymers. Typically, the gel-spun DUHMWPE filaments require a fluid medium to dilute and disentangle the extremely long
20 chain molecules, thus enabling a drawing process to highly orient the molecular chain for increased tenacity and tensile modulus. In gel spinning method, a compact polymeric gel is prepared from a composition comprising DUHMWPE, additives, and at least one fluid medium which requires large quantities of the fluid medium for producing DUHMWPE filaments.

- 25 Paraffin oil and decalin are the conventionally used as fluid media in the gel spinning method for preparing DUHMWPE filaments. Both these media, however, have limitations. For example, higher time is required to extract the paraffin oil from the polymer filaments and also the extraction efficiency is poor. Decalin is an expensive, highly flammable, toxic, and environmentally hazardous material.

There is, therefore, felt a need for a process for the preparation of compact polymeric gel comprising DUHMWPE and a process for the preparation of DUHMWPE filaments from the compact polymeric gel that overcomes the drawbacks mentioned herein above.

OBJECTS

- 5 Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows:

It is an object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

- 10 An object of the present disclosure is to provide a compact polymeric gel comprising DUHMWPE.

Another object of the present disclosure is to provide a process for preparing the compact polymeric gel comprising DUHMWPE.

Yet another object of the present disclosure is to provide a process for preparing DUHMWPE filaments from the compact polymeric gel.

- 15 Still another object of the present disclosure is to provide low denier DUHMWPE filaments having high tensile strength.

Other objects and advantages of the present disclosure will be more apparent from the following description, which is not intended to limit the scope of the present disclosure.

SUMMARY

- 20 The present disclosure provides a compact polymeric gel. The compact polymeric gel of the present disclosure comprises disentangled ultra-high molecular weight polyethylene (DUHMWPE) in an amount in the range of 2.5% to 30% w/w; at least one nucleator in an amount in the range of 0.08% to 4.0% w/w; at least one filler in an amount in the range of 0.03% to 1.5% w/w; at least one antioxidant in an amount in the range of 0.06% to 5.0%
25 w/w; turpentine in an amount in the range of 70% to 97% w/w and optionally, at least one lubricant in an amount in the range of 0% to 1.5% w/w.

The DUHMWPE used in the compact polymeric gel of the present disclosure can be characterized by a viscosity average molecular weight in the range of 2 million Dalton to 20 million Dalton, molecular weight distribution in the range of 2 to 20, bulk density in the range of 0.05 g/cm³ to 0.08 g/cm³, crystallinity of at least 80% and an elastic modulus of less than 1.0 MPa. The nucleator can be selected from the group consisting of 1,3:2,4-di(3,4-dimethylbenzylidene)-d-sorbitol, 1,3:2,4-di-(4-tolylidene)-d-sorbitol, 1,3:2,4-di(benzylidene)-d-sorbitol, 1,3:2,4-di-(4-ethylbenzylidene)-d-sorbitol, sodium 2,2'-methylene-bis-(4,6-di-tert-butylphenyl)phosphate, and aluminum hydroxy-bis(2,2'-methylene-bis[4,6-di(tert-butylphenyl)]phosphate. The filler can be montmorillonite modified with a quaternary ammonium salt. The antioxidant can be selected from the group consisting of tris(2,4-di-tert-butylphenyl)phosphite, pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate), 2,6-di-tert-butyl-4-methylphenol, octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate, aromatic phosphites, amines, 2,6-di-tert-butyl-4-methyl-phenol, tetrakis[methylene (3,5-di-tert-butyl-4-hydroxyhydrocinnamate)]methane, tris(2,4-di-tert-butylphenyl)phosphite, octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-1,3,5-triazine-2,4,6(1H,3H,5H)-trione, and 2,5,7,8-tetramethyl-2-(4',8',12'-trimethyltridecyl)chroman-6-ol. The lubricant can be at least one selected from the group consisting of calcium stearate, aluminium stearate, zinc stearate, stearic acid, polyethylene glycol, and polybutene. The antioxidant of the compact polymeric gel of the present disclosure is a mixture comprising a first antioxidant and a second antioxidant, wherein the weight ratio of the first antioxidant and the second antioxidant in the range of 1:2 to 2:1.

A process for preparing the compact polymeric gel is also provided. Disentangled ultra-high molecular weight polyethylene in an amount in the range of 2.5% to 30% w/w, turpentine in an amount in the range of 70% to 97% w/w, at least one nucleator in an amount in the range of 0.08% to 4.0 % w/w, at least one filler in an amount in the range of 0.03% to 1.5% w/w, at least one antioxidant in an amount in the range of 0.06% to 5.0% w/w, and optionally at least one lubricant in an amount in the range of 0% to 1.5% w/w are mixed under stirring to obtain a suspension. The suspension is heated under stirring at a temperature in the range of 50 °C to 160 °C for a time period in the range of 2 hours to 25 hours under inert atmosphere to obtain a dispersion. The dispersion is cooled to a temperature in the range of 25 °C to 40 °C to obtain a bi-phasic mixture comprising a first layer comprising free turpentine, and a second layer comprising compact polymeric gel containing turpentine. The second layer is separated from

the bi-phasic mixture using conventional methods, typically filtration/ decantation, to obtain the compact polymeric gel containing turpentine. The excess/free turpentine, which is present in the first layer can be recovered in an amount of at least 40%.

Further, DUHMWPE filaments are prepared from the compact polymeric gel, wherein the
5 DUHMWPE filaments are characterized by a tensile strength in the range of 2.5 GPa to 15 GPa, tensile modulus in the range of 150 GPa to 250 GPa, and denier in the range of 0.5 d to 5 d. The DUHMWPE filaments, preferably, are twisted to obtain a gel spun yarn, characterized by, denier in the range of 2.45 d to 18 d and the tensile strength of the gel spun yarn being at least 3 times the tensile strength of DUHMWPE filaments at a temperature in
10 the range of 140 °C to 150 °C.

A process for preparing the DUHMWPE filaments from the compact polymeric gel of the present disclosure is disclosed. The compact polymeric gel is spun at a temperature ranging from 120 °C to 200 °C, at a pressure ranging from 2 kg/cm² to 3 kg/cm² to obtain a tow of gel spun filaments. The tow of gel spun filaments can be quenched at a temperature in the range
15 of 5 °C to 30 °C to obtain a quenched tow of gel spun filaments. The quenched tow of gel spun filaments is passed through at least one washing medium to remove excess turpentine and to obtain a wet tow of gel spun filaments. The wet tow of gel spun filaments can be dried at a temperature in the range of 80 °C to 125 °C to obtain a dried tow of gel spun filaments. Finally, the dried tow of gel spun filaments is subjected to hot stretching, either in single step
20 or in multi-stage hot stretching, at a temperature in the range of 90 °C to 150 °C and at a draw ratio in the range of 5 to 100 to obtain DUHMWPE filaments.

The process step of spinning can be carried out by passing the compact polymeric gel through a spinneret having hole size in the range of 0.3 mm to 1.0 mm. An extruder can also be used for the spinning of compact polymeric gel.

25 The quenching medium can be at least one selected from the group consisting of water, and cooling gas.

The washing medium used for the removal of excess turpentine from the quenched filament can be at least one selected from the group consisting of acetone, water, hexane, ethanol, ether, cyclohexanone, 2-methylpentanone, dichloromethane, and dioxane.

30

DETAILED DESCRIPTION

Gel spinning is used for processing the disentangled ultra-high molecular weight polyethylene (DUHMWPE) to produce high strength and high modulus filaments. The unique properties of DUHMWPE filaments are due to their fully extended and aligned chain configuration. In fact, the gel-spinning process discourages the formation of folded chain lamellae and favors formation of “extended chain” structure that more efficiently transmit the tensile loads.

The conventional gel spinning method involves the use of a fluid medium, such as, decalin for the preparation of DUHMWPE filaments. However, decalin is a hazardous chemical. Further, the use of high amount of decalin makes the process uneconomical due to high cost of decalin.

The present disclosure envisages a compact polymeric gel and a process for preparing the same that mitigates the drawback mentioned hereinabove.

In one aspect, the present disclosure provides a compact polymeric gel. The compact polymeric gel of the present disclosure comprises DUHMWPE, at least one nucleator, at least one filler, at least one antioxidant, turpentine, and optionally, at least one lubricant.

Turpentine is found to be an effective and environment friendly fluid medium for preparing the compact polymeric gel of the present disclosure. Turpentine helps in preparing the compact polymeric gel and subsequently for spinning the compact polymeric gel and producing DUHMWPE filaments showing high quality and uniform denier in the range of 0.5 d to 150 d.

The compact polymeric gel of the present disclosure comprises DUHMWPE in an amount in the range of 2.5% to 30% w/w, at least one nucleator in an amount in the range of 0.08% to 4.0% w/w, at least one filler in an amount in the range of 0.03% to 1.5% w/w, at least one antioxidant in an amount in the range of 0.06% to 5.0% w/w, turpentine in an amount in the range of 70% to 97% w/w, and optionally, at least one lubricant in an amount in the range of 0% to 1.5% w/w.

The viscosity average molecular weight of the DUHMWPE can be in the range of 2 million Dalton to 20 million Dalton. The molecular weight distribution of the DUHMWPE can be in the range of 2 to 20. The bulk density of the DUHMWPE is significantly lower (less than

0.15 g/cm³), as compared to the bulk density of the conventional UHMWPE (greater than 0.40 g/cm³), which facilitates faster absorption of fluid medium and thereby produces homogeneous slurry. The bulk density of the DUHMWPE can be in the range of 0.05 g/cm³ to 0.08 g/cm³. The crystallinity of the DUHMWPE polymer can be at least 80%. The initial
 5 elastic modulus of the DUHMWPE can be less than 1.0 MPa.

The nucleator can be at least one selected from the group consisting of 1,3:2,4-di(3,4-dimethylbenzylidene)-d-sorbitol, 1,3:2,4-di-(4-tolylidene)-d-sorbitol, 1,3:2,4-di(benzylidene)-d-sorbitol, 1,3:2,4-di-(4-ethylbenzylidene)-d-sorbitol, sodium 2,2'-methylene-bis-(4,6-di-tert-butylphenyl)phosphate, and aluminum hydroxy-bis(2,2'-methylene-bis[4,6-di(tert-butyl)phenyl]phosphate.
 10

Fillers are added to improve the creep resistance and thermal stability of the DUHMWPE filaments produced from the compact polymeric gel. The filler is montmorillonite modified with a quaternary ammonium salt.

Further, antioxidants are used to prevent the thermal degradation of the gel during spinning,
 15 stretching and/or extrusion. The antioxidant of the present disclosure can be selected from the group consisting of tris(2,4-di-tert-butylphenyl)phosphite, pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate), 2,6-di-tert-butyl-4-methylphenol, octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate, aromatic phosphites, amines, 2,6-di-tert-butyl-4-methyl-phenol, tetrakis[methylene (3,5-di-tert-butyl-4-hydroxyhydrocinnamate)]methane,
 20 tris(2,4-di-tert-butylphenyl) phosphite, octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-1,3,5-triazine-2,4,6(1H,3H,5H)-trione, and 2,5,7,8-tetramethyl-2-(4',8',12'-trimethyltridecyl)chroman-6-ol. The lubricant can be at least one selected from the group consisting of calcium stearate, aluminium stearate, zinc stearate, stearic acid, polyethylene glycol, and polybutene

25 In accordance with the present disclosure, the antioxidant is a mixture comprising a first antioxidant and a second antioxidant, wherein the weight ratio of the first antioxidant and the second antioxidant in the range of 1:2 to 2:1.

In another aspect of the present disclosure, there is provided a process for preparing the compact polymeric gel. The process for preparing the compact polymeric gel is described
 30 herein below.

Initially, the following ingredients are mixed under stirring to obtain a suspension:

DUHMWPE in an amount in the range of 2.5% to 30% w/w, turpentine in an amount in the range of 70% to 97% w/w, at least one nucleator in an amount in the range of 0.08% to 4.0% w/w, at least one filler in an amount in the range of 0.03% to 1.5% w/w, at least one antioxidant in an amount in the range of 0.06% to 5.0% w/w, and optionally, at least one
5 lubricant in an amount in the range of 0% to 1.5% w/w. The suspension is heated under stirring at a temperature in the range of 50 °C to 160 °C for a time period in the range of 2 hours to 25 hours under inert atmosphere to obtain a dispersion. Preferably, nitrogen gas is used to provide the inert atmosphere. The dispersion is cooled to a temperature in the range of 25 °C to 40 °C to obtain a bi-phasic mixture comprising a first layer and a second layer.
10 The first layer comprises free turpentine and the second layer comprises the compact polymeric gel containing turpentine. The second layer, comprising the compact polymeric gel, is separated from the bi-phasic mixture using conventional methods, typically filtration/decantation, to obtain the compact polymeric gel containing turpentine. The excess/free turpentine, which is present in the first layer can be recovered in an amount of at least 40%.
15 The turpentine so recovered can be recycled and reused. The recovery and the recycling of turpentine make the process of the present disclosure economical and efficient.

The compact polymeric gel of the present disclosure is used to prepare DUHMWPE filaments. The presence of turpentine in an amount in the range of 70% to 97% w/w in the compact polymeric gel has an advantage to enhance the production efficiency of the gel spun
20 DUHMWPE filaments preparation process as well as to improve mechanical properties of the DUHMWPE filaments produced therefrom. The shelf life of the compact polymeric gel is high; the gel spun DUHMWPE filaments can be prepared even after a time period of six months from the compact polymeric gel. Further, DUHMWPE filaments having improved mechanical properties can be obtained after hot stretching the gel spun DUHMWPE
25 filaments.

In accordance with an embodiment of the present disclosure, DUHMWPE filaments are prepared from the compact polymeric gel. The DUHMWPE filaments, so obtained, are characterized by a tensile strength in the range of 2.5 GPa to 15 GPa, tensile modulus in the range of 150 GPa to 250 GPa, and denier in the range of 0.5 d to 5 d. Further, the
30 DUHMWPE filaments, preferably, are twisted to obtain a gel spun yarn which is characterized by denier in the range of 2.45 d to 18 d and the tensile strength of the gel spun yarn being at least 3 times the tensile strength of DUHMWPE filaments at a temperature in the range of 140 °C to 150 °C.

In still another aspect of the present disclosure, there is provided a process for preparing disentangled ultra-high molecular weight polyethylene (DUHMWPE) filaments from the compact polymeric gel. The process for preparing the DUHMWPE filaments from the compact polymeric gel is described herein below.

- 5 Firstly, the compact polymeric gel is subjected to spinning at a temperature in the range of 120 °C to 200 °C, and at a pressure in the range of 2 kg/cm² to 3 kg/cm² to obtain a tow of gel spun filaments.

In an embodiment of the present disclosure, the compact polymeric gel can be subjected to spinning through a spinneret having hole size in the range of 0.3 mm to 1.0 mm to produce a
10 tow of gel spun filaments.

In another embodiment of the present disclosure, the compact polymeric gel can be subjected to extrusion. The compact gel can be extruded on a single screw extruder to produce a tow of gel spun filaments, at a temperature in the range of 220 °C to 250 °C. The extruder can be operated at an rpm in the range of 5 to 150.

- 15 In accordance with the embodiments of the present disclosure, the tow of gel spun filaments can be one of a monofilament tow and a multifilament tow.

The tow of gel spun filaments can be quenched in a quenching medium at a temperature in the range of 5 °C to 30 °C to obtain a quenched tow of gel spun filaments. At this temperature, the amorphosity and crystallinity are regulated, so that the DUHMWPE
20 filaments produced from the tow of gel spun filaments show improved tensile properties.

The tow of gel spun filaments obtained after extrusion and/or spinning can be aerially stretched by adjusting the distance between spinneret and the quench bath, such that quenched tow of gel spun filaments shows uniform amorphosity and crystallinity.

In an exemplary embodiment of the present disclosure, water at a temperature of 8 °C is used
25 as a quenching media.

The quenching of the tow of gel spun filaments can be achieved by using different quenching media arranged in a series. The quenching media can be selected from the group consisting of water, and cooling gas. The cooling gas used for the quenching of the tow of gel spun filaments can be at least one selected from the group consisting of air, carbon dioxide, and
30 nitrogen.

Further, the tow of gel spun filaments can be passed through a washing medium to remove maximum amount of turpentine from the quenched tow of gel spun filaments and to produce a wet tow of gel spun filaments. The washing medium used for the removal of turpentine from the quenched tow of gel spun filaments can be at least one selected from the group
5 consisting of acetone, water, hexane, ethanol, ether, cyclohexanone, 2-methylpentanone, dichloromethane, and dioxane. The wet tow of gel spun filaments can be dried at a temperature in the range of 80 °C to 125 °C to obtain a dried tow of gel spun filaments.

The dried tow of gel spun filaments can be hot stretched at a temperature in the range of 90 °C to 150 °C, and at a draw ratio in the range of 5 to 100 to obtain DUHMWPE filaments.

10 The DUHMWPE filaments produced by the process of the present disclosure show high tenacity and high modulus. These properties make them suitable for use in defense sector and medical sector.

In accordance with the process of the present disclosure, the hot stretching can be either single step or multi-step hot stretching. The DUHMWPE filaments of the present disclosure
15 obtained after hot stretching of the gel spun filaments show high tensile strength and high tensile modulus. The mechanical properties of the DUHMWPE filaments are retained at higher temperatures, such as at 145 °C which make them suitable for application at high temperatures.

DUHMWPE filaments obtained by the process of the present disclosure involving turpentine
20 as the fluid medium showed improved mechanical properties as compared to the DUHMWPE filaments prepared using decalin as the fluid medium.

In accordance with the embodiments of the present disclosure, the DUHMWPE filaments obtained by the process of the present disclosure can be further twisted to obtain a gel spun yarn.

25 The present disclosure is further illustrated herein below with the help of the following experiments. The experiments used herein are intended merely to facilitate an understanding of the ways in which the embodiments herein may be practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the experiments should not be construed as limiting the scope of embodiments herein. These laboratory scale
30 experiments can be scaled up to industrial/commercial scale and the results obtained can be extrapolated to industrial/commercial scale.

Experiments:**Experiment 1: Low denier DUHMWPE filaments having improved mechanical properties***Experiment 1A: Preparation of Turpentine based compact polymeric gel*

- 5 A turpentine based compact polymeric gel, GEL-1; of disentangled ultra-high molecular weight polyethylene (DUHMWPE) was prepared. The materials used for the preparation of the compact polymeric gel, are provided in **Table 1** herein below.

Table 1: Materials used for the preparation of the compact polymeric gel

S.No.	Component	Material(s)	Quantity (%) w/w
			GEL-1
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 4.5 million Dalton	12.00
2.	Fluid Medium	Turpentine	85.50
3.	Antioxidant	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)	0.50
		Second Antioxidant- tris(2,4-di-tert-butylphenyl)phosphite	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20
5.	Filler	Montmorillonite modified with a quaternary ammonium salt	0.05
6.	Lubricant	Calcium stearate	1.50

- 10 DUHMWPE, having viscosity average molecular weight of 4.5 million Dalton, was used for the preparation of the compact polymeric gel. The bulk density of the DUHMWPE was 0.06 g/cm³ and crystallinity was 96 %. Materials provided in **Table 1** were mixed in a reactor under stirring to obtain a suspension. The suspension was heated under continuous stirring at 200 rpm at 145 °C under inert atmosphere of nitrogen for 3 hours to obtain a dispersion.
- 15 After complete dissolution, the reactor was allowed to cool to 25°C to obtain a bi-phasic mixture containing a first layer comprising turpentine and a second layer comprising the

compact polymeric gel. The second layer was separated from the bi-phasic mixture to obtain the compact polymeric gel, GEL-1.

Further, GEL-2, GEL-3, and GEL-4 were prepared using the similar procedure as mentioned for GEL-1 above. The materials used for the preparation of GEL-2, GEL-3 and GEL-4 are given herein **Table 2** below.

10

Table 2: Materials used for the preparation of compact polymeric gels

S.No.	Component	Material(s)	Quantity (%) w/w		
			GEL-2	GEL-3	GEL-4
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 5 million Dalton	5.00	---	---
		DUHMWPE: Viscosity average Molecular weight = 1.87 million Dalton	---	14.00	---
		DUHMWPE: Viscosity average Molecular weight = 4.5 million Dalton	---	---	5.00
2.	Fluid Medium	Turpentine	92.50	83.50	92.50
3.	Antioxidant	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)	0.50	0.50	0.50
		Second Antioxidant- tris(2,4-di-tert-butylphenyl) phosphite	0.25	0.25	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20	0.20	0.20
5.	Filler	Montmorillonite modified with a quaternary ammonium salt	0.05	0.05	0.05
6.	Lubricant	Calcium stearate	1.50	1.50	1.50

Experiment 1B: Preparation of the gel spun DUHMWPE filaments

The compact polymeric gel, GEL-1, obtained in **Experiment 1A** was extruded through a spinneret to obtain a tow of gel spun filaments of GEL-1. The tow of gel spun filaments was quenched using a water bath maintained at 8 °C to obtain a quenched tow of gel spun
 5 filaments. The quenched tow of gel spun filaments was passed through a water bath, followed by an acetone bath at 25 °C, and subsequently dried to obtain a dried tow of gel spun DUHMWPE filaments.

Further, using the procedure described hereinabove for GEL-1, dried tow of gel spun DUHMWPE filaments were prepared from the turpentine based compact polymeric gels,
 10 GEL-2, GEL-3 and GEL-4.

The hole size of the spinneret used for spinning and the denier of the dried tow of gel spun DUHMWPE filaments obtained from the GEL-1, GEL-2, GEL-3 and GEL-4 are tabulated in **Table 3**.

15 **Table 3: Spinneret size and the denier of the dried tow of gel spun DUHMWPE filaments obtained**

Experiment No.	Corresponding GEL	Spinneret hole size (mm)	Denier of the DUHMWPE filament
2	GEL-1	0.7	65 d
3	GEL-2	0.5	45 d
4	GEL-3	0.5	70 d
5	GEL-4	0.4	44 d

Experiment 1C: Hot stretching of the gel spun DUHMWPE filaments

The dried tow of gel spun DUHMWPE filaments obtained from GEL-1 in **Experiment 1B**
 20 was hot stretched to obtain DUHMWPE filaments. Similarly, the tow of gel spun DUHMWPE filaments obtained from GEL-2, GEL-3 and GEL-4 in **Experiment 1B** were also hot stretched to obtain corresponding DUHMWPE filaments.

Mechanical properties of the DUHMWPE filaments and the tow of gel spun DUHMWPE filaments obtained from GEL-1, GEL-2, GEL-3 and GEL-4 are provided in **Table 4** herein below.

Table 4: Comparison of the mechanical properties of the DUHMWPE filaments (hot stretched) with the gel spun DUHMWPE filaments

S. No.	Gel	Filament Type	Hot stretching		Denier	TS (GPa)	TM (GPa)	Elongation (%)
			Temp. (°C)	Draw ratio (TDR)				
1.	GEL-1	gel spun DUHMWPE filaments	---	---	65 d	0.55	1.75	125
		DUHMWPE filaments (hot stretched)	145	25	2.25 d	4.76	175	2.67
2.	GEL-2	gel spun DUHMWPE filaments	---	---	45 d	0.85	3.50	110
		DUHMWPE filaments (hot stretched)	145	25	1.80 d	5.78	189	2.25
3.	GEL-3	gel spun DUHMWPE filaments	---	---	70 d	0.49	1.55	110
		DUHMWPE filaments (hot stretched)	145	23	1.95 d	3.79	182	2.75
4.	GEL-4	gel spun DUHMWPE filaments	---	---	44 d	0.55	1.25	109
		DUHMWPE filaments (hot stretched)	143	21	2 d	3.15	169	2.79

TS = tensile Strength, TM = tensile Modulus

From **Table 4**, it is evident that the tensile strength (TS) and tensile modulus (TM) of the DUHMWPE filaments obtained after the hot stretching were higher than the corresponding gel spun DUHMWPE filaments. Further, denier and % elongation for the DUHMWPE

filaments were lower as compared to the gel spun DUHMWPE filaments. Thus, the compact polymeric gel of the present disclosure produces DUHMWPE filaments having improved mechanical properties.

Experiment 2 to 7: Comparative Experiments Set-1

5 Experiment 2: Compact polymeric gel and gel spun DUHMWPE filaments using Turpentine as the fluid medium

Experiment 2A: Preparation of Turpentine based compact polymeric gel

A turpentine based compact polymeric gel; GEL-5 was prepared from DUHMWPE. The materials used for the preparation of compact polymeric gel, are provided in **Table 5** herein below.

Table 5: Materials used for the preparation of compact polymeric gel

S.No.	Component	Material(s)	Quantity (%) w/w
			GEL-5
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 4.5 million Dalton	7.50
2.	Fluid Medium	Turpentine	90.00
3.	Antioxidant	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4- hydroxyphenyl)propionate)	0.50
		Second Antioxidant- tris(2,4-di-tert- butylphenyl) phosphite	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20
5.	Filler	Montmorillonite modified with a quaternary ammonium salt	0.05
6.	Lubricant	Calcium stearate	1.50

DUHWPPE, having viscosity average molecular weight 4.5 million Dalton, was used for the preparation of the compact polymeric gel. The bulk density of the DUHWPPE was 0.06 g/cm³ and crystallinity was 96 %.

Materials provided in **Table 5** were mixed in a reactor under stirring to obtain a suspension.

- 5 The suspension was heated at a temperature of 145 °C under inert atmosphere of nitrogen with continuous stirring at 200 rpm, and the temperature of the reactor was maintained at 145 °C for 3 hours to obtain a dispersion. After complete dissolution, the reactor was allowed to cool to 25°C, to obtain a bi-phasic mixture, containing a first layer comprising turpentine and a second layer comprising the compact polymeric gel. The second layer was separated to
10 obtain the compact polymeric gel, GEL-5.

Experiment 2B: Preparation of gel spun DUHWPPE filaments

- The compact polymeric gel, GEL-5, obtained in **Experiment 2A** was extruded at 160 °C through a spinneret having hole size of 0.5 mm, at a pressure of 3 kg/cm² to obtain a
15 monofilament tow of gel spun filament obtained from GEL-5. The gel spun filament had a denier of 3.5 d. The tow of gel spun filament obtained from GEL-5 was quenched using a water bath maintained at 8 °C to obtain a quenched monofilament tow of gel spun filament. After quenching in the water bath, the quenched monofilament tow of gel spun filament obtained from GEL-5 was passed through a water bath, followed by an acetone bath at 25 °C,
20 and subsequently dried to obtain a dried monofilament tow of gel spun filament collected on a spool at a winding speed of 25 m/min.

Experiment 3: Compact polymeric gel and the gel spun DUHWPPE filaments using Decalin as the fluid medium

- 25 *Experiment 3A: Preparation of Decalin based compact polymeric gel*

- A compact polymeric gel of DUHWPPE, GEL-6, was prepared using procedure similar to **Experiment 2A**. The materials and their respective amount(s) of used for the preparation of compact polymeric gel were same as those used in **Experiment 2A**, except that decalin was used as the fluid medium in place of turpentine. The decalin based compact polymeric gel
30 was prepared at a temperature of 170 °C, which was higher as compared to the process for the preparation of turpentine based compact polymeric gel.

Experiment 3B: Preparation of gel spun DUHWPPE filaments

The compact polymeric gel, GEL-6, was spun at 172 °C and a dried monofilament tow of gel

spun filament was obtained using the experimental procedure similar to **Experiment 2B**.

The mechanical properties of the dried monofilament tow of the gel spun filament obtained from GEL-5 and GEL-6 are provided in **Table 6**. The gel spun DUHMWPE filament obtained from GEL-5 is represented by TMF-1 and the gel spun DUHMWPE filament
5 obtained from GEL-6 is represented by DMF-1.

Table 6: Comparison of mechanical properties of gel spun DUHMWPE filament produced from Turpentine based compact polymeric gel and Decalin based compact polymeric gel

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Expt. no.	Filament sample	Fluid medium	Dissolution temp. (°C)	Denier	TS (GPa)	TM (GPa)	Elongation (%)
2B	TMF-1	Turpentine	145	3.50 d	1.25	4.56	76
3B	DMF-1	Decalin	170	15 d	0.69	1.59	125

TS = Tensile strength, TM = Tensile Modulus

From **Table 6**, it is evident that the dissolution temperature for the spinning was lower (145 °C) for turpentine as compared to decalin. Further, turpentine based gel spun DUHMWPE
15 filament showed higher tensile strength and tensile modulus with lower denier as compared to decalin based gel spun DUHMWPE filament.

Experiments 4 to 7:

Hot stretching of the gel spun DUHMWPE filaments of GEL-5 and GEL-6

The dried monofilament tow of gel spun filament prepared from GEL-5 and GEL-6, were
20 further subjected to two-step hot stretching at 125 °C and 145 °C respectively.

The results are summarized in **Table 7**. Precursor from GEL-5, i.e. gel spun DUHMWPE filament obtained from GEL-5 is represented by TMF-1L. The gel spun DUHMWPE filament of GEL-5 was subjected to two step hot stretching at 125 °C and 145 °C at a total draw ratio of 8.5. The hot stretched filament so obtained is represented by TMF-1H.
25 Similarly, precursor from GEL-6, i.e. gel spun DUHMWPE filament obtained from GEL-6, is represented by DMF-1L. For comparison, the gel spun DUHMWPE filament of GEL-6

was also subjected to two step hot stretching at 125 °C and 145 °C at a total draw ratio of 8.25. The hot stretched filament so obtained is represented by DMF-1H. Mechanical properties of both the gel spun filaments and the hot stretched filaments were measured at ambient temperature of 25 °C.

5 **Table 7: Mechanical properties of the DUHMWPE filaments obtained after two-step hot stretching:**

Exp. No.	Fluid medium	Filament sample		TS (GPa)	TM (GPa)	Elongation (%)	Denier after Hot Stretching	Total draw ratio (TDR)
4	Turpentine	TMF-1L		1.25	4.56	76	---	8.5
5		TMF-1H		5.21	185	2.21	0.65 d	
6	Decalin	DMF-1L		0.45	0.79	133	---	8.25
7		DMF-1H		5.26	195	1.95	2.32 d	

TS = Tensile strength, TM = Tensile modulus

From **Table 7**, it is evident that the turpentine based DUHMWPE filaments obtained after two-step hot stretching had lower denier as compared to decalin based DUHMWPE filaments produced under the identical experimental conditions. Even though, the turpentine based DUHMWPE filaments had lower denier, the mechanical properties of the turpentine based DUHMWPE filaments were similar to the decalin based DUHMWPE filaments having higher denier.

These results indicated a rapid ordering or orientation of macromolecular chains during hot stretching process with turpentine as the fluid medium as compared to decalin. Thus, the turpentine based low denier DUHMWPE filaments show similar mechanical properties as compared to the decalin based higher denier DUHMWPE filaments.

Experiments 8 to 11: Comparative Experiments Set-2

Experiment 8A: Preparation of Turpentine based compact polymeric gel

A turpentine based compact polymeric gel, GEL-7, containing 5.5 wt% DUHMWPE was prepared using the experimental procedure similar to **Experiment 2A**. DUHMWPE having a viscosity average molecular weight of 3.5 million Dalton was used. The materials used for the preparation of compact polymeric gel are given in **Table 8** herein below.

5 **Table 8: Materials used for the preparation of Turpentine based compact polymeric gel**

S.No.	Component	Material(s)	Quantity (%) w/w GEL-7
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 3.5 million Dalton	5.50
2.	Fluid Medium	Turpentine	92.00
3.	Antioxidant(s)	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4- hydroxyphenyl)propionate)	0.50
		Second Antioxidant- tris(2,4-di- tert-butylphenyl) phosphite	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20
5.	Filler(s)	Montmorillonite modified with a quaternary ammonium salt	0.05
6.	Lubricant	Calcium stearate	1.50

DUHMWPE, having viscosity average molecular weight 3.5 million Dalton, was used for the preparation of the compact polymeric gel. Bulk density of the DUHMWPE was 0.06 g/cm³ and crystallinity was 96 %. Materials provided in **Table 8** were mixed in a reactor under stirring to obtain a suspension. The turpentine based compact polymeric gel, GEL-7, was prepared using the experimental procedure similar to **Experiment 2A**.

Experiment 8B: Preparation of gel spun DUHMWPE filaments

The turpentine based compact polymeric gel, GEL-7, obtained in **Experiment 8A** was extruded at 160 °C through a spinneret having hole size of 0.5 mm, at a pressure of 3 kg/cm² to obtain a monofilament tow of gel spun filament of GEL-7 having a denier of 2.45 d, followed by quenching the monofilament tow of gel spun filament of GEL-7 using a water

bath maintained at 8 °C to obtain a quenched monofilament tow of gel spun DUHMWPE filament. After quenching in the water bath, the quenched monofilament tow of gel spun DUHMWPE filament obtained from GEL-7 was passed through a water bath, followed by an acetone bath at 25 °C, and subsequently dried to obtain a dried monofilament tow of gel spun DUHMWPE filament which was collected on a spool at a winding speed of 25 m/min.

Experiment 9: Decalin based compact polymeric gel and gel spun DUHMWPE filaments obtained therefrom

Experiment 9A: Preparation of Decalin based DUHMWPE gel spun filaments

For comparison, a compact polymeric gel, GEL-8, was prepared using the similar composition as provided in **Table 8** above, except that decalin was used as the fluid medium. The process steps and conditions were similar to **Experiment 2A** for the preparation of the decalin based compact polymeric gel, GEL-8.

Experiment 9B: Preparation of gel spun DUHMWPE filaments

The decalin based compact polymeric gel, GEL-8, was used to obtain a dried monofilament tow of gel spun DUHMWPE filament using the experimental procedure similar to **Experiment 8B**.

The gel spun DUHMWPE filament of the dried monofilament tow of gel spun filament obtained from GEL-7 and GEL-8 were compared for their mechanical properties, and the data thus obtained is given herein **Table 9** below. The gel spun DUHMWPE filaments obtained from GEL-7 and GEL-8 are represented by TMF-2 and DMF-2, respectively.

Table 9: Mechanical properties of Turpentine and Decalin based gel spun DUHMWPE filaments

Experiment No.	Filament Sample	Fluid medium	Denier	TS (GPa)	TM (GPa)	% Elongation
8B	TMF-2	Turpentine	2.45 d	1.40	5.35	72
9B	DMF-2	Decalin	18 d	0.45	0.79	133

TS = Tensile strength, TM = tensile Modulus

Experiments 10 and 11:**Hot stretching of the gel spun DUHMWPE filaments of GEL-7 and GEL-8**

Further, the dried monofilament tow of gel spun DUHMWPE filament obtained from GEL-7 and GEL-8 were separately subjected to hot stretching at elevated temperatures (143 °C and 150 °C) to obtain hot stretched DUHMWPE filaments. The mechanical properties of the DUHMWPE filaments obtained after hot stretching were compared with the mechanical properties of the dried monofilament tow of gel spun DUHMWPE filaments.

The data thus obtained is tabulated in **Table 10** herein below. The DUHMWPE filaments obtained from GEL-7 are represented by TMF-3, and the DUHMWPE filaments obtained from GEL-8 are represented by DMF-3.

Table 10: Comparison of the mechanical properties of the Turpentine based DUHMWPE filaments and Decalin based DUHMWPE filaments at high temperatures

Exp. No.	Filament Sample (hot stretched)	TS at 143 °C (GPa)	TM at 150 °C (GPa)	Filament Sample (gel spun)	TS at 25 °C (GPa)	TM at 25 °C (GPa)
10	TMF-3	3.55	5.52	TMF-2	1.40	5.35
11	DMF-3	1.04	1.50	DMF-2	0.45	0.79

TS = Tensile strength, TM = tensile Modulus

Thus, the DUWMWPE filaments obtained from turpentine based compact polymeric gel, GEL-7, showed higher tensile strength and tensile modulus at higher temperature (143 °C and 150 °C) as compared to the DUWMWPE filaments obtained from the decalin based compact polymeric gel, GEL-8.

Turpentine based DUHMWPE filaments showed rapid orientation of macromolecular chain at higher temperature. The retention of higher tensile strength at higher temperature makes turpentine based DUHMWPE filaments useful for high temperature applications.

Experiments 12 to 17: Multi-stage hot stretching of the gel spun DUHMWPE filaments

A turpentine based compact polymeric gel, GEL-9, containing 4.5 wt% DUHMWPE was prepared using the experimental procedure similar to **Experiment 2A**. DUHMWPE having

viscosity average molecular weight of 4.5 million Dalton, bulk density of 0.06 g/cm³, and crystallinity of 95 % was used. The materials used for the preparation of the compact polymeric gel are given in **Table 11** herein below.

Table 11: Materials used for the preparation of compact polymeric gel

S.No.	Component	Material(s)	Quantity (%) w/w GEL-9
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 4.5 million Dalton	4.50
2.	Fluid medium	Turpentine	93.00
3.	Antioxidant(s)	First Antioxidant - pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4- hydroxyphenyl)propionate)	0.50
		Second Antioxidant - tris(2,4-di-tert- butylphenyl) phosphite	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20
5.	Filler(s)	Montmorillonite modified with a quaternary ammonium salt	0.05
6.	Lubricant	calcium stearate	1.50

5

For comparison, a compact polymeric gel, GEL-10, was prepared using the similar composition as given in **Table 11** above, except that decalin was used as the fluid medium. The gels, GEL-9 and GEL-10, were used to prepare gel spun DUHMWPE filaments. The compact polymeric gels, GEL-9 and GEL-10, were separately extruded at 170 °C and at 50 rpm using single hole spinneret having hole size of 0.7 mm. The gel spun DUHMWPE filament (THDF) obtained from GEL-9 and gel spun DUHMWPE filament (DHDF) obtained from GEL-10 were separately subjected to two step hot stretching (HS-1 and HS-2) to obtain DUHMWPE filaments. The DUHMWPE filaments were tested for their mechanical properties. The DUHMWPE filaments obtained from GEL-9 are represented by TMF-0, TMF-4 and TMF-5. The DUHMWPE filaments obtained from GEL-10 are represented by DMF-0, DMF-4 and DMF-5. The results thus obtained are provided herein **Table 12** below.

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Table 12: Comparison of the mechanical properties of Turpentine based DUHMWPE filaments and Decalin based DUHMWPE filaments before and after two step hot stretching

Expe- riment	Filament sample	Filament type	TS (GPa)	TM (GPa)	% Elongation	Denier	Total Draw Ratio (TDR)
12	TMF-0	Before- HS	0.55	2.5	135	180 d	---
13	TMF-4	After- HS-1	3.95	95	4.75	4.18 d	33.00
14	TMF-5	After- HS-2	4.76	175	1.85	1.75 d	45.00
15	DMF-0	Before- HS	0.25	0.52	150	257 d	---
16	DMF-4	After- HS-1	3.80	60	5.25	7.5 d	33.00
17	DMF-5	After- HS-2	4.53	170	1.50	2.15 d	44.55

TS = Tensile strength, TM = tensile Modulus

- 5 From **Table 12**, it is evident that the turpentine based DUHMWPE filament showed higher tensile strength and tensile modulus as compared to the decalin based DUHMWPE filament.

Experiment 18 to 20: Effect of DUHMWPE concentration on the Mechanical properties of the gel spun DUHMWPE filaments

- 10 A turpentine based compact polymeric gel, GEL-11; containing 2.75 wt% DUHMWPE polymer was prepared using the experimental procedure similar to **Experiment 2A**. DUHMWPE having viscosity average molecular weight of 4 million Dalton, bulk density of 0.06 g/cm^3 and crystallinity of 95 % was used. The amounts of different materials used for the preparation of the compact polymeric gel are given herein **Table 13** below.

Table 13: Materials used for the preparation of compact polymeric gel

S.No.	Component	Material(s)	Quantity (%)
-------	-----------	-------------	--------------

			GEL-11
1.	Polyethylene	DUHMWPE	2.75
2.	Fluid Medium	Turpentine	94.50
3.	Antioxidant	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)	0.50
		Second Antioxidant- tris(2,4-di-tert-butylphenyl) phosphite	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20
5.	Filler	Montmorillonite modified with a quaternary ammonium salt	0.05
6.	Lubricant	Calcium stearate	1.75

Further, two more compact polymeric gels, GEL-12 and GEL-13, were prepared using 5% w/w and 10% w/w DUHMWPE, respectively. The amounts of turpentine used for the preparation of GEL-12 and GEL-13 were 92.25% w/w and 87.25% w/w respectively. The compact polymeric gels GEL-11, GEL-12 and GEL-13 were separately extruded at a temperature of 165 °C to obtain the gel spun DUHMWPE filaments of GEL-11, GEL-12 and GEL-13 using the experimental procedure similar to **Experiment 2B**. The mechanical properties of the gel spun DUHMWPE filaments of GEL-11, GEL-12 and GEL-13 were compared. The spinning conditions used and the mechanical properties of the gel spun DUHMWPE filaments of GEL-11, GEL-12 and GEL-13 are tabulated in **Table 14** below. The gel spun DUHMWPE filaments obtained from GEL-11, GEL-12 and GEL-13 are represented by TMF-6, TMF-7 and TMF-8, respectively.

Table 14: Spinning conditions and the mechanical properties of the gel spun DUHMWPE filaments with different amounts of DUHMWPE used in the compact polymeric gel

Exp. No.	Filament sample	DUHMWPE amount (wt. %)	Quenching bath Temp. °C	Wash bath Temp. (°C)	Winding speed (m/min)	TS (GPa)	TM (GPa)

18	TMF-6	2.75	5	25	25	2.50	135
19	TMF-7	5.00	5	25	25	3.75	162
20	TMF-8	10.00	5	25	25	5.25	175

TS = Tensile strength, TM = tensile Modulus

From **Table 14**, it is evident that the tensile strength and tensile modulus of the gel spun DUHMWPE filaments obtained from the compact polymeric gel comprising 2.75 wt% of the DUHMWPE were lowest among these three concentrations. Whereas, the tensile strength and tensile modulus of the gel spun DUHMWPE filaments obtained from the compact polymeric gel comprising 10 wt% of the DUHMWPE were the highest among the three concentrations. Thus, as the amount of DUHMWPE in the compact polymeric gel increased, the tensile strength and tensile modulus of the gel spun DUHMWPE filaments also increased.

Experiment 21 and 22: Effect of the amount of fillers, additives, nucleators and antioxidants on the mechanical properties of the DUHMWPE filaments obtained after hot stretching:

Two compact polymeric gels, GEL-14 and GEL-15, both containing DUHMWPE having viscosity average molecular weight of 5 million Dalton, were prepared using turpentine as a fluid medium under the same DUHMWPE polymer concentration with the variation in the amount of fillers, additives, nucleators and antioxidants. The amounts of these components used for preparing GEL-15 was half of the amount used for preparing GEL-14. The amounts of the materials used for the preparation of the compact polymeric gel are given herein **Table 15** below.

Table 15: Materials used for the preparation of compact polymeric gel

S.N o.	Component	Materials	Quantity (%) w/w	Quantity (%) w/w
			GEL-14	GEL-15
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 5.0 million Dalton	4.50	4.50
2.	Fluid Medium	Turpentine	94.00	94.75
3.	Antioxidant(s)	First Antioxidant -	0.50	0.25

		pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)		
		Second Antioxidant- tris(2,4-di-tert-butylphenyl) phosphite	0.25	0.125
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20	0.08
5.	Filler(s)	Montmorillonite modified with a quaternary ammonium salt	0.05	0.03
6.	Lubricant	Calcium stearate	0.50	0.25

GEL-14 and GEL-15 were used to obtain gel spun DUHMWPE filaments using the experimental procedure similar to **Experiment 1B**. The gel spun DUHMWPE filaments obtained from GEL-14 and GEL-15, having denier of 25 d, were further subjected to single step hot stretching separately at 145 °C with total draw ratio of 9 to obtain DUHMWPE filaments. The mechanical properties of the DUHMWPE filaments were compared. The results thus obtained are tabulated herein **Table 16** below.

Table 16: Mechanical properties of the DUHMWPE filaments obtained from GEL-14 and GEL-15 after hot stretching

Experiment No.	GEL	Batch No.	TS (GPa)	TM (GPa)	% Elongation	Denier(d)
21	GEL-14	TGC-1	4.32	174	2.53	3.23
22	GEL-15	TGC-2	3.59	169	2.98	4.23

TS = Tensile strength, TM = tensile Modulus

From **Table 16**, it is evident that the tensile strength and the tensile modulus of the DUHMWPE filaments obtained from the turpentine based DUHMWPE compact polymer gel, GEL-15, with reduced amounts of additives, fillers, nucleator and antioxidants is comparable to that of DUHMWPE filaments obtained from GEL-14 comprising higher (double) amounts of additives, fillers, nucleator and antioxidants.

Experiment 23 to 27: Effect of quench bath temperature on the mechanical properties of the DUHMWPE filaments obtained after hot stretching

A turpentine based compact polymeric gel, GEL-16, of DUHMWPE having viscosity average molecular weight of 5 million Dalton, was prepared with DUHMWPE polymer concentration of 5% using the same experimental conditions and procedure as given in **Experiment 2A**.

- 5 The amount(s) of different materials used for the preparation of the compact polymeric gel are given herein **Table 17** below.

Table 17: Materials used for the preparation of compact polymeric gel

S.No.	Component	Material(s)	Quantity (%) w/w GEL-16
1.	Polyethylene	DUHMWPE: Viscosity average Molecular weight = 5.0 million Dalton Molecular weight distribution = 8.50	5.50
2.	Fluid Medium	Turpentine	92.00
3.	Antioxidant	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4- hydroxyphenyl)propionate)	0.50
		Second Antioxidant- tris(2,4-di-tert- butylphenyl) phosphite	0.25
4.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol,	0.20
5.	Filler	Montmorillonite modified with a quaternary ammonium salt	0.05
6.	Lubricant	Calcium stearate	1.50

- 10 The compact polymeric gel, GEL-16, was used to obtain DUHMWPE filaments using different quench bath temperature, ranging from 5 °C to 25 °C. DUHMWPE filaments samples quenched at 5 °C, 10 °C, 15 °C, 20 °C, and 25 °C are represented as TQBF-1, TQBF-2, TQBF-3, TQBF-4, and TQBF-5, respectively. The results obtained are tabulated herein **Table 18** below.

- 15 **Table 18: Mechanical properties of the hot stretched DUHMWPE filaments quenched at different quench bath temperature:**

Experiment No.	Filament Sample	Quench bath temperature °C	TS (GPa)	TM (GPa)
23	TQBF-1	5	5.25	190
24	TQBF-2	10	4.75	179
25	TQBF-3	15	4.12	175
26	TQBF-4	20	3.15	163
27	TQBF-5	25	2.65	159

TS = Tensile strength, TM = tensile Modulus

From **Table 18**, it is evident that the tensile strength and the tensile modulus of the DUHMWPE filaments decrease as quenching temperature increases from 5 °C to 25 °C. Thus, the quench bath temperature plays an important role to regulate morphology and microstructure of the DUHMWPE filaments.

Experiment 28: Effect of Turpentine as the fluid medium on the spinnability of DHUMWPE and UHMWPE

The following experiments have been conducted in order to evaluate the efficacy of turpentine as a fluid medium for the gel spun filaments obtained from either DHUMWPE or UHMWPE or in combination of both the polyethylenes.

A set of compact polymeric gels, set-1, was prepared using 3 wt% DUHMWPE having viscosity average molecular weight of 4.5 million Dalton and molecular weight distribution of 7.50. For comparison, a set of gels, set-2, was prepared using UHMWPE of viscosity average molecular weight of 4.5 million Dalton with the concentration of UHMWPE in the range from 0.5 to 3 wt%. Another set of gels, set-3 was prepared using a combination of 50:50 ratio of DHUMWPE having viscosity average molecular weight of 4.5 million Dalton and UHMWPE having molecular weight 4.5 million Dalton at 3 wt % concentration each using turpentine as a fluid medium. The experimental procedure for the preparation of gels was similar to **Experiment 1A**. Remaining composition of the gels were the same as used in **Experiment 1A**. The experimental details are given herein **Table 19** below.

Table 19: Amounts of remaining materials used for the preparation of compact polymeric gel

S. No.	Component	Material	Quantity (%) W/W		
			Set-1	Set-2	Set-3
1.	Fluid Medium	Turpentine	94.5	97.0 - 94.5	94.5
2.	Antioxidant	First Antioxidant- pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate)	0.50	0.50	0.50
		Second Antioxidant- tris(2,4-di-tert-butylphenyl) phosphite	0.25	0.25	0.25
3.	Nucleator	1,3:2,4-di-(4-tolylidene)-d-sorbitol	0.20	0.20	0.20
4.	Filler	Montmorillonite modified with a quaternary ammonium salt (Closite-15A)	0.05	0.05	0.05
5.	Lubricant	Calcium stearate (Cast)	1.50	1.50	1.50

These above mentioned sets of gels were subjected to the process of spinning described in **Experiment 2B**. A comparative study of the spinnability performance of the gels is provided in **Table 20**.

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Table 20: Comparative study of spinnability performance of Turpentine and Decalin as fluid medium for UHMWPE and DUHMWPE polymer

Precursor	Ratio of precursor DUHMWPE : UHMWPE (w/w)	Gel Forming capability		Spinnability	
		Decalin	Turpentine	Decalin	Turpentine
UHMWPE	0:100	Capable	Capable	Spinnable	*Not

					spinnable
DUHMWPE: UHMWPE	50:50	Capable	Capable	Spinnable	*Not spinnable
DUHMWPE	100:0	Capable	Capable	Spinnable	Spinnable

From **Table 20**, it is evident that turpentine is a suitable fluid medium for DUHMWPE but not for UHMWPE even at wide range of viscosity average molecular weight and the concentration of UHMWPE polymer. Processability of DUHMWPE in the presence of

In decalin based compact polymeric gel, the fluid medium flowed from the gels, syneresis occurred during crystallization but syneresis is slow with turpentine based compact polymeric gel, which is an incentive to ease of processing the gel.

TECHNICAL ADVANCEMENTS

The present disclosure described herein above has several technical advantages including, but not limited to, the realization of:

- use of turpentine as a fluid medium to produce compact polymeric gel;
- an economical and environment friendly process for the production of compact polymeric gel;
- production of high and low denier DUHMWPE filaments from the compact polymeric gel; and
- production of hot stretched DUHMWPE filaments with improved tensile properties.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the invention to achieve

one or more of the desired objects or results. While certain embodiments of the inventions have been described, these embodiments have been presented by way of experiments only, and are not intended to limit the scope of the inventions. Variations or modifications to the formulation of this invention, within the scope of the invention, may occur to those skilled in the art upon reviewing the disclosure herein. Such variations or modifications are well within the spirit of this invention.

The numerical values given for various physical parameters, dimensions and quantities are only approximate values and it is envisaged that the values higher than the numerical value assigned to the physical parameters, dimensions and quantities fall within the scope of the invention unless there is a statement in the specification to the contrary.

While considerable emphasis has been placed herein on the specific features of the preferred embodiment, it will be appreciated that many additional features can be added and that many changes can be made in the preferred embodiment without departing from the principles of the disclosure. These and other changes in the preferred embodiment of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

CLAIMS:

1. A compact polymeric gel, comprising:

- i. disentangled ultra-high molecular weight polyethylene (DUHMWPE);
- ii. at least one nucleator;
- 5 iii. at least one filler;
- iv. at least one antioxidant;
- v. turpentine; and
- vi. optionally, at least one lubricant.

2. The compact polymeric gel as claimed in claim 1, wherein

- the amount of said disentangled ultra-high molecular weight polyethylene is in the range of 2.5% to 30% w/w;
- the amount of said at least one nucleator is in the range of 0.08% to 4.0% w/w;
- the amount of said at least one filler is in the range of 0.03% to 1.5% w/w;
- the amount of said antioxidant is in the range of 0.06% to 5.0% w/w;
- 15 • the amount of said turpentine is in the range of 70% to 97% w/w; and
- the amount of said optional lubricant is in the range of 0% to 1.5% w/w.

3. The compact polymeric gel as claimed in claim 1, wherein

- said disentangled ultra-high molecular weight polyethylene is characterized by:

- a viscosity average molecular weight in the range of 2 million Dalton to 20 million Dalton;
- molecular weight distribution in the range of 2 to 20;
- a bulk density in the range of 0.05 g/cm³ to 0.08 g/cm³;
- crystallinity of at least 80%; and
- 25 ○ an elastic modulus of less than 1.0 MPa;

- said nucleator is selected from the group consisting of 1,3:2,4-di(3,4-dimethylbenzylidene)sorbitol, 1,3:2,4-di-(4-tolylidene)-d-sorbitol, 1,3:2,4-di(benzylidene)-d-sorbitol, 1,3:2,4-di-(4-ethylbenzylidene)-d-sorbitol, sodium 2,2'-methylene-bis-(4,6-di-tert-butylphenyl)phosphate, and aluminum hydroxy-bis(2,2'-methylene-bis[4,6-di(tert-butyl)phenyl]phosphate;
- 30 • said filler is montmorillonite modified with a quaternary ammonium salt;
- said antioxidant is selected from the group consisting of tris(2,4-di-tert-butylphenyl)phosphite, pentaerythritol tetrakis(3-(3,5-di-tert-butyl-4-

hydroxyphenyl)propionate), 2,6-di-tert-butyl-4-methylphenol, octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate, aromatic phosphites, amines, 2,6-di-tert-butyl-4-methylphenol, tetrakis[methylene (3,5-di-tert-butyl-4-hydroxyhydrocinnamate)]methane, tris(2,4-di-tert-butylphenyl) phosphite, octadecyl 3,5-di-tert-butyl-4-hydroxyhydrocinnamate, 1,3,5-tris(3,5-di-tert-butyl-4-hydroxybenzyl)-1,3,5-triazine-2,4,6(1H,3H,5H)-trione, and 2,5,7,8-tetramethyl-2-(4',8',12'-trimethyltridecyl)chroman-6-ol; and

- said lubricant is selected from the group consisting of calcium stearate, aluminium stearate, zinc stearate, stearic acid, polyethylene glycol, and polybutene.

4. The compact polymeric gel as claimed in claim 1, wherein said antioxidant is a mixture comprising a first antioxidant and a second antioxidant, and the weight ratio of said first antioxidant and said second antioxidant is in the range of 1:2 to 2:1.

5. A process for preparing a compact polymeric gel, said process comprising:

- mixing
 - disentangled ultra-high molecular weight polyethylene in an amount in the range of 2.5% to 30% w/w;
 - turpentine in an amount in the range of 70% to 97% w/w;
 - at least one nucleator in an amount in the range of 0.08% to 4.0% w/w;
 - at least one filler in an amount in the range of 0.03% to 1.5% w/w;
 - at least one antioxidant in an amount in the range of 0.06% to 5.0% w/w;
 - and
 - optionally, at least one lubricant in an amount in the range of 0% to 1.5% w/w;

under stirring to obtain a suspension;

- heating said suspension under stirring at a temperature in the range of 50 °C to 160 °C for a time period in the range of 2 hours to 25 hours under inert atmosphere to obtain a dispersion;
- cooling said dispersion to a temperature in the range of 25 °C to 40 °C to obtain a bi-phasic mixture comprising a first layer comprising free turpentine; and a second layer comprising compact polymeric gel containing turpentine; and

- separating said second layer from said bi-phasic mixture to obtain said compact polymeric gel containing turpentine.

6. The process as claimed in claim 5, wherein at least 40% turpentine is recovered.

7. Disentangled ultra-high molecular weight polyethylene filaments prepared from said compact polymeric gel as claimed in claim 1, characterized by;

- a tensile strength in the range of 2.5 GPa to 15 GPa;
- a tensile modulus in the range of 150 GPa to 250 GPa; and
- denier in the range of 0.5 d to 5 d;

wherein, preferably, said filaments are twisted to obtain a gel spun yarn, characterized by;

- denier in the range of 2.45 d to 18 d; and
- tensile strength of said gel spun yarn being at least 3 times the tensile strength of DUHMWPE filaments at a temperature in the range of 140 °C to 150 °C.

8. A process for preparing disentangled ultra-high molecular weight polyethylene filaments from said compact polymeric gel as claimed in claim 1, said process comprising:

- spinning said compact polymeric gel of claims 1 to 4 at a temperature ranging from 120 °C to 200 °C, at a pressure ranging from 2 kg/cm² to 3 kg/cm² to obtain a tow of gel spun filaments, wherein said tow of gel spun filaments is one of a monofilament tow and a multifilament tow;
- quenching said tow of gel spun filaments in a quenching medium at a temperature in the range of 5 °C to 30 °C to obtain a quenched tow of gel spun filaments;
- passing said quenched tow of gel spun filaments through at least one washing medium to remove excess turpentine and to obtain a wet tow of gel spun filaments;
- drying said wet tow of gel spun filaments at a temperature in the range of 80 °C to 125 °C to obtain a dried tow of gel spun filaments; and
- hot stretching said dried tow of gel spun filaments at a temperature in the range of 90 °C to 150 °C and at a draw ratio in the range of 5 to 100 to obtain disentangled ultra-high molecular weight polyethylene (DUHMWPE) filaments.

9. The process as claimed in claim 8, wherein the step of spinning said compact polymeric gel involves at least one method selected from the group consisting of passing through an extruder, and a spinneret having hole size in the range of 0.3 mm to 1.0 mm.

5 10. The process as claimed in claim 8, wherein:

- said quenching medium is at least one selected from the group consisting of water, and cooling gas; and
- said washing medium is at least one selected from the group consisting of acetone, water, hexane, ethanol, ether, cyclohexanone, 2-methylpentanone, dichloromethane, and dioxane.

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INTERNATIONAL SEARCH REPORT

International application No.
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A. CLASSIFICATION OF SUBJECT MATTER

C08J3/075, D01F6/04, D01D13/00 Version=2018.01

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C08J, D01D,

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

TotalPatent One, IPO Internal Database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 2015125064 A1 (RELIANCE INDUSTRIES LIMITED) Published on 27 August 2015 (abstract, claims 1-19, examples 1-10)	1-10
Y	CN 106120045 A (EAST CHINA SEA FISHERIES RESEARCH INSTITUTE, CHINESE ACADEMY OF FISHERY SCIENCES) published on 16 November 2016 (abstract, claim 1, para [0005], [0008])	1-10



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents:

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“E” earlier application or patent but published on or after the international filing date

“L” document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

“O” document referring to an oral disclosure, use, exhibition or other means

“P” document published prior to the international filing date but later than the priority date claimed

“T” later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

“X” document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

“Y” document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

“&” document member of the same patent family

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INTERNATIONAL SEARCH REPORT
Information on patent family members

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Citation	Pub.Date	Family	Pub.Date
WO 2015125064 A1	27-08-2015	CN 106062066 A	26-10-2016
		EP 3107961 A1	28-12-2016
		JP 2017507216 A	16-03-2017
		US 2017009382 A1	12-01-2017