



(11)

EP 4 335 308 A1

(12)

EUROPEAN PATENT APPLICATION
published in accordance with Art. 153(4) EPC

(43) Date of publication:

13.03.2024 Bulletin 2024/11

(21) Application number: **22876749.7**

(22) Date of filing: **21.09.2022**

(51) International Patent Classification (IPC):

A24D 3/10 (2006.01) **A24D 3/06** (2006.01)
A24D 3/14 (2006.01) **D01F 8/02** (2006.01)
D01F 2/00 (2006.01) **D01D 5/06** (2006.01)
D01D 10/06 (2006.01) **A24D 3/02** (2006.01)

(52) Cooperative Patent Classification (CPC):

A24D 3/02; A24D 3/06; A24D 3/10; A24D 3/14;
D01D 5/06; D01D 10/06; D01F 2/00; D01F 8/02

(86) International application number:

PCT/KR2022/014137

(87) International publication number:

WO 2023/054976 (06.04.2023 Gazette 2023/14)

(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 MK MT NL NO
PL PT RO RS SE SI SK SM TR

Designated Extension States:

BA ME

Designated Validation States:

KH MA MD TN

(30) Priority: **30.09.2021 KR 20210129846**

20.09.2022 KR 20220118769

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(54) **LYOCELL MATERIAL FOR CIGARETTE FILTER AND METHOD FOR MANUFACTURING SAME**

(57) The present disclosure relates to a lyocell material for a cigarette filter and a method of manufacturing the same, in which, by setting the optimal total denier range of crimped lyocell tow, which may realize filter properties equivalent to cellulose acetate (CA) currently used

as a cigarette filter, the optimal processability for manufacturing a cigarette filter may be secured, and due to the excellent biodegradability thereof, environmental pollution factors caused by conventional discarded cigarette butts may be reduced.

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Description

Technical Field

5 CROSS-REFERENCE TO RELATED APPLICATION(S)

[0001] This application claims the benefit of Korean Patent Application Nos. 10-2021-0129846 filed on September 30, 2021, and 10-2022-0118769 filed on September 20, 2022 in the Korean Intellectual Property Office, the disclosures of which are incorporated herein in their entirety by reference.

10 **[0002]** The present disclosure relates to a lyocell material for a cigarette filter and a method of manufacturing same.

Background Art

15 **[0003]** In general, cigarette filters include a cellulose acetate tow obtained by extracting cellulose from wood pulp and acetylating the cellulose.

[0004] In addition, cigarette filters are assembled into cigarette products, distributed to consumers, and provided for smoking, and then finally discarded after smoking. Also, cigarette filters may directly be discarded as manufacturing residue from plants for manufacturing the cigarette filters. Such cigarette filter waste is collected as garbage and buried for disposal. In addition, in some cases, cigarette filter waste is not collected as garbage but may remain in the natural environment.

20 **[0005]** Thus, in order to manufacture biodegradable cigarette filters, there have been suggested methods such as a method of adding an additive formed of a biodegradable polymer to increase a degradation rate of cellulose acetate, a method of using cellulose acetate having a low degree of substitution to increase biodegradability, or a method of using a polymer composition having high biodegradability such as poly-hydroxybutyrate (PHB)/polyvinyl butyral (PVB) and starch as raw materials for filter tows.

25 **[0006]** In addition, studies have recently been conducted to replace cellulose acetate tows with eco-friendly materials to protect the natural environment and reduce costs. For example, the development of tows using lyocell fibers obtained by fiberizing cellulose, unlike cellulose acetate, is in progress.

30 **[0007]** However, a material satisfying filter performance equivalent to or superior to that of CA currently used as a cigarette filter material and reducing environmental pollution problems to desired levels has not been developed.

Disclosure

Technical problem

35 **[0008]** Provided are a lyocell material for a cigarette filter capable of realizing cigarette filter performance equivalent to that of cellulose acetate (CA) products currently used by using the lyocell material and a method of manufacturing same.

[0009] Provided also are a lyocell material for a cigarette filter capable of reducing environmental pollution factors caused by the waste of CA cigarette butts and a method of manufacturing same.

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Technical Solution

45 **[0010]** According to an aspect of the present disclosure, a method of manufacturing a lyocell material for a cigarette filter includes: (S1) spinning a lyocell spinning dope including a cellulose pulp and an N-methylmorpholine-N-oxide (NMMO) aqueous solution;

(S2) coagulating the lyocell spinning dope spun in the step (S1) to obtain lyocell multifilaments;

(S3) water-washing the lyocell multifilaments obtained in the step (S2);

(S4) oil-treating the lyocell multi-filaments water-washed in the step (S3);

50 (S5) crimping the lyocell multifilaments oil-treated in the step (S4) to obtain a crimped tow; and

(S6) drying the crimped tow,

wherein the step (S5) is performed by crimping the lyocell multifilaments oil-treated in the step (S4) at the crimper draft ratio of 1.01 to 1.3 calculated by Equation 1 below, and

55 the crimped tow has a fineness range of 25000 to 45000 De:

[Equation 1]

$$\text{Crimper draft ratio} = V1/V0$$

wherein in Equation 1, V0 is a speed immediately before feeding the oil-treated lyocell multi-filaments to a crimper M/C, and V1 is a roller rotation speed of the crimper M/C to provide crimps to the oil-treated lyocell multi-filaments.

[0011] Also, according to another aspect of the present disclosure, a lyocell material for a cigarette filter includes a crimped tow formed of lyocell multi-filaments spun from a lyocell spinning dope including cellulose pulp and an N-methylmorpholine-N-oxide (NMMO) aqueous solution, wherein the crimped tow has a fineness range of 25000 to 45000 De.

[0012] Also, according to another aspect of the present disclosure, a cigarette filter includes the lyocell material for a cigarette filter.

[0013] Hereinafter, a lyocell material for a cigarette filter and a method of manufacturing same according to an embodiment of the present disclosure will be described in detail.

[0014] Before that, technical terms used in the present specification are merely used to describe particular embodiments, and are not intended to limit the present disclosure, unless they have clearly different meanings in the context.

[0015] Throughout the specification, an expression used in the singular encompasses the expression of the plural, unless otherwise indicated.

[0016] As used herein, the term 'include' specifies particular properties, regions, integers, steps, operations, elements, and/or components and are not intended to preclude the possibility that one or more other properties, regions, integers, steps, operations, elements, and/or components thereof may exist or may be added.

[0017] As used herein, the term 'at least one of' or "one or more", when preceding a list of elements, should be understood to include all possible combinations of the elements.

[0018] Hereinafter, the present disclosure will be described in detail.

Method of Manufacturing Lyocell Material

[0019] According to an embodiment of the present disclosure, provided is a method of manufacturing a lyocell material for a cigarette filter including: (S1) spinning a lyocell spinning dope including cellulose pulp and an N-methylmorpholine-N-oxide (NMMO) aqueous solution; (S2) coagulating the lyocell spinning dope spun in the step (S1) to obtain lyocell multi-filaments; (S3) water-washing the lyocell multifilaments obtained in the step (S2); (S4) oil-treating the lyocell multi-filaments water-washed in the step (S3); (S5) crimping the lyocell multifilaments oil-treated in the step (S4) to obtain a crimped tow; and (S6) drying the crimped tow, wherein the step (S5) is performed by crimping the lyocell multifilaments oil-treated in the step (S4) at the crimper draft ratio of 1.01 to 1.3 calculated by Equation 1 below, and the crimped tow has a fineness range of 25000 to 45000 De.

[Equation 1]

$$\text{Crimper draft ratio} = V1/V0$$

[0020] In Equation 1, V0 is a speed immediately before feeding the oil-treated lyocell multifilaments to a crimper M/C, and V1 is a roller rotation speed of the crimper M/C to provide crimps to the oil-treated lyocell multi-filaments.

[0021] The present disclosure provides a lyocell material for a cigarette filter capable of replacing a conventional cigarette filter material by manufacturing a cigarette filter tow to which a lyocell with excellent biodegradability is applied and a method of manufacturing same.

[0022] In addition, in the present disclosure, a tow for a cigarette filter having excellent biodegradability and replacing cigarette filter performance equivalent to that of cellulose acetate (CA) products which are currently used, is manufactured by using the lyocell material.

[0023] Specifically, the present disclosure may obtain optimal processability for manufacturing a cigarette filter by setting an optimal fineness range of a lyocell tow provided with crimps capable of realizing physical properties of filters equivalent to those of CA, currently used in cigarette filters, by controlling the crimper draft ratio in a crimping process while a lyocell cigarette filter tow is manufactured.

[0024] According to an embodiment of the present disclosure, process draft conditions of 1.01 to 1.3 times may be applied to filaments in a crimping process to provide uniform wrinkles to the lyocell cigarette filter tow.

[0025] In the crimping process, a crimper draft ratio may be calculated by Equation 1 below.

[Equation 1]

Crimper draft ratio = $V1/V0$

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[0026] In Equation 1, $V0$ is a speed immediately before feeding the oil-treated lyocell multifilaments to a crimper M/C, and $V1$ is a roller rotation speed of the crimper M/C to provide crimps to the oil-treated lyocell multi-filaments.

[0027] The Crimper M/C refers to a crimper machine.

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[0028] In this regard, if the crimper draft ratio is less than 1.01 times, a tensile strength of a filament tow fed into a crimper is not stable, causing a problem of providing non-uniform crimps. If the crimper draft ratio is greater than 1.3 times, crimps are excessively provided to the filaments fed into the crimper, causing problems of deterioration of physical properties or failing to provide uniform crimps to left and right edges of the tow due to variation of filament width.

[0029] In addition, to realize physical properties of the cigarette filter, the tow has a number of crimps of 25 to 50 per inch.

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[0030] Additionally, ultimately, the lyocell tow is characterized to have a fineness range (i.e., thickness) of 25000 to 45000 De to realize optimal cigarette filter performance.

[0031] In this case, if the fineness of the lyocell tow is less than 25000 De or greater than 45000 De, it is difficult to obtain processibility and physical properties of the cigarette filter. The fineness of the lyocell tow may be realized by controlling the crimper draft ratio and adjusting a single fiber fineness of the lyocell multi-filaments to a range of 2.0 to 2.8 De during a spinning process.

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[0032] Also, according to an embodiment of the present disclosure, cigarette filters may generally be classified into a regular type and an ultra-slim type, and the present disclosure may be applied to regular type cigarette filters.

[0033] The cigarette filter manufactured by applying the tow may realize a suction resistance required for various cigarettes and may uniformly realize physical properties (e.g., filter circumference and suction resistance) required for cigarette filters.

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[0034] Hereinafter, each of the operations of the method of manufacturing a lyocell material for cigarette filters of the present disclosure will be described in more detail.

[Operation (S1)]

30

[0035] In the present disclosure, the step (S1) is a step of spinning the lyocell spinning dope including cellulose pulp and an N-methylmorpholine-N-oxide (NMMO) aqueous solution. In this regard, the lyocell spinning dope may include 8 to 13 wt% of the cellulose pulp; and 87 to 92 wt% of the N-methylmorpholine-N-oxide aqueous solution, wherein the cellulose pulp may include 85 to 97 wt% of alpha-cellulose and have a degree of polymerization (DPw) of 600 to 1700.

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[0036] If an amount of the cellulose pulp is less than 8 wt%, it is difficult to realize fibrous properties, and if the amount of the cellulose pulp exceeds 13 wt%, it may be difficult to dissolve the cellulose pulp in the aqueous solution. In addition, if an amount of the N-methylmorpholine-N-oxide aqueous solution is less than 87 wt%, dissolution viscosity significantly increases which is not desirable, and if the amount of the aqueous solution exceeds 92 wt%, spinning viscosity significantly decreases, making it difficult to manufacture uniform fibers in the spinning process.

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[0037] In the N-methylmorpholine-N-oxide aqueous solution, a weight ratio of N-methylmorpholine-N-oxide to water may be from 93:7 to 85:15. If the weight ratio of the N-methylmorpholine-N-oxide to water exceeds 93:7, dissolution temperature increases causing degradation of cellulose during dissolution, and if the weight ratio is less than 85:15, solubility of a solvent decreases, making it difficult to dissolve cellulose.

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[0038] The above-described spinning dope is discharged from a spinning nozzle of a flat or donut-shaped spinneret. In this case, the spinneret serves to discharge the spinning dope in the form of filaments into a coagulation solution contained in a coagulation bath through an air gap zone. The operation of discharging the spinning dope from the spinneret may be performed at 100 to 120 °C.

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[0039] In addition, the step (S1) may be performed by controlling a discharge amount and a spinning speed such that a single fiber fineness of the lyocell multi-filaments is adjusted to 2.0 to 2.8 De. Specifically, deterioration in processibility of cigarette filters may be prevented and physical properties required for the industry may be more effectively achieved by adjusting a crimper draft ratio, which will be described below, and performing spinning by adjusting the single fiber fineness of the lyocell multi-filaments within the above-described range while spinning the lyocell filaments.

[Operation (S2)]

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[0040] In the present disclosure, the step (S2) is a step of coagulating the lyocell spinning dope spun in the step (S1) to obtain lyocell multi-filaments, and the coagulating in the step (S2) may include: primary coagulation performed by air quenching (Q/A) by coagulating the spun dope while supplying cooling air thereto; and secondary coagulation performed by immersing the primarily coagulated spun dope in a coagulation solution.

[0041] In the step (S1), after discharging the spinning dope through the flat or donut-shaped spinneret, the spun dope may pass through the air gap zone in a space between the above-described spinneret and the coagulation bath. In such an air gap zone, the cooling air may be supplied in a direction from one side to the opposite side of the spinneret in the case of the flat spinneret, and the cooling air may be supplied outwards from an air-cooling part located inside the spinneret in the case of the donut-shaped spinneret and a suction function may also be provided on the opposite side or the outside. The primary coagulation may be performed by air quenching including supplying the cooling air to the spun dope.

[0042] The lyocell multi-filaments of the step (S2) used in the coagulation has a single fiber fineness of 2.0 to 2.8 De.

[0043] In this regard, factors affecting physical properties of the lyocell multi-filaments obtained in the step (S2) are temperature and wind velocity of cooling air in the air gap zone, and the coagulation of the step (S2) may be performed by supplying cooling air at a temperature of 4 to 15 °C and with an airflow rate of 50 to 250 L/m³.

[0044] If the temperature of the cooling air is below 4 °C during the primary coagulation, a surface temperature of the spinneret decreases to form a non-uniform cross-section of the lyocell multi-filaments and deteriorate spinning processibility, and if the temperature is above 15 °C, the primary coagulation by the cooling air does not proceed sufficiently, resulting in deterioration of spinning processibility.

[0045] In addition, if the airflow rate of cooling air is less than 50 L/m³ during the primary coagulation, the primary coagulation by the cooling air does not proceed sufficiently to deteriorate spinning processibility, thereby causing breakage of filaments in the tow, and if the airflow rate exceeds 250 L/m³, the spinning dope discharged from the spinneret is shaken by the air, resulting in deterioration of spinning processibility.

[0046] After the primary coagulation by air quenching, secondary coagulation may proceed by supplying the spun dope to a coagulation bath containing a coagulation solution. Meanwhile, for appropriate progress of the secondary coagulation, a temperature of the coagulation solution may be 30 °C or below. This is because the temperature for the secondary coagulation is maintained at a level not to be excessively higher than necessary, so that coagulation rate is appropriately maintained. In this regard, the coagulation solution is not particularly limited because it may be prepared and used in a composition commonly available in the art to which the present disclosure belongs.

[Operation (S3)]

[0047] In the present disclosure, the step (S3) is a step of washing the lyocell multi-filaments obtained in the step (S2) described above with water. More specifically, the lyocell multifilaments obtained in the step (S2) were fed into a draw roller and then into a water-washing bath to be water-washed.

[0048] In the operation of water-washing the filaments, a water-washing solution at 0 to 100 °C may be used taking into consideration of recovery and reuse of the solvent after the water-washing process. Water may be used as the water-washing solution, and other additives may further be added thereto if required.

[Operation (S4)]

[0049] In the present disclosure, the step (S4) is a step of oil-treating the water-washed lyocell multi-filaments obtained in the step (S3).

[0050] For the oil treatment, a method of immersing the filament tow in an oil bath made-up of oil and releasing the filament tow may be used.

[0051] The oil treatment is performed in a state where the multi-filaments are completely immersed in the oil, and an amount of oil applied to the multi-filaments is uniformly maintained by squeezing rollers provided to an entry and release rollers of an oil treatment device.

[0052] In addition, the oil used for application to the filaments may be developed exclusively for lyocells unlike oil used for commonly used fibers.

[0053] According to a preferred embodiment, the oil may provide a lubricating function capable of uniformly imparting physical properties of the cigarette filter tow, may optimize processibility of a filter manufacturing process by imparting an appropriate level of smoothness and cohesion, and may provide hydrophobicity, which delays penetration of smoker's saliva into the cigarette filter, to reduce a filter hardness decrease rate for minimizing a phenomenon in which hardness of a filter decreases as a result of deformation of a shape of the filter by smoker's saliva during smoking. In addition, the oil may be composed of components harmless to a smoker's body even when a certain amount of the oil remains in the cigarette filter.

[0054] However, the oil serves to decrease friction of the filaments upon contact with a drying roller and a guide and in the crimping process and also to effectively form crimps on fibers, and the oil is not particularly limited so long as the oil is commonly available for production of lyocell filaments.

[0055] As a preferred example, the oil may include at least one selected from the group consisting of a lubricating component, a cohesion component, a smoothness component, and a hydrophobic component.

[0056] The oil may be used such that a concentration of the oil is maintained in a range of 2 to 8 wt% or 3 to 7 wt% based on the lyocell multi-filaments. Maintaining of the amount of the oil within the ranges described above may play a role in reducing friction generated while the lyocell multi-filaments are brought into contact with the machine and facilitating formation of crimps between fibers during the crimping operation.

[Operation (S5)]

[0057] The step (S5) is a step of crimping the lyocell multi-filaments oil-treated in the step (S4) to obtain the crimped tow.

[0058] Specifically, the method is a step of crimping the oil-treated lyocell multi-filaments by applying steam and pressure thereto, and a crimped tow may be obtained via the step (S5).

[0059] The term "crimping" used throughout the specification refers to forming wrinkles in filaments mainly to provide bulky texture to fibers. In the present disclosure, the crimping process may be performed by using a stuffer box provided with a press roller. The press roller may be a nip roller.

[0060] The lyocell tow for cigarette filters provided in the present disclosure has a crimper draft ratio in the tow manufacturing process to realize optimized physical properties of a filter.

[0061] According to a preferred embodiment, processing draft conditions of 1.01 to 1.3 times calculated by Equation 1 above may be applied to the filaments to form wrinkles in the lyocell cigarette filter tow in the crimping process.

[0062] In addition, conditions for the machine to form uniform crimps may vary according to the crimper draft conditions, and factors of variation may be controlling of a pressure applied to the steam box of a front of the crimper M/C, a pressure applied to a roller of the crimper M/C, and pressure conditions of a doctor blade.

[0063] For example, the front of the stuffer box is provided with the steam box for steam treatment of the filament tow, the conditions may be controlled to satisfy a pressure of steam supplied to the steam box of 0.1 to 3.0 kgf/cm², a pressure of 1.5 to 4 kgf/cm² applied to a press roller, and a doctor blade pressure of 0.1 to 3 kgf/cm². As the above-described conditions are controlled, uniform crimps may be provided.

[0064] If the steam pressure is less than 0.1 kgf/cm², a modulus of the filaments cannot be lowered to a level appropriate for the process, failing to easily form crimps, and if an excessive amount of steam exceeding 3.0 kgf/cm² is supplied, the modulus of the filaments is minimized but the number of required crimps is not uniform due to a too high water content.

[0065] In addition, if the pressure applied by the press roller is less than 1.5 kgf/cm², the fineness of the fed filament may not be uniform, failing to uniformly form the crimps in a desired number, and if the pressure exceeds 4 kgf/cm², a too strong pressing force drastically decreases an amount of water or oil present in the tow so that the filaments may not easily pass through the stuffer box. In this case, it is preferable to maintain a gap between the press rollers in a range of 0.01 to 3.0 mm. If the gap between the press rollers is less than 0.01 mm, a pressure applied to the filaments by the roller increases, failing to formation of crimps or resulting in damage caused by surface friction of the filaments after formation of crimps, and if the gap exceeds 3.0 mm, a filament slipping phenomenon may occur between the press rollers, making it difficult to form uniform crimps.

[0066] Also, the doctor blade moves up and down to uniformly form crimps after passing through the press rollers. If the pressure of the doctor blade is less than 0.1 kgf/cm², an upper plate is not fixed due to an internal pressure of the stuffer box and the tow is retained in the stuffer box for a long period of time, so that continuity of the process cannot be maintained or an appropriate retention time cannot be provided, failing to control the number of crimps in the tow. If the pressure exceeds 3.0 kgf/cm², the tow may not be efficiently discharged from the stuffer box so that the crimps may be non-uniformly shaped.

[0067] Tows crimped under the above-described conditions may include and maintain 25 to 50 crimps per inch, or 30 to 45 crimps per inch. In addition, in the case of forming a monofilter and a composite filter with filter rods having diameters of 24.2 mm and 24.5 mm, suction resistance of about 240 to 590 PD (mmH₂O) may be obtained based on KS H ISO 6565 measurement standards. Various suction resistance values are applied to filters according to cigarette products. Accordingly, the lyocell crimped tow of the present disclosure is biodegraded and removed in a short period of time, and thus it is not only eco-friendly but also more effectively satisfies physical properties required for cigarette filters, such as suction resistance, filter hardness, and filter removability, thereby maximizing the effects.

[Operation (S6)]

[0068] The step (S6) is a step of drying the crimped tow obtained in the step (S5) at a constant temperature.

[0069] Specifically, the oil contained in the oil bath and uniformly applied to the filament tow immersed therein is picked up as a level capable of providing the uniform number of crimps in the crimping process.

[0070] Subsequently, the crimped tow is dried by using a continuous drying device to prepare a final lyocell crimped tow containing a uniform amount of oil.

[0071] The operation of drying the crimped tow may be performed by using a continuous drying device at a temperature of 105 to 135 °C for 15 minutes to 45 minutes or at a temperature of 110 to 130 °C for 20 minutes to 40 minutes.

[0072] When the drying temperature is 105 °C or below, the tow is not completely dried failing to retain a required regain. When the drying temperature is 135 °C or above, whiteness may be lowered due to a yellowing phenomenon. Also, if the drying time is 15 minutes or less, there may be an irregularly dried portion. If the drying time is 45 or more, the tow surface may be damaged due to the excessive drying time and whiteness may be lowered due to a yellowing phenomenon.

[0073] In addition, the lyocell tow for a cigarette filter of the finally prepared tow after the drying process may have a fineness in the range of 25000 to 45000 De.

[0074] In this case, where the fineness of the lyocell tow is less than 25000 De or greater than 45000 De, it is difficult to obtain processability and desired physical properties of the cigarette filter.

[0075] Also, the fineness range of the lyocell tow may be obtained by adjusting the single fiber fineness of the lyocell multi-filaments to a range of 2.0 to 2.8 De in the step (S1) and controlling the crimper draft ratio of the step (S5).

Lyocell Material for Cigarette Filter

[0076] According to another embodiment, provided is a lyocell material for a cigarette filter including a crimped tow formed of lyocell multi-filaments obtained by spinning a lyocell spinning dope including cellulose pulp and an N-methyl-morpholine-N-oxide (NMMO) aqueous solution, wherein the crimped tow is a lyocell tow having a fineness in a range of 25000 to 45000 De.

[0077] The number of crimps of the lyocell tow may be adjusted to 25 to 50 per inch.

[0078] The lyocell spinning dope may include 8 to 13 wt% of the cellulose pulp; and 87 to 92 wt% of the N-methyl-morpholine-N-oxide aqueous solution.

[0079] The cellulose pulp may include 85 to 99 wt% of alpha-cellulose and may have a degree of polymerization (DPw) of 600 to 1700.

[0080] In addition, according to another embodiment of the present disclosure, a cigarette filter including the lyocell material for a cigarette filter as a lyocell tow may be provided.

[0081] The composition of the cigarette filter is not limited except for the lyocell tow.

[0082] For example, the cigarette filter may include a cylindrical lyocell tow including a plurality of lyocell fibers, a plasticizer, a curing agent, and the like.

[0083] The plurality of lyocell fibers are connected to each other by the curing agent to exhibit a mutually bonded structure in which surfaces of the fibers are in contact with the curing agent.

[0084] The curing agent may be any known material used to increase hardness of the filter. For example, the curing agent may include a cellulose derivative, a vinyl derivative, a vinyl emulsion derivative, a natural polymer including sodium alginate, and a polymer resin such as starch and a starch derivative.

[0085] Therefore, the present disclosure may provide a lyocell cigarette filter tow capable of realizing filter performance (manufacturing processability and physical properties of a filter) equivalent to that of CA, which is a material for cigarette filters currently used in the art. In addition, according to the present disclosure, currently available CA cigarette butts (filter) may be manufactured by an eco-friendly method compared to conventional methods.

[0086] In addition, by applying the lyocell tow prepared according to the above-described method, a regular-type cigarette filter may be uniformly manufactured and required physical properties (filter circumference and suction resistance) may be uniformly realized.

Advantageous Effects

[0087] According to the present disclosure, optimal processability for manufacturing a cigarette filter may be obtained by setting an optimal fineness range of a lyocell tow provided with crimps capable of realizing physical properties of filters equivalent to those of CA, currently used in cigarette filters.

[0088] Also, the cigarette filter prepared by using the lyocell tow may have a suction resistance required for various cigarettes and may exhibit physical properties thereof (filter circumference and suction resistance).

[0089] In addition, because the lyocell tow is provided by using an eco-friendly method according to the present disclosure, conventional CA cigarette filters may be replaced thereby, and thus environmental pollution factors caused by discarded cigarette butts may be reduced.

[0090] That is, the present disclosure may provide excellent biodegradability, processability, and physical properties, and thus economical effect may be obtained thereby by replacing conventional cigarette filter materials.

Best Mode

[0091] Hereinafter, the present disclosure will be described in more detail with reference to the following examples. However, the following examples are merely presented to exemplify the present disclosure, and the scope of the present

disclosure is not limited thereto.

Example 1

[0092] Cellulose pulp having a degree of polymerization (DPw) of 820 and including 93.9 % of alpha-cellulose was mixed with a NMMO/H₂O mixed solvent (weight ratio of 90/10) including 0.01 wt% of propyl gallate to prepare a spinning dope for preparing a tow for a cigarette filter with a concentration of 12 wt%. First, the spinning dope was maintained at a spinning temperature of 110 °C in a spinning nozzle and spun by adjusting a discharge amount and a spinning rate such that a single fiber fineness of filaments was adjusted to 2.4 De.

[0093] The spinning dope in the form of filaments discharged from the spinning nozzle was supplied to a coagulation solution contained in a coagulation bath through an air gap zone. In this case, the spinning dope was primarily coagulated in the air gap zone using cooling air at a temperature of 8 °C and an airflow rate of 100 L/m³. A coagulation solution including 75 wt% of water and 25 wt% of NMMO was used at 25 °C. In this case, a concentration of the coagulation solution was continuously monitored by using a sensor and a refractometer.

[0094] The coagulated filaments were washed by a water-wash solution sprayed by a water-washing device via a draw roller to remove NMMO remaining in the filaments.

[0095] Subsequently, the water-washed filaments were immersed in an oil bath made-up of 2 wt% of oil.

[0096] The filaments immersed in the bath were treated at a pressure of 2 kgf/cm² by using a nip roller provided at a release portion of the bath and fed into a crimper M/C to form crimps.

[0097] In this regard, a ratio of providing crimps, i.e., crimper draft ratio, was set to 1.1 times, the tow was prepared by supplying steam to the steam box at 0.3 kgf/cm², setting a roller pressure of the crimper M/C roller to 2.5 kgf/cm², and setting a doctor blade pressure to 1.0 kgf/cm². The prepared tow passed through a continuous drying device set to a temperature of 120 °C to obtain a dried tow product.

[0098] The obtained tow was used to prepare a filter for a cigarette filter by targeting a regular filter suction resistance of 450 ± 12% (mmH₂O).

Example 2

[0099] A lyocell tow for a cigarette filter was prepared in the same manner as in Example 1, except that the single fiber fineness was adjusted to 2.6 De, and the crimper draft ratio was adjusted to 1.05 times.

Example 3

[0100] A lyocell tow for a cigarette filter was prepared in the same manner as in Example 1, except that the single fiber fineness was adjusted to 2.8 De, and the crimper draft ratio was adjusted to 1.2 times.

Comparative Example 1

[0101] A lyocell tow for a cigarette filter was prepared in the same manner as in Example 1, except that the crimper draft ratio was adjusted to 1.0 times.

Comparative Example 2

[0102] A lyocell tow for a cigarette filter was prepared in the same manner as in Example 1, except that the crimper draft ratio was adjusted to 1.35 times.

Comparative Example 3

[0103] A lyocell tow for a cigarette filter was prepared in the same manner as in Example 1, except that the crimper draft ratio was adjusted to 1.5 times.

Comparative Example 4

[0104] A lyocell tow for a cigarette filter was prepared in the same manner as in Example 1, except that the single fiber fineness was adjusted to 3.0 De, and the crimper draft ratio was adjusted to 1.1 times.

[Experimental Example]

[0105] Physical properties of the lyocell tows of the examples and comparative examples described above were measured according to the following method, and the results are shown in Table 1.

Method of Measuring Physical Property**(1) Measurement of Tow Fineness**

[0106] A sample of the tow to be measured was collected in a size of 2 m and allowed to stand in a constant temperature and humidity room at 20 °C with a humidity of 65 %. After fixing one end of the stabilized tow, the other end thereof was mounted with a 2 kg-weight. After stabilizing the tow elongated by the weight for 5 seconds, the sample was cut to a size of 90 cm and weighed. The fineness of the tow was converted into a measured weight \times 10000 value according to a denier conversion method.

(2) Measurement of Number of Crimps

[0107] According to the KS K 0326 standards, samples of 20 strands where crimps were not damaged were collected and each strand was added onto a previously prepared glossy paper sheet (spacing distance of 25 mm) with a celluloid 4 to 5 % amyl acetate adhesive to be stretched by 25 ± 5 % relative to a length of a single strand, and then was left stand for drying the adhesive. The number of crimps of each sample was counted by applying a primary load of 1.96/1000 cN (=2 mgf) per 1 De to each strand using a crimp tester and the number of crimps in 25 mm was determined and averaged by excluding decimal places.

(3) Measurement of Shape of Crimp

[0108] A shape of crimps was visually identified using an optical microscope after sampling in the same manner as in the measurement of the number of crimps.

(4) Processibility of Cigarette Filter Manufacturing

[0109] Processibility of the cigarette filter manufacturing process was identified by evaluating processibility maintainability until a sample of one batch was completely consumed after feeding the tow to a cigarette filter manufacturing facility.

(5) Measurement of Suction Resistance and Circumference of Cigarette Filter

[0110] Cigarette filter rods were prepared by using the tows prepared in the above-described examples and comparative examples, and suction resistance thereof was measured according to the KS H ISO 6565 standards and suction resistance of each rod was measured using a circumference measurer.

[Table 1]

	Finness of tow (De)	Number of crimps of tow (per inch)	Crimp shape	cigarette filter Processibility of manufacture	Filter suction resistance (mmH ₂ O)	Filter circumference (mm)
Example 1	36200	36~40	good	good (no breakage)	446~454	24.22
Example 2	34500	30~34	good	good (no breakage)	446~454	24.18
Example 3	39600	40~43	good	good (no breakage)	446~454	24.24
Comparative Example 1	impossible to manufacture	impossible to measure	-	-	-	-
Comparative Example 2	37000	34~46	poor	poor (impossible to continuously manufacture)	375~420	24.52

(continued)

	Finness of tow (De)	Number of crimps of tow (per inch)	Crimp shape	cigarette filter Processibility of manufacture	Filter suction resistance (mmH ₂ O)	Filter circumference (mm)
Comparative Example 3	38400	32~50	poor	poor (impossible to continuously manufacture)	220~400	24.55
Comparative Example 4	45500	28~52	poor	poor (impossible to continuously manufacture)	200~380	24.58

[0111] As shown in Table 1, in the case of Examples 1 to 3, the number of crimps was uniform according to the tow manufacturing conditions, and the tows manufactured by setting the conditions of the facility during manufacturing of cigarette filters had physical properties required for cigarette filters.

[0112] However, in the case of Comparative Example 1, conditions for manufacturing a tow were not appropriate, and thus tows could not be manufactured even by setting the conditions of the manufacturing process, and filter performance could not be identified.

[0113] In addition, although the tows were manufactured in the case of Comparative Examples 2 to 4, the tows were non-uniformly formed due to inappropriate manufacturing conditions.

[0114] That is, in Comparative Examples 1 to 3, the crimper draft ratio was out of the range of the present disclosure, so that it was impossible to manufacture the tow. Even when the tow was manufactured, the shape of crimps was poor, so that the cigarette filter was not able to be continuously manufactured, and accordingly suction resistance thereof was lower than those of the examples. In addition, in the case of spinning the spinning dope of Comparative Example 4, the filaments had a single fiber fineness of 3.0 De, which exceeded the range of the single fiber fineness of the filaments of the present disclosure, so that the fineness range of the tow exceeded 4000 De, and thus it was confirmed that processibility and physical properties of the cigarette filter thereof were inferior to those of Examples 1 to 3.

[0115] Therefore, it was confirmed that processibility was not maintained due to non-uniformity of the tow during manufacturing of the cigarette filter to which the tow manufactured according to the comparative examples, and it was difficult to realize filter performance due to non-uniformity of the tow.

[0116] On the contrary, in Example 1 to 3, the lyocell cigarette filter tow may have a fineness range of 25000 to 45000 De capable of realizing optimal cigarette filter performance by adjusting the crimper draft ratio in the crimping process. Therefore, according to the present disclosure, optimal processibility for manufacturing a cigarette filter may be obtained by providing the lyocell tow having crimps capable of realizing physical properties of the cigarette filter equivalent to those of CA currently used in the art.

Claims

1. A method of manufacturing a lyocell material for a cigarette filter, the method comprising:

(S1) spinning a lyocell spinning dope comprising cellulose pulp and an N-methylmorpholine-N-oxide (NMMO) aqueous solution;

(S2) coagulating the lyocell spinning dope spun in the step (S1) to obtain lyocell multifilaments;

(S3) water-washing the lyocell multifilaments obtained in the step (S2);

(S4) oil-treating the lyocell multi-filaments water-washed in the step (S3);

(S5) crimping the lyocell multifilaments oil-treated in the step (S4) to obtain a crimped tow; and

(S6) drying the crimped tow,

wherein the step (S5) is performed by crimping the lyocell multifilaments oil-treated in the step (S4) at the crimper draft ratio of 1.01 to 1.3 calculated by Equation 1 below, and the crimped tow has a fineness range of 25000 to 45000 De:

[Equation 1]

Crimper draft ratio = $V1/V0$

wherein in Equation 1, V_0 is a speed immediately before feeding the oil-treated lyocell multi-filaments to a crimper M/C, and V_1 is a roller rotation speed of the crimper M/C to provide crimps to the oil-treated lyocell multi-filaments.

- 5 **2.** The method of claim 1, wherein the lyocell multi-filaments obtained in the step (S2) have a single fiber fineness of 2.0 to 2.8 denier.
- 3.** The method of claim 1, wherein the lyocell tow is formed to have a number of crimps of 25 to 50 per inch.
- 10 **4.** The method of claim 1, wherein the step (S5) is performed by using a stuffer box of the crimper M/C.
- 5.** The method of claim 4, wherein the stuffer box is controlled to satisfy conditions of a steam pressure of 0.1 to 3.0 kgf/cm², a pressure of 1.5 to 4 kgf/cm² applied to a press roller of the crimper M/C, and a doctor blade pressure of 0.1 to 3.0 kgf/cm².
- 15 **6.** The method of claim 1, wherein the drying of the crimped tow obtained in step (S6) is performed by using a continuous drying device at a temperature of 105 to 135 °C for 15 minutes to 45 minutes.
- 20 **7.** The method of claim 1, wherein the coagulating in step (S2) is performed by supplying cooling air to the spinning dope at a temperature of 4 to 15 °C and at an airflow rate of 50 to 250 L/m³.
- 8.** The method of claim 1, wherein the lyocell spinning dope of (S1) comprises 8 to 13 wt% of the cellulose pulp and 87 to 92 wt% of the N-methylmorpholine-N-oxide aqueous solution.
- 25 **9.** The method of claim 1, wherein the cellulose pulp comprises 85 to 99 wt% of alpha-cellulose and has a degree of polymerization (DPw) of 600 to 1700.
- 30 **10.** A lyocell material for a cigarette filter, comprising a crimped tow formed of lyocell multi-filaments spun from a lyocell spinning dope comprising cellulose pulp and an N-methylmorpholine-N-oxide (NMMO) aqueous solution, wherein the crimped tow has a fineness range of 25000 to 45000 De.
- 11.** The lyocell material for a cigarette filter of claim 10, wherein the crimped tow is formed to have a number of crimps of 25 to 50 per inch.
- 35 **12.** The lyocell material for a cigarette filter of claim 10, wherein the lyocell spinning dope comprises 8 to 13 wt% of the cellulose pulp and 87 to 92 wt% of the N-methylmorpholine-N-oxide aqueous solution.
- 13.** The lyocell material for a cigarette filter of claim 10, wherein the cellulose pulp comprises 85 to 99 wt% of alpha-cellulose and has a degree of polymerization (DPw) of 600 to 1700.
- 40 **14.** A cigarette filter comprising the lyocell material for a cigarette filter according to any one of claims 10 to 13.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/KR2022/014137

A. CLASSIFICATION OF SUBJECT MATTER A24D 3/10(2006.01)i; A24D 3/06(2006.01)i; A24D 3/14(2006.01)i; D01F 8/02(2006.01)i; D01F 2/00(2006.01)i; D01D 5/06(2006.01)i; D01D 10/06(2006.01)i; A24D 3/02(2006.01)i 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) A24D 3/10(2006.01); A24D 3/02(2006.01); A24D 3/04(2006.01); A24D 3/06(2006.01); B65B 27/12(2006.01)																		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Korean utility models and applications for utility models: IPC as above Japanese utility models and applications for utility models: IPC as above Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) eKOMPASS (KIPO internal) & keywords: 담배필터(cigarette filter), 라이오 셀(lyocell), 셀룰로오스 펄프(cellulose pulp), 크림프 투우(crimped tow)																		
C. DOCUMENTS CONSIDERED TO BE RELEVANT <table border="1"> <thead> <tr> <th>Category*</th> <th>Citation of document, with indication, where appropriate, of the relevant passages</th> <th>Relevant to claim No.</th> </tr> </thead> <tbody> <tr> <td>Y A</td> <td>KR 10-2017-0075849 A (KOLON INDUSTRIES, INC. et al.) 04 July 2017 (2017-07-04) See paragraphs [0087] and [0091]; example 1; and claims 1 and 8.</td> <td>10-14 1-9</td> </tr> <tr> <td>Y</td> <td>KR 10-2016-0048738 A (KT & G CORPORATION et al.) 04 May 2016 (2016-05-04) See example 1.</td> <td>10-14</td> </tr> <tr> <td>A</td> <td>WO 02-087366 A2 (RHODIA ACTOW GMBH) 07 November 2002 (2002-11-07) See entire document.</td> <td>1-14</td> </tr> <tr> <td>A</td> <td>KR 10-2020-0047672 A (ACETATE INTERNATIONAL LLC) 07 May 2020 (2020-05-07) See entire document.</td> <td>1-14</td> </tr> <tr> <td>A</td> <td>KR 10-2016-0081726 A (KOLON INDUSTRIES, INC. et al.) 08 July 2016 (2016-07-08) See entire document.</td> <td>1-14</td> </tr> </tbody> </table>	Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.	Y A	KR 10-2017-0075849 A (KOLON INDUSTRIES, INC. et al.) 04 July 2017 (2017-07-04) See paragraphs [0087] and [0091]; example 1; and claims 1 and 8.	10-14 1-9	Y	KR 10-2016-0048738 A (KT & G CORPORATION et al.) 04 May 2016 (2016-05-04) See example 1.	10-14	A	WO 02-087366 A2 (RHODIA ACTOW GMBH) 07 November 2002 (2002-11-07) See entire document.	1-14	A	KR 10-2020-0047672 A (ACETATE INTERNATIONAL LLC) 07 May 2020 (2020-05-07) See entire document.	1-14	A	KR 10-2016-0081726 A (KOLON INDUSTRIES, INC. et al.) 08 July 2016 (2016-07-08) See entire document.	1-14
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Date of the actual completion of the international search 16 January 2023	Date of mailing of the international search report 16 January 2023																	
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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/KR2022/014137

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
KR 10-2017-0075849 A	04 July 2017	KR 10-2296584 B1	01 September 2021
KR 10-2016-0048738 A	04 May 2016	KR 10-2015-0116612 A	16 October 2015
		WO 2015-156608 A1	15 October 2015
WO 02-087366 A2	07 November 2002	EP 1389922 A2	25 February 2004
		EP 1389922 B1	15 November 2006
		KR 10-0579703 B1	15 May 2006
		KR 10-2004-0015229 A	18 February 2004
		US 2004-0237982 A1	02 December 2004
		US 2005-0126582 A2	16 June 2005
		WO 02-087366 A3	30 October 2003
KR 10-2020-0047672 A	07 May 2020	CN 111201342 A	26 May 2020
		EP 3679182 A1	15 July 2020
		JP 2020-533496 A	19 November 2020
		US 2019-0075842 A1	14 March 2019
		WO 2019-049086 A1	14 March 2019
KR 10-2016-0081726 A	08 July 2016	CN 107109707 A	29 August 2017
		CN 107109707 B	07 April 2020
		EP 3241452 A1	08 November 2017
		JP 2018-504897 A	22 February 2018
		JP 6535742 B2	26 June 2019
		KR 10-2211186 B1	03 February 2021
		US 10617146 B2	14 April 2020
		US 2018-0007952 A1	11 January 2018
		WO 2016-108508 A1	07 July 2016

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REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- KR 1020210129846 [0001]
- WO 1020220118769 A [0001]