(19) Europäisches Patentamt
European Patent Office
Office européen des brevets

(11) EP 4 566 464 A1

(12) **EUROPEAN PATENT APPLICATION**

(43) Date of publication:
11.06.2025 Bulletin 2025/24

(21) Application number: **24218087.5**

(22) Date of filing: **06.12.2024**

(51) International Patent Classification (IPC):
A24D 3/10 *(2006.01)* ***D01F 2/00*** *(2006.01)*

(52) Cooperative Patent Classification (CPC):
A24D 3/10; A24D 3/063; A24D 3/068; A24D 3/16; D01F 2/00

(84) Designated Contracting States:
AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HR HU IE IS IT LI LT LU LV MC ME MK MT NL NO PL PT RO RS SE SI SK SM TR
Designated Extension States:
BA
Designated Validation States:
GE KH MA MD TN

(30) Priority: **08.12.2023 KR 20230178048**
04.12.2024 KR 20240178343

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(54) **LYOCELL MATERIAL, SMOKING ARTICLES, AND MANUFACTURING METHOD THEREOF**

(57) The present disclosure relates to a lyocell material, a smoking article including the same, and a method for preparing the lyocell material, wherein the lyocell material may be prepared from a dope solution including cellulose pulp and N-methylmorpholine-N-oxide (NMMO), wherein the cellulose pulp may have a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight.

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Description

TECHNICAL FIELD

[0001] The present disclosure relates to: a lyocell material having favorable spinning processability, appearance, and cigarette-filter cuttability, as well as a monofilament strength suitable for application to a filter for smoking articles; a smoking article including the lyocell material; and a method of preparing the lyocell material.

BACKGROUND ART

[0002] Until now, cellulose acetate fiber has primarily been used as a cigarette filter material. Although cellulose acetate is known to be a biodegradable substance, filters for smoking articles made of cellulose acetate maintain their original shape for about one to two years after burial in soil, and take considerable time to fully biodegrade. Cellulose acetate, being a synthetic plastic, decomposes into microplastics through physical and photochemical reactions when exposed to the external environment. Microplastics are known as harmful substances that disrupt ecosystems.

[0003] Therefore, research is being conducted in various fields on chemically unmodified biodegradable materials for smoking articles to replace cellulose acetate. Recently, filters made of lyocell material have been studied for smoking articles; however, there remains a need for a lyocell material suitable for continuous manufacturing processes of filters for smoking articles.

DESCRIPTION OF EMBODIMENTS

TECHNICAL PROBLEM

[0004] The present disclosure aims to provide a lyocell material having favorable spinnability, appearance, and cigarette-filter cuttability, and a suitable monofilament strength for application to filters for smoking articles; a smoking article including the lyocell material; and a method of preparing the lyocell material.

SOLUTION TO PROBLEM

[0005] According to an aspect, provided is a lyocell material prepared from a dope solution including cellulose pulp and N-methylmorpholine-N-oxide (NMMO),

wherein the cellulose pulp has a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight.

[0006] The lyocell material may include at least one lyocell monofilament.

[0007] The lyocell multifilament may include at least one lyocell monofilament.

[0008] According to another aspect, provided is a smoking article including the lyocell material.

[0009] According to another aspect, provided is a method of preparing a lyocell material, the method including: spinning, through a spinneret, a dope solution comprising cellulose pulp and N-methylmorpholine-N-oxide (NMMO);

> coagulating the spun dope solution to obtain a lyocell multifilament;
> washing the lyocell multifilament and treating the lyocell multifilament with emulsion; and
> crimping the emulsion-treated multifilament to obtain a crimped tow,
> wherein the cellulose pulp has a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight.

ADVANTAGEOUS EFFECTS OF DISCLOSURE

[0010] According to one aspect, by using, as a raw material, a pulp having a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight, the lyocell material not only satisfies a monofilament strength range suitable for application as a filter for smoking articles, but also enables the production of a cigarette filter having favorable cuttability.

MODE OF DISCLOSURE

[0011] The present inventive concept, which will be more fully described hereinafter, may have various variations and various embodiments, and specific embodiments will be illustrated in the accompanied drawings and described in greater detail. However, the present inventive concept should not be construed as being limited to specific embodiments set forth herein. Rather, these embodiments are to be understood as encompassing all variations, equivalents, or alternatives included in the scope of the present inventive concept.

[0012] The terminology used hereinbelow is used for the purpose of describing particular embodiments only, and is not intended to limit the present inventive concept.

[0013] As used herein, the singular forms are intended to include the plural forms as well, unless the context clearly indicates otherwise. As used herein, the terms "comprises" and/or "comprising," or "includes" and/or "including," or "has," unless otherwise indicated, indicate that certain features, numbers, steps, operations, components, parts, constituents, materials, or any combination thereof described in the specification are present, but do not preclude the presence or addition of one or more other features, numbers, steps, operations, components, parts, constituents, materials, or combinations thereof.

[0014] Unless otherwise defined, all terms (including technical terms and scientific terms) used herein have the same meaning as commonly understood by one of ordinary skill in the art to which the present disclosure belongs. It will be further understood that terms, such as those defined in commonly used dictionaries, should be interpreted as having a meaning that is consistent with their meaning in the present specification and the relevant art and should not be interpreted in an idealized or overly formal sense unless expressly so defined herein.

[0015] In the present specification, "smoking article" may refer to an article capable of generating an aerosol, such as a cigarette (cigar) or a cigar. In this regard, the smoking article may include an aerosol-generating material or an aerosol-forming substrate. Also, the smoking article may include a solid material based on a tobacco raw material, such as reconstituted tobacco, cut tobacco, or cast-leaf tobacco. In addition, the smoking article may include a volatile compound.

[0016] Unless otherwise defined herein, where the characteristics of a lyocell material, a filter for smoking articles, or relevant components or configurations are affected by temperature, the temperature at which such characteristics are identified or measured may be room temperature. Room temperature, in the absence of intentional cooling or heating, may be, for example, from 10 °C to 35 °C, specifically from 15 °C to 35 °C, from 20 °C to 30 °C, or about 25 °C.

[0017] In the present specification, the term "different" may indicate that the entities differ in a qualitative sense. For example, "A and B are different" may mean that although the amounts of A and B are the same, A and B are qualitatively distinguishable; or that A and B differ quantitatively and are also qualitatively distinguishable. The term "qualitative" may refer to non-quantitative properties. For example, differences in color, shape, texture, structure, or composition may fall under "qualitative" differences.

[0018] In the present specification, the term "crimp" may refer to a wavelike, curled, or undulating configuration imparted inherently or through mechanical, thermal, and/or chemical processing to a material such as a fiber, filament, multifilament, or yarn. A crimp may be characterized by periodic deviations from a linear axis along the length of the material, fiber, filament, multifilament, and/or yarn. One crimp in the material, fiber, filament, multifilament, and/or yarn may be defined as one repeating unit of the periodic deviation. The presence of crimp affects properties such as elasticity, bulk, resilience, and texture of the material and textiles made from the material.

[0019] In the present specification, the term "degree of polymerization" (DP) may refer to the number of monomer units and/or repeating units in a macromolecule, polymer, or oligomer molecule. The DP may be expressed as M_n/M_0, where M_n is the number-average molecular weight of the macromolecule, polymer, or oligomer, and M_0 is the molecular weight of one monomer or repeating unit.

[0020] Hereinafter, a more detailed description will be provided regarding the lyocell material according to exemplary embodiments, a smoking article including the lyocell material, and a method of manufacturing the lyocell material.

[Lyocell Material]

[0021] In the present disclosure, the term "lyocell material" refers to a monofilament material or an aggregate of monofilaments (multifilament) obtained by spinning, coagulating, and drying a dope solution in which cellulose pulp is dissolved in an N-methylmorpholine-N-oxide (NMMO) solution. It will be understood in the art that the term "lyocell material" as used herein is distinct from a "cellulose acetate material".

[0022] According to one aspect, a lyocell material may be prepared from a dope solution including cellulose pulp and N-methylmorpholine-N-oxide (NMMO), wherein the cellulose pulp has a CED viscosity of 4.0 to 11.0, and based on the total weight of the cellulose pulp, contains less than 5 wt% hemicellulose, from 1 ppm to less than 30 ppm of Fe by weight, and from 1 ppm to less than 100 ppm of Si by weight.

[0023] The CED viscosity was measured according to TAPPI Standard T230 om-94.

[0024] The hemicellulose content was measured according to KS M 7044:2016.

[0025] Inorganic components, such as Fe and Si, in the pulp were measured using ICP-OES (PerkinElmer Avio-550).

[0026] The cellulose pulp may have an alpha-cellulose content of 85 wt% to 97 wt% based on 100 wt% of the total weight of all cellulose in the pulp and/or relative to the total weight of the cellulose pulp.

[0027] The cellulose pulp may have a hemicellulose content of 1 wt% or more and less than 5 wt% based on 100 wt% of the total weight of all cellulose in the pulp and/or relative to the total weight of the cellulose pulp.

[0028] By employing cellulose pulp having a specific 0.5 % CED viscosity, hemicellulose content, and inorganic content as the raw material, the final lyocell material may exhibit improved processability and workability.

[0029] By adjusting the CED viscosity of the cellulose pulp used as the raw material for the lyocell material to between 4.0 and 11.0, spinnability may be improved. If the CED viscosity is below 4.0, breakage of filament may occur repeatedly during spinning lyocell dope through spinneret, leading to the deterioration of the spinning process and making continuous filament production difficult. If the viscosity exceeds 11.0, an excessively high pressure may be required to extrude the spinning dope, or the spinneret nozzle may become clogged.

[0030] By adjusting the hemicellulose content of the raw-material cellulose pulp for the lyocell material to less than 5 wt%, stable and continuous production of lyocell filaments may become possible. If the hemicellulose content is 5 wt% or more, not only does contamination of the spinneret by hemicellulose released from the NMMO solvent become problematic, but the decrease in filament strength due to the increased hemicellulose content in the lyocell material may render it difficult to apply as a filter for smoking articles. Furthermore, from the standpoint of NMMO recovery, removing the hemicellulose released from the NMMO solvent may be essential, which increases costs and burdens on the NMMO recovery/regeneration system.

[0031] By controlling the Fe content in the cellulose pulp for the lyocell material to less than 30 ppm by weight, the pulp's decomposition reaction by NMMO may be accelerated, thereby preventing discoloration of the NMMO-and thus the filaments-and preventing loss of filament tenacity. If the Fe content in the cellulose pulp is 30 ppm or more by weight, filament strength is reduced, thereby degrading the cuttability needed for cigarette filter manufacturing.

[0032] By controlling the Si content of the cellulose pulp, used as the raw material for the lyocell material, to less than 100 ppm by weight, it is possible to prevent discoloration of the filament and a decrease in filament tenacity caused by the discoloration of NMMO. If the Si content in the cellulose pulp is 100 ppm by weight or higher, filament tenacity is reduced, resulting in poor cuttability when configuring a cigarette filter and making it difficult to apply in the filter manufacturing process.

[0033] Conventionally, lyocell staple fibers have typically been produced using pulp with a hemicellulose content of 5 wt% or more, so filament strength was not especially high. However, the lyocell material according to one embodiment of the present disclosure may achieve monofilament strength sufficient for use in cigarette filters by employing a pulp with less than 5 wt% hemicellulose. On the other hand, merely lowering the hemicellulose content alone was not enough to achieve the filament cuttability required for cigarette filters. The inventors of the present invention discovered that, by controlling the Fe and Si contents present in trace amounts as inorganic components in the pulp, the decrease in filament tenacity may be suppressed, thereby improving cuttability during the cigarette filter manufacturing process. Building upon this insight, the inventors completed the lyocell material of the present disclosure.

[0034] Moreover, because cigarette filters are typically preferred to be white for aesthetic reasons from the smoker's perspective, conventional lyocell manufacturing processes have been designed to pass filaments through a whitening stage. However, the inventors confirmed that controlling the Fe and Si contents in the pulp suppresses discoloration caused by NMMO, thus making it possible to produce the appearance (i.e., color) required of cigarette filters. Building upon this insight, the inventors completed the lyocell material of the present disclosure.

[0035] Without being bound by theory, by using a pulp having specific CED viscosity, hemicellulose content, and Fe and Si contents as the raw-material cellulose pulp, the lyocell material may possess sufficient appearance and monofilament strength for application to cigarette filters-while preventing a decline in filament tenacity-and may also have the cuttability required in the cigarette filter manufacturing process.

[0036] According to one embodiment, the cellulose pulp may have a hemicellulose content of 4 wt% or less, based on the total weight of the cellulose pulp. For example, the cellulose pulp may have a hemicellulose content of 3.9 wt% or less, 3.8 wt% or less, or 3.7 wt% or less. While the cellulose pulp may be rendered hemicellulose-free, in practice, refining the cellulose pulp to be hemicellulose-free would involve complex steps and high costs, making it generally undesirable.

[0037] According to one embodiment, the cellulose pulp may include various inorganic substances introduced during extraction. For example, the cellulose pulp may include inorganic substances such as iron (Fe), copper (Cu), and silicon (Si). Conventional lyocell research has explored the strength of lyocell multifilaments in relation to the hemicellulose content of the cellulose pulp, but no research has been conducted on the control of inorganic content.

[0038] According to one embodiment, the inorganic content of the cellulose pulp may be controlled by contacting the pulp with a diluted acid solution, for example, a sulfurous acid, hydrochloric acid, or sulfuric acid solution, to form and remove inorganic salts; however, the present disclosure is not necessarily limited thereto.

[0039] According to one embodiment, the cellulose pulp may contain 1 ppm to 20 ppm of Fe by weight relative to the total weight of the cellulose pulp. For example, the cellulose pulp may contain 1 ppm to 19 ppm, 1 ppm to 18 ppm, 1 ppm to 17 ppm, 1 ppm to 16 ppm, 1 ppm to 15 ppm, 1 ppm to 14 ppm, 1 ppm to 13 ppm, 1 ppm to 12 ppm, 1 ppm to 11 ppm, 1 ppm to 10 ppm, 1 ppm to 9 ppm, 1 ppm to 8 ppm, 1 ppm to 7 ppm, 1 ppm to 6 ppm, 1 ppm to 5 ppm, 1 ppm to 4 ppm, 1 ppm to 3 ppm, to 1 ppm to 2 ppm of Fe by weight.

[0040] According to one embodiment, the cellulose pulp may contain 1 ppm to 100 ppm of Si by weight relative to the total weight of the cellulose pulp. For example, the cellulose pulp may contain 1 ppm to 90 ppm, 1 ppm to 80 ppm, 1 ppm to 70 ppm, 1 ppm to 60 ppm, 1 ppm to 50 ppm, 1 ppm to 40 ppm, 1 ppm to 30 ppm, 1 ppm to 20 ppm, or 1 ppm to 10 ppm of Si by weight, but without being limited to these ranges, any lower and upper limits among the aforementioned values may be combined to form a suitable range.

[0041] According to one embodiment, the cellulose pulp may contain, relative to the total weight of the cellulose pulp, 1 ppm to 10 ppm of Fe by weight and 1 ppm to 90 ppm of Si by weight. For example, the cellulose pulp may contain less than 5 ppm of Fe by weight and 5 ppm to 80 ppm of Si by weight.

[0042] According to one embodiment, the cellulose pulp may have a degree of polymerization of 600 to 1,700. In some embodiments, the degree of polymerization may refer to the number of repeating units and/or monomer units in cellulose and/or hemicellulose contained in the cellulose pulp.

[0043] According to one embodiment, the lyocell material may include one or more lyocell monofilaments, and each of these monofilaments may have a strength of about 0.265 N/tex to about 0.706 N/tex (3 g/d to 8 g/d). For example, the monofilaments may have a strength of about 0.274 N/tex to about 0.698 N/tex (3.1 g/d to 7.9 g/d), about 0.283 N/tex to about 0.689 N/tex (3.2 g/d to 7.8 g/d), about 0.291 N/tex to about 0.680 N/tex (3.3 g/d to 7.7 g/d), about 0.300 N/tex to about 0.671 N/tex (3.4 g/d to 7.6 g/d), or about 0.309 N/tex to about 0.662 N/tex (3.5 g/d to 7.5 g/d). In the present specification, the term "strength" refers to textile strength and/or tenacity.

[0044] Strength may be measured by the following method: after pre-drying a multifilament specimen at 110 °C for 2 hours, the specimen is allowed to stand under the standard conditions described in KS K 0901 for at least 24 hours to reach a moisture equilibrium state. A monofilament specimen is then separated from the multifilament. The separated monofilament specimen is tested for tensile strength at a drawing speed of 60 mm/min using a low-speed elongation-type tensile tester (Instron).

[0045] The lyocell material, when having a monofilament strength within the above-specified range, may be readily processable as a filter for cigarettes. If the monofilament strength is below about 0.265 N/tex (3 g/d), the cigarette filter formed therefrom may easily collapse when bitten by the smoker, making it difficult to function as a filter. If the monofilament strength exceeds about 0.706 N/tex (8 g/d), the high tensile strength in the cigarette filter manufacturing process hinders filter cutting, resulting in poor cut surfaces.

[0046] According to one embodiment, the lyocell material may be imparted with crimp. As will be described in greater detail below in the manufacturing process of the lyocell material, the lyocell material may acquire crimp by undergoing a crimping process, and thus possess the suction resistance and filtration capability required when used as a cigarette filter material. Moreover, the degree of tow opening during the filter manufacturing process is enhanced, facilitating continuous filter production.

[0047] According to one embodiment, the lyocell material may have about 25.4 crimps to about 127 crimps per centimeter (about 10 crimps to about 50 crimps per inch).

[0048] According to one embodiment, the lyocell material may be in the form of lyocell tow. Lyocell tow refers to a banded aggregate of lyocell multifilaments, which is used as a feed material in the manufacturing process of cigarette filters.

[0049] According to one embodiment, the lyocell tow may have a non-circular cross-section. "Non-circular cross-section" may refer to any cross-sectional shape departing from a standard circular shape. For example, the cross-sectional shape may be Y-shaped, rectangular, star-shaped, leaf-shaped, or hexagonal, or have other polygonal cross-sections. From the standpoint of application to cigarette filters, the lyocell tow may preferably have a Y-shaped cross-section.

[0050] According to one embodiment, the lyocell tow may have a total fineness of about 1.67 g/m to about 6.11 g/m (15,000 to 55,000 denier).

[0051] According to one embodiment, the lyocell tow may be used for manufacturing filters for smoking articles.

[METHOD OF PREPARING LYOCELL MATERIAL]

[0052] According to one aspect, a method of preparing a lyocell material includes: spinning, through a spinneret, a dope solution including cellulose pulp and N-methylmorpholine-N-oxide (NMMO); coagulating the spun dope solution to obtain a lyocell multifilament; washing the lyocell multifilament and treating the lyocell multifilament with emulsion; and crimping the emulsion-treated multifilament to obtain a crimped tow, wherein the cellulose pulp has a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight. The CED viscosity was measured according to TAPPI Standard T230 om-94.

[0053] The method of preparing a lyocell material according to one embodiment will be described in greater detail below.

<(a) Lyocell Dope Spinning Process>

[0054] A lyocell spinning dope may be prepared by dissolving in an N-methylmorpholine-N-oxide (NMMO) solution a cellulose pulp having the above-described CED viscosity of 4.0 to 11.0, and based on the total weight of the cellulose pulp, containing less than 5 wt% hemicellulose, from at least 1 ppm and less than 30 ppm of Fe by weight, and from at least 1 ppm and less than 100 ppm of Si by weight.

[0055] According to one embodiment, the content of the cellulose pulp in the spinning dope may be 5 wt% to 15 wt% based on 100 wt% of the total weight of the dope. If the content of the cellulose pulp is excessively low, it may be difficult to realize the characteristics of the lyocell fiber; on the other hand, if the content exceeds the aforementioned range, it may be difficult to dissolve the pulp in the solvent. In view of the foregoing, the content of the cellulose pulp in the spinning dope may be 6 wt% or more, 7 wt% or more, 8 wt% or more, 9 wt% or more, or 10 wt% or more, and may have its upper limit of, for example, 14 wt% or less, 13 wt% or less, 12 wt% or less, 11 wt% or less, 10 wt% or less, or 9 wt% or less.

[0056] According to one embodiment, the spinning dope may include an aqueous N-methylmorpholine-N-oxide (NMMO) solution. Taking into account the extent of cellulose dissolution, process temperature, and the like, for example, the aqueous solution may include 80 parts by weight to 95 parts by weight of N-methylmorpholine-N-oxide, and 5 parts by weight to 20 parts by weight of water.

[0057] According to one embodiment, the cellulose or cellulose pulp may have an alpha-cellulose content of 85 wt% to 97 wt% relative to 100 wt% of the total cellulose.

[0058] According to one embodiment, the cellulose or cellulose pulp may have a hemicellulose content of 1 wt% and less than 5 wt% relative to 100 wt% of the total cellulose. By adjusting the hemicellulose content to the aforementioned range, stable physical properties (for example, mechanical properties such as tensile strength or elongation) and processability of the lyocell material may be more readily achieved.

[0059] In the spinning process, the shape of the spinneret used for discharging the spinning dope is not particularly limited. For example, a donut-shaped spinneret may be used.

[0060] The nozzle temperature of the spinneret may be appropriately selected by those skilled in the art, taking into account the concentration of the spinning dope and the desired properties of the filaments. For example, spinning may be carried out at a temperature of 100 °C to 120 °C, or 100 °C to 110 °C.

[0061] The spinning dope discharged through the spinneret may undergo a coagulation process described below.

<(b) Process of Coagulation and Obtaining Multifilament >

[0062] The extruded dope discharged from the spinneret may be coagulated, yielding lyocell multifilaments.

[0063] The coagulation may employ a method that brings the extruded dope into contact with air and/or a coagulation liquid.

[0064] According to one embodiment, the coagulation may include: a primary coagulation process of supplying cooled air to the extruded dope; and a secondary coagulation process of immersing the primarily coagulated extruded dope into a coagulation liquid for coagulation.

[0065] The primary coagulation process may be carried out in a space (air gap region) between the spinneret and a coagulation bath. In the air gap region, cooled air may be supplied from the inside of the spinneret outward, but is not limited thereto; cooled air may also be supplied from the outside inward, or simultaneously in both directions. In addition, the primary coagulation process may be performed by appropriately referring to known air-quenching methods, as recognized by those skilled in the art.

[0066] According to one embodiment, the upper temperature limit of the cooled air used in the primary coagulation process may be, for example, 15 °C or below. For example, the cooled air may be air having a temperature of 14 °C or less, 13 °C or less, 12 °C or less, 11 °C or less, or 10 °C or less. If the temperature exceeds this range, the extruded dope may not sufficiently coagulate via the air, and spinning-related processability deteriorates.

[0067] The lower temperature limit of the cooled air may be determined considering spinning processability and/or cross-sectional uniformity of the filaments. For example, if the temperature of the cooled air is below 4 °C, the spinneret surface cools, causing irregularities in the filament surface and deteriorating spinning processability. In light of the foregoing, the cooled air may be 5 °C or more, 6 °C or more, 7 °C or more, 8 °C or more, or 9 °C or more.

[0068] The degree to which the cooled air is supplied may be adjusted, in consideration of sufficient coagulation, spinning processability, and the effects on filament properties. For example, the cooled air may be supplied at an airflow rate of 70 Nm^3/h to 400 Nm^3/h to the extruded spinning dope. More specifically, the airflow rate may be 100 Nm^3/h or more, 150 Nm^3/h or more, 200 Nm^3/h or more, or 250 Nm^3/h or more, and the upper limit of the airflow rate may be, for example, 350 Nm^3/h or less, 300 Nm^3/h or less, 250 Nm^3/h or less, 200 Nm^3/h or less, or 150 Nm^3/h or less.

[0069] After such primary coagulation, the cooled extruded dope may be supplied to a coagulation tank or bath containing a coagulation liquid (secondary coagulation). For suitable coagulation, the temperature of the coagulation liquid may be, for example, 30 °C or less, or 25 °C or less. In addition, the temperature of the coagulation liquid may be at least 10 °C, at least 15 °C, or at least 20 °C. Maintaining this temperature allows for an appropriate coagulation rate.

[0070] The type of coagulation liquid for the secondary coagulation as described above is not particularly limited. For example, the coagulation liquid may include one or more of water and N-methylmorpholine-N-oxide (NMMO).

[0071] According to one embodiment, when the coagulation liquid contains water and NMMO, the coagulation liquid may include 60 wt% to 90 wt% of water and 10 wt% to 40 wt% of NMMO, based on 100 wt% of the total weight of the coagulation liquid. Alternatively, the coagulation liquid may include 70 wt% to 80 wt% of water and 20 wt% to 30 wt% of NMMO. The concentration of such a coagulation liquid may be controlled using a concentration control device so as to remain stable during the manufacturing process.

<(c) Washing Process>

[0072] A washing process may be carried out on the multifilaments obtained after the spinning dope is coagulated. By the washing process, the residual NMMO and/or other impurities in the filaments may be controlled to a desired amount.

[0073] The method of performing the washing is not particularly limited. For example, the washing may be carried out by introducing each coagulated lyocell multifilament into a washing bath using a take-up roller. Alternatively, the washing may be performed by spraying a washing liquid onto the multifilament as they are conveyed to the next process by a take-up roller.

[0074] The components of the washing liquid are not particularly limited. For example, the washing liquid may include water, and may further include other known additives.

[0075] Additionally, in consideration of reuse after washing, the washing liquid may be regulated to a temperature of up to 100 °C.

<(d) Emulsion Treatment Process>

[0076] After washing the multifilaments, an emulsion treatment process may be performed to apply an emulsion to their surfaces. By applying the emulsion to the multifilaments, friction on the filaments in the subsequent crimping process may be reduced, ensuring that crimp formation proceeds smoothly. The emulsion treatment process may be performed once, or two or more times as needed. If the emulsion treatment is to be carried out two or more times, the process may be referred to as a primary emulsion treatment, a secondary emulsion treatment, and so on, in chronological order.

[0077] Without particular limitation, the emulsion treatment may be carried out by immersing each lyocell multifilament in a bath filled with the emulsion so that each filament is fully submerged. Alternatively, the emulsion treatment may be carried out by spraying an emulsion solution onto the multifilament as they are conveyed to the next stage by a take-up roller.

[0078] After the emulsion treatment as described above, in order to make the amount of emulsion applied onto each lyocell multifilament uniform, a process may further be carried out in which a roll (or the like), positioned before and/or after the emulsion treatment, squeezes the emulsion off the surface of the lyocell multifilaments.

[0079] After such an emulsion treatment, drying may be performed before a crimping process.

<(e) Crimp-Imparting Process>

[0080] A crimp-imparting process may be a process of obtaining crimped multifilaments, preferably crimped tow, by applying pressure to the emulsion-treated multifilaments through steam and/or a pressing roller. The crimp-imparting process may be referred to as a crimping step.

[0081] Through crimping, each lyocell multifilament may be imparted with waves, and the fiber may obtain a bulky characteristic. Crimping may be performed using known crimping devices, including a stuffer box and/or a steam box.

[0082] According to one embodiment, the crimp-imparting process may be performed by first supplying steam to each lyocell multifilament to preheat and swell the lyocell multifilaments, followed by pressing each lyocell multifilament with a pressing roller to form crimps to the lyocell multifilaments. In this case, a steam box may be used for steam supply, and such a steam box may be positioned upstream of the crimping device. In the crimp-imparting process, the supply of steam may be omitted if necessary.

[0083] According to one embodiment, the crimp-imparting process may be performed by simultaneously applying pressure to the lyocell multifilaments with a pressing roller and supplying steam.

[0084] According to one embodiment, the crimp-imparting process may be performed by pressing each lyocell multifilament with a pressing roller to form crimps in each lyocell multifilament. In addition, steam supply may not be performed before pressing, steam supply may not be performed simultaneously with pressing, and steam supply may not be performed both before and simultaneously with pressing.

[0085] According to one embodiment, in the crimp-imparting process, a doctor blade that applies a predetermined pressure to each lyocell multifilament may be employed. The doctor blade helps control the dwell time of the filaments loaded into the crimper's stuffer box, thus contributing to adjusting the number of crimps. Such a doctor blade may be positioned, for example, in the travel path of each lyocell multifilament discharged from the pressing point after being pressed by the aforementioned roller.

[0086] According to one embodiment, the crimp-imparting process may be performed at a temperature ranging from 120 °C to 250 °C. If the temperature is excessively low, the shape-stability effect of the crimp may become poor; if the temperature is excessively high, the concentration of oil components in the stuffer box may increase, making crimp formation difficult.

[0087] The lyocell material according to the present disclosure may be obtained by the above-described method of preparing a lyocell material.

[0088] The lyocell material according to the present disclosure may be a material obtainable by the above-described method of preparing a lyocell material.

[SMOKING ARTICLE]

[0089] The above lyocell material may be included in a smoking article. A smoking article may be an aerosol-generating article. The aerosol-generating article may include an aerosol-generating material or an aerosol-forming substrate.

[0090] For example, the lyocell material may be included in a combustible cigarette or a heat-not-burn cigarette, and the heat-not-burn cigarette may be used with an aerosol-generating device.

[0091] For example, when used as a heat-not-burn smoking article, the smoking article may be inserted separately into the aerosol-generating device. Here, the aerosol-generating device may include a receiving recess to accommodate the aerosol-generating article, and may further include a heater for heating the aerosol-generating article to produce an aerosol, a controller for overall operation of the aerosol-generating device, a battery for providing power for the aerosol-generating device's operation, and a sensor to detect that the aerosol-generating article has been inserted into the aerosol-generating device.

[0092] The smoking article may include a tobacco medium part, a filter for a smoking article, and a wrapper, wherein the filter for a smoking article may be located at one end of the tobacco medium part, for example, at the rear end or the front end. The tobacco medium part and the filter for a smoking article may each include a single segment, or may independently include a plurality of segments.

[0093] The tobacco medium part may include a tobacco material, and the tobacco material may include nicotine. Additionally, the tobacco medium part may further include an excipient.

[0094] The excipient may include a binder, a filler, and other additives. For example, a tobacco medium contained in the tobacco medium part may be produced in the form of granules, including a tobacco material, a excipient, and the like.

[0095] For example, to maintain a consistent shape, strength, and mass of the tobacco medium part, a packing material may additionally be included. For example, the lyocell material may be included in the tobacco medium part. In addition, the lyocell material may be used as a filler.

[0096] The wrapper may be subdivided into a cigarette paper wrapping the tobacco medium part, a filter wrapping paper enclosing the filter part, a tipping wrapper joining the tobacco medium part and the filter, and the like.

[FILTER FOR SMOKING ARTICLE]

[0097] The lyocell material may be used in a filter for a smoking article. The lyocell material may be lyocell tow. In one example, the lyocell tow may be imparted with crimps.

[0098] For example, the present application relates to a filter for a smoking article. The filter for a smoking article may include a lyocell material, and the lyocell material may be the same as described above. In addition, the filter for a smoking article may include lyocell tow, and the lyocell tow may be the same as described above.

[0099] In one embodiment, the filter for a smoking article may further include wrapping paper (which may be referred to as winding paper, filter paper, or filter wrapping paper). For example, the wrapping paper may enclose the aforementioned lyocell tow and may be a porous or nonporous paper that maintains the shape of the filter (e.g., cylindrical).

[0100] In one embodiment, the filter for a smoking article may have a predetermined shape and size.

[0101] In one embodiment, the filter may have a rod shape. For example, the filter for a smoking article may have a shape similar to a cylinder. Although a filter for a smoking article may be manufactured to have shapes other than cylindrical, a cylindrical shape may be advantageous for including the maximum volume of lyocell material within the filter space.

[0102] In one embodiment, the filter may have a length of 10 mm to 50 mm, for example. For example, the length of the filter may have its lower limit of 15 mm or more, 20 mm or more, 25 mm or more, 30 mm or more, 35 mm or more, 40 mm or more, or 45 mm or more, and may have its upper limit of 45 mm or less, 40 mm or less, 35 mm or less, 30 mm or less, 25 mm or less, 20 mm or less, or 15 mm or less.

[0103] In one embodiment, the filter having the above length may have a circular cross section, and the circumference of the circular cross section may be 10 mm to 40 mm. For example, the circumference of the filter may have its lower limit of 15 mm or more, 20 mm or more, 25 mm or more, 30 mm or more, or 35 mm or more, and may have its upper limit of 35 mm or less, 30 mm or less, 25 mm or less, 20 mm or less, or 15 mm or less.

[0104] In one embodiment, the filter for a smoking article may include lyocell tow and filter wrapping paper. Descriptions of the lyocell tow and the filter wrapping paper are the same as set forth above and are therefore omitted.

[0105] The wrapping paper may enclose the aforementioned lyocell tow and may be porous or nonporous paper that maintains the filter shape (e.g., cylindrical).

[0106] In one embodiment, when porous wrapping paper is used, the wrapping paper may have a porosity of 10 CU to 50,000 CU (Coresta Units). A Coresta Unit may be defined as the volumetric flow rate ($cm^3 \cdot min^{-1}$) of air passing through a 1 cm^2 substrate sample (i.e., the porous wrapping paper) under a 1 kPa pressure difference.

[0107] In one embodiment, the basis weight of the wrapping paper may be 15 g/cm^2 to 60 g/cm^2.

[0108] In one embodiment, the weight of a rod-shaped filter may be 50 mg or more.

[0109] Descriptions of other filters for smoking articles or materials contained therein are the same as set forth above and are therefore omitted.

[0110] The present disclosure is described in greater detail with reference to the following Preparation Examples, Examples, and Comparative Examples. However, the examples are provided only to illustrate the present disclosure, and the scope of the present disclosure is not limited thereto.

Preparation Example

[0111] A spinning dope having a concentration of 11 wt% was prepared by mixing a cellulose pulp, whose viscosity measured by the CED (cupriethylene diamine) solution method, hemicellulose content, Fe content, and Si content as described in Table 1 below, with an NMMO/H_2O solvent containing 0.01 wt% propyl gallate. Next, while maintaining a spinning temperature of 110 °C at the spinning nozzle, the discharge amount and spinning speed were appropriately controlled, and the spinning dope was spun.

[0112] The spinning dope on the filaments discharged from the spinning nozzle was passed through an air-gap region and then fed into a coagulation bath containing a coagulation liquid (with a concentration of 75 wt% water and 25 wt% NMMO, at a temperature of 25 °C). In this process, the cooled air in the air-gap region provides a temperature of 8 °C and an airflow rate of 200 Nm^3/h to primarily coagulate the spinning dope. In addition, the concentration of the coagulation liquid was continuously monitored using a sensor and a refractometer.

[0113] Then, the coagulated lyocell filaments were washed. Specifically, the filaments were fed to a take-up roller, and the residual NMMO in the filaments was removed by washing solution sprayed in the washing device. Then, the washed filaments were immersed in a bath designed with a predetermined emulsion concentration.

[0114] The filaments immersed in the bath were then passed through nip rolls installed at the bath outlet and fed into a crimp machine for crimp formation. The crimp machine was adjusted to impart an appropriate number of crimps to the filaments, and crimped tow was produced through this crimp machine.

[Table 1]

	Example 1	Example 2	Example 3	Example 4	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4	Comparative Example 5
0.5 % CED (cP)	5.2	5.0	10.0	11.0	5.2	3.5	23.0	5.2	5.1
Hemicellulose content (wt%)	3.8	4.0	3.7	3.9	7.0	3.8	3.8	4.0	4.0
Fe (wt ppm)	1	1	1	1	1	1	1	30	5
Si (wt ppm)	10	50	80	90	50	50	50	50	110

[Evaluation Methods]

[0115]

(1) 0.5% CED (cP) - was measured according to TAPPI Standard T230 om-94. Specifically, about 0.25 g of a pulp sample was taken and dissolved in 25 mL of a 0.5% CED solution, and then measured at 25 ± 0.1 °C using a viscometer. The flow time was measured and substituted into the formula below to calculate the viscosity value.

$$V = C \times t \times d$$

(V: viscosity (cPs) of the solution; C: viscometer constant; t: flow time; d: density of the solution)

(2) Hemicellulose content - measured according to KS M 7044:2016.

Alpha cellulose: carbohydrates not dissolved in a 17.5 % NaOH aqueous solution at 20 °C
Beta cellulose: substances that precipitate upon acidification of extracted precipitate
Gamma cellulose: substances that do not precipitate upon acidification after being dissolved in an alkali
Beta cellulose and gamma cellulose are collectively referred to as "hemicellulose".

Measurement of alpha cellulose content

[0116] 5 g of dried pulp sample was placed into a 300 mL beaker covered with a lid and was allowed to stand alone in a 20 °C constant-temperature water bath for 30 minutes. The sample was evenly moistened with two 50-mL portions of a 17.5 % NaOH aqueous solution at 20 °C and allowed to stand. After sufficient dissolution, the sample was filtered under reduced pressure using a pump, washed repeatedly by adding distilled water and applying vacuum filtration. The dry weight of the residual fibers was obtained.

Alpha cellulose content (%) = (dry weight of the residual fibers/weight of the sample) × 100

Measurement of hemicellulose content

[0117]

$$\text{Hemicellulose content (\%)} = 100 - \text{alpha cellulose content (\%)}$$

[0118] **(3) Fe (ppm) and Si (ppm) content by weight** - Inorganic components in the pulp were measured using ICP-OES (Avio-550 from PerkinElmer).

[0119] **(4) Spinning processability** - The spinning properties were evaluated as favorable or poor based on the uniformity of the discharge pressure, the occurrence of filament breakage in the extruded multifilaments from the spinneret, and the presence of contamination on the spinneret. The ratings ○, △, and X were assessed according to the following criteria.

[0120] ○: The discharge pressure is uniform, no filament breakage occurs from the spinneret, and there is no contamination in the spinneret, allowing stable long-term production.

[0121] △: The discharge pressure is uniform, but filament breakage occasionally occurs from the spinneret. During long-term production, contaminants accumulate in the spinneret.

[0122] X: The discharge pressure fluctuates severely, or filament breakage occurs in the extruded multifilaments, making production impossible.

[0123] **(5) Appearance** - The appearance of the produced sample was visually inspected and classified as favorable or poor.

[0124] Favorable: The sample is close to white.

[0125] Poor: The sample appears yellowish or reddish.

[0126] **(6) Monofilament strength** - The multifilament specimen obtained following the emulsion treatment was pre-dried at 110 °C for 2 hours to reduce the moisture content below the process moisture level, and was left under the standard conditions of KS K 0901 for 24 hours or more to reach moisture equilibrium. Thereafter, the monofilament specimen was separated from the multifilament specimen. The separated monofilament specimen was tested for tensile strength at a drawing speed of 60 mm/min using a low-speed elongation-type tensile tester (Instron).

[0127] **(7) Cigarette filter cuttability** - To evaluate the quality of cuttability, a cigarette rod was produced, and its cut cross section was visually observed and classified as favorable or poor.

[0128] Favorable: The cut cross-section of the filter rod is smooth.

[0129] Poor: The cut cross-section of the filter rod is not smooth, with fibers either clumped together or protruding.

[Table 2]

	Example 1	Example 2	Example 3	Example 4	Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4	Comparative Example 5
Spinning processability	○	○	○	○	△	△	X	△	X
Appearance	○	○	○	○	X	X	X	X	X
Monofilament	3.7	3.6	6.8	7.2	2.5	1.2	8.9	1.8	1.5
strength									
Cigarette filter cuttability	○	○	○	○	○	X	X	X	X
※ Evaluation Results: ○ - Favorable; △ - Average; X - Poor									

[0130] As seen in Table 2, when the cellulose pulp has a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight, the sample exhibits favorable spinning processability, favorable appearance, and favorable cigarette cuttability, while providing a monofilament strength of 3 g/d to 7 g/d, which is sufficient for application as a filter for smoking articles. Furthermore, through Comparative Examples 1 to 5, it was confirmed that if any one of the CED viscosity, hemicellulose content, Fe content, or Si content was not satisfied, the lyocell material would be unsuitable for producing a filter for smoking articles.

[0131] While preferred embodiments according to the present disclosure have been described with reference to the drawings and examples, these are merely illustrative, and those skilled in the art would understand that various modifications and other equivalent embodiments are possible. Therefore, the scope of protection of the present disclosure should be determined by the appended claims.

Claims

1. A lyocell material prepared from a dope solution, the dope solution comprising cellulose pulp and N-methylmorpholine-N-oxide (NMMO),
   wherein the cellulose pulp has a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight.

2. The lyocell material of claim 1,
   wherein the cellulose pulp has a hemicellulose content of 4 wt% or less.

3. The lyocell material of claim 1 or 2,
   wherein the cellulose pulp comprises 1 ppm to 20 ppm by weight of iron (Fe).

4. The lyocell material of any one of claims 1 to 3,
   wherein the cellulose pulp comprises 1 ppm to 90 ppm by weight of silicon (Si).

5. The lyocell material of any one of claims 1 to 4,
   wherein the cellulose pulp has a degree of polymerization of 600 to 1,700.

6. The lyocell material of any one of claims 1 to 5,

   wherein the lyocell material comprises at least one lyocell monofilament,
   wherein the lyocell monofilament has a strength of 3 g/d to 8 g/d.

7. The lyocell material of any one of claims 1 to 6,

wherein the lyocell material is crimped, and
optionally, the lyocell material has 10 crimps to 50 crimps per inch.

8. The lyocell material of any one of claims 1 to 7,
wherein the lyocell material is a lyocell tow.

9. The lyocell material of claim 8,

wherein the lyocell tow has a non-circular cross-section, and/or
the lyocell tow has a total fineness of 15,000 to 55,000 denier.

10. The lyocell material of any one of claims 1 to 9,
wherein the lyocell material is for use in a smoking article filter.

11. A smoking article, comprising the lyocell material according to any one of claims 1 to 10.

12. A method of preparing a lyocell material, the method comprising:
spinning, through a spinneret, a dope solution comprising cellulose pulp and N-methylmorpholine-N-oxide (NMMO);

coagulating the spun dope solution to obtain a lyocell multifilament;
washing the lyocell multifilament and treating the lyocell multifilament with emulsion; and
crimping the emulsion-treated multifilament to obtain crimped tow,
wherein the cellulose pulp has a CED viscosity of 4.0 to 11.0, a hemicellulose content of less than 5 wt%, an Fe content of at least 1 ppm and less than 30 ppm by weight, and an Si content of at least 1 ppm and less than 100 ppm by weight, relative to a total weight of the cellulose pulp.

13. The method of claim 12,
wherein the cellulose pulp has a hemicellulose content of 4 wt% or less.

14. The method of claim 12 or 13,

wherein the cellulose pulp comprises 1 ppm to 20 ppm by weight of Fe, and/or
the cellulose pulp comprises 1 ppm to 90 ppm by weight of Si.

15. The method of any one of claims 12 to 14,
wherein the cellulose pulp has a degree of polymerization of 600 to 1,700.

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EUROPEAN SEARCH REPORT

Application Number
EP 24 21 8087

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The present search report has been drawn up for all claims

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Place of search	Date of completion of the search	Examiner
Munich	27 March 2025	Lindner, Claudia

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X : particularly relevant if taken alone
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A : technological background
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