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(54) **PREPARATION METHOD FOR LONG CONTINUOUS ELECTROSPUN POLYACRYLONITRILE NANOFIBER YARN, AND APPLICATION THEREOF**

(57) A method a for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps: a. dissolving a polyacrylonitrile raw material in a polar solvent under mechanical stirring to obtain a uniform spinning solution; b. making the polyacrylonitrile solution in the step a into a polyacrylonitrile nonwoven fabric in an electrospinning machine; c. cutting the nonwoven fabric in the step b into slender strips with

a width of 0.5 to 6 cm; d. drafting the slender strips in the step c in a water bath at 80-95°C with a draft ratio of 2 to 5 times, then drafting in air at 110 to 150°C with a draft ratio of 3 to 10 times to obtain a fiber bundle with highly oriented internal fibers; e. twisting the fiber bundle in the step d to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers with a length of not less than 2000 meters.

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Description**Technical Field**

[0001] The present invention relates to the field of continuous filament yarn of polymer fibers, in particular to a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers and uses thereof.

Background

[0002] Polyacrylonitrile fiber, commonly known as acrylic fiber or artificial wool, is a commonly used artificial fiber that can be used to replace or blended with wool to make wool fabrics. It has a good effect of warmth retention, and it also has excellent weather resistance and light resistance, after one year of open-air exposure, the intensity only decreases by 20%, so it is often used to make curtains, tarpaulins, etc.

[0003] Electrostatic spinning is a special fiber manufacturing process, and has been used more and more in the past ten years to prepare nanofiber materials. Its working method is as follows: the polymer solution or the melt is jetted out in a high-voltage electric field, solidified in a running distance, and finally the spinning is received by a receiving device. Due to the process of electrostatic spinning is simple, and various forms of fibers can also be produced according to demand, such as solid fibers, hollow fibers, core-shell structure fibers, etc., it has broad prospects in many fields.

[0004] However, the current electrostatic spinning technology is only used to manufacture a non-woven fabric or spray a thin layer of nano-cobweb on industrial non-woven fabrics (generally with an areal density of about 1 g/m²), which can also manufacture discontinuous and higher linear density of thick yarn, and there is no technology in the world that can continuously manufacture ultra-small linear density or ultra-high count yarn of electrospun nanofibers. The linear density of conventional fiber yarns is above 6 Tex, and the counts of the yarns are generally less than 150 counts, and most of them are below 100 counts.

Summary

[0005] To solve the above technical problems, the first aspect of the present invention provides a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

- a. dissolving a polyacrylonitrile raw material in a polar solvent under mechanical stirring to obtain a uniform spinning solution;
- b. making the polyacrylonitrile solution in the step a into a polyacrylonitrile nonwoven fabric in an electrospinning machine;
- c. cutting the nonwoven fabric in the step b into slender strips with a width of 0.5 to 6 cm;
- d. drafting the slender strips in the step c in a water bath at 80 to 95°C with a draft ratio of 2 to 5 times, then drafting in air at 110 to 150°C with a draft ratio of 3 to 10 times to obtain a fiber bundle with highly oriented internal fibers;
- e. twisting the fiber bundle in the step d to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers with a length of not less than 2000 meters.

[0006] According to a preferred embodiment, the polar solvent in the step a is one or more selected from the group consisting of N, N-dimethylformamide, N-methylpyrrolidone, dimethyl sulfoxide, and N, N-dimethylacetamide.

[0007] According to another preferred embodiment, the water bath draft and air draft in the step d are 5-roll drafts.

[0008] According to another yet preferred embodiment, the unwinding speed of the water bath draft in the step d is 2 to 8 m/min.

[0009] According to another yet preferred embodiment, the unwinding speed of the air draft in the step d is 3 to 8 m/min.

[0010] According to another yet preferred embodiment, the fiber orientation degree of the fiber bundle in the step d is 90% to 95%.

[0011] According to another yet preferred embodiment, the unwinding speed of twisting in the step e is 5 to 50 m/min.

[0012] According to another yet preferred embodiment, the twist degree of twisting in the step e is 500 to 1500 twist/m.

[0013] The second aspect of the present invention provides a continuous filament yarn of electrospun polyacrylonitrile nanofibers, which is prepared by the above method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

[0014] The third aspect of the present invention provides a use of the continuous filament yarn of electrospun polyacrylonitrile nanofibers, that is, for pure or blended spinning to weave a light-weight and warm high-grade fabric.

[0015] Beneficial effects: the present invention provides a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, which enhance the mechanical properties of polyacrylonitrile fibers through draft operation and can produce continuously, and the produced filament yarns have a length of not less than 2000 meters and

a metric count of more than 500, which can be used for pure or blended spinning to obtain a light-weight, warm and durable high-grade fabric.

Detailed Description of The Embodiments

[0016] The content of the present invention can be further understood in conjunction with the following detailed description of the preferred implementation methods of the present invention and the included embodiments. Unless otherwise stated, all the technical and scientific terms used herein have the same meaning as commonly understood by those of ordinary skill in the art to which the application belongs. If the definition of a specific term disclosed in the prior art is inconsistent with any definition provided in this application, the definition of the term provided in this application shall prevail.

[0017] As used herein, unless the context clearly indicates otherwise, features that do not define singular and plural forms are also intended to include features of the plural form. It should also be understood that, as used herein, the term "prepared from" is synonymous with "comprise", "include", "including", "having", "comprise" and/or "comprising", when used in this specification, they mean the stated composition, step, method, article or device, but do not exclude the presence or addition of one or more other compositions, steps, methods, articles or devices. In addition, when describing the embodiments of the present application, the use of "preferred", "preferably", "more preferably", etc. refers to an embodiment of the present invention that can provide certain beneficial effects under certain circumstances. However, other embodiments may also be preferred under the same or other circumstances. In addition, the expression of one or more preferred embodiments does not imply that other embodiments are not available, nor is it intended to exclude other embodiments from the scope of the present invention.

[0018] In order to solve the above technical problems, the present invention provides a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

- a. dissolving a polyacrylonitrile raw material in a polar solvent under mechanical stirring to obtain a uniform spinning solution;
- b. making the polyacrylonitrile solution in the step a into a polyacrylonitrile nonwoven fabric in an electrospinning machine;
- c. cutting the nonwoven fabric in the step b into slender strips with a width of 0.5 to 6 cm;
- d. drafting the slender strips in the step c in a water bath at 80 to 95°C with a draft ratio of 2 to 5 times, then drafting in air at 110 to 150°C with a draft ratio of 3 to 10 times to obtain a fiber bundle with highly oriented internal fibers;
- e. twisting the fiber bundle in the step d to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers with a length of not less than 2000 meters.

Step a

[0019] The objective of step a is to prepare a polymer solution suitable for electrostatic spinning, and because there are a large number of polar cyano groups in polyacrylonitrile, selecting a suitable polar solvent helps to prepare a spinning solution with suitable concentration and adjustable viscosity.

[0020] Step a. dissolving a polyacrylonitrile raw material in a polar solvent under mechanical stirring to obtain a uniform spinning solution.

[0021] In some preferred embodiments, the polyacrylonitrile raw material and the polar solvent are added together in a stainless steel reactor and dissolved under mechanical stirring to obtain a uniform polyacrylonitrile solution for spinning.

[0022] The polyacrylonitrile raw material in the present invention is not particularly limited, and may be commercially available, and the CAS number is 25014-41-9.

[0023] In some embodiments, the polar solvent in step a is selected from one or more of N, N-dimethylformamide (CAS number: 68-12-2), N-methylpyrrolidone (CAS number: 872-50-4), dimethyl sulfoxide (CAS No. 67-68-5) and N, N-dimethylacetamide (CAS No. 127-19-5).

[0024] Both dimethyl sulfoxide and N-methylpyrrolidone are good solvents for dissolving polyacrylonitrile, and dimethyl sulfoxide is non-toxic, but both have boiling points above 200°C, and the spun yarns are not easy to dry and have serious adhesion to each other. N, N-dimethylformamide has the lowest boiling point among several listed solvents and has the best solubility, while N, N-dimethylacetamide has relatively low solubility, but has the advantage of low toxicity which can be compounded with N, N-dimethylformamide to make a mixed solvent for spinning.

[0025] In some preferred embodiments, the polar solvent in the step a is N, N-dimethylformamide and/or N, N-dimethylacetamide; further preferably, the N, N-Dimethylformamide and N, N-dimethylacetamide; further, the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide is 4: 1.

[0026] The concentration of the spinning solution determines the viscosity of the solution, if the viscosity is too large, the electrostatic force needs to overcome a greater surface tension, making the spinning diameter too large or even

impossible to spin; and too small viscosity will make the spinning too thin, insufficient strength, or beads may appear on the spinning. In some preferred embodiments, the mass concentration of the solution in the step a is 12 to 22%; further preferably, the mass concentration of the solution in the step a is 13 to 18%.

[0027] In some preferred embodiments, the absolute viscosity of the solution in the step a is 1.5 to 5 Pa.S; further preferably, the absolute viscosity of the solution in the step a is 2 to 4 Pa.S.

[0028] On the one hand, appropriate dissolution temperature can speed up the dissolution rate and improve the processing efficiency, on the other hand can reduce the solubility of the gas in the solution, and remove the gas from the solution. In some preferred embodiments, the dissolution temperature in the step a is 30 to 55°C. and the stirring time is 4 to 10 hours; further preferably, the dissolution temperature in the step a is 38 to 48°C. and the stirring time is 6 to 9 hours.

Step b

[0029] The objective of the step b is to make polyacrylonitrile from solution into a nonwoven fabric, in this process, the polymer solution is sprayed into a strong electric field. Under the action of the electric field, the sprayed droplets change from spherical to Taylor cone, from the tip of which a tiny jet is extended, and after running for a certain distance, the jet is solidified into fiber filaments, which are collected by a stainless steel mesh belt to obtain a nonwoven fabric.

[0030] Step b. making the polyacrylonitrile solution in the step a into a polyacrylonitrile nonwoven fabric in an electro-spinning machine.

[0031] In some preferred embodiments, the polyacrylonitrile solution is injected into the spinning device of an electro-spinning machine, spraying spun in a high-voltage electric field, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric.

[0032] The magnitude of the voltage of the electric field will affect the shape of the sprayed droplets. Too small voltage can not make the spherical droplets of the spinneret form a Taylor cone, and too large voltage will cause the formed Taylor cone to retreat or even back into the spinneret, resulting a large number of beads appeared in the spinning fibers. In some preferred embodiments, the DC voltage of the high-voltage electric field in the step b is 30 to 50 kV; further preferably, the DC voltage of the high-voltage electric field in the step b is 40 to 45 kV.

[0033] The distance between the spinneret and the stainless steel mesh belt collector requires to ensure that the jet can be solidified during running without adhesion, and improper receiving distance will cause beads appeared in the spinning fibers. In some preferred embodiments, the distance between the spinneret and the stainless steel mesh belt collector in the step b is 25 to 55 cm; further preferably, the distance between the spinneret and the stainless steel mesh belt collector is 28 to 35 cm.

[0034] The travel speed of the stainless steel mesh belt can affect the pore size and thickness of the nonwoven fabric, which in turn affects the strength of the filament yarn processed from the nonwoven fabric. In some preferred embodiments, the travel speed of the stainless steel mesh belt in the step b is 1 to 5 m/min; further preferably, the travel speed of the stainless steel mesh belt is 2 to 4 m/min.

[0035] In some preferred embodiments, the diameter of the spinning in step b is 100-1500 nm; further preferably, the diameter of the spinning in the step b is 100 to 500 nm.

Step c

[0036] The objective of the step c is to pretreat the polyacrylonitrile nonwoven fabric into a form suitable for further processing.

[0037] Step c: cutting the nonwoven fabric in the step b into slender strips with a width of 0.5 to 6 cm.

[0038] The width of the cut strip will affect the subsequent further processing, and too narrow cut strip is not conducive to continuous production, making the resulting yarn easily broken, and it is impossible to achieve the ideal length to obtain a filament yarn; while too wide cut strip is difficult to process to obtain a fiber bundle with highly oriented internal fibers. In some preferred embodiments, the width of the slender strip in the step c is 0.5 to 6 cm; further preferably, the width of the slender strip is 2 to 5.5 cm.

Step d

[0039] The objective of the step d is, on the one hand, to obtain a fiber bundle length sufficient to produce continuous filament yarns through drafting, and on the other hand, to change the degree of orientation of the internal fibers, making the strength of the fiber bundle in the orientation direction increased greatly.

[0040] Step d: drafting the slender strips in the step c in a water bath at 80 to 95°C with a draft ratio of 2 to 5 times, then drafting in air at 110 to 150°C with a draft ratio of 3 to 10 times to obtain a fiber bundle with highly oriented internal fibers.

[0041] In some preferred embodiments, the water bath draft and air draft in the step d are 5-roll drafts.

[0042] In some preferred embodiments, the unwinding speed of the water bath draft is 2 to 8 m/min; further preferably, the unwinding speed of the water bath draft is 4 to 7 m/min.

[0043] In some preferred embodiments, the unwinding speed of the air draft is 3 to 8 m/min; further preferably, the unwinding speed of the air draft is 4 to 7 m/min.

[0044] In some preferred embodiments, the fiber orientation degree of the fiber bundle in the step d is 90% to 95%.

Step e

[0045] The objective of the step e is to twist the fiber bundle into a filament yarn. After twisting, the outer fiber and the inner fiber squeeze each other to generate pressure, making the yarn obtain frictional force along the fiber length, and the fiber strip is longitudinally fixed, and the fiber after yarn formation has improved properties such as strength, elongation, gloss, and feel.

[0046] Step e: twisting the fiber bundle in the step d to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers with a length of not less than 2000 meters.

[0047] In some preferred embodiments, the unwinding speed of twisting in the step e is 5 to 50 m/min; further preferably, the unwinding speed of twisting is 20 to 40 m/min.

[0048] In some preferred embodiments, the twist degree of twisting in the step e is 500 to 1500 twist/m; further preferably, the twist degree of twisting in the step e is 800 to 1200 twist/m.

[0049] Compared with other fibers, the strength of polyacrylonitrile nanofibers is lower. In terms of the original excellent properties of polyacrylonitrile such as weather resistance and warmth retention, the strength of polyacrylonitrile fiber after high drafting is further improved, which broadens its range of application. After the fiber is drafted and twisted, filament yarn can be produced continuously, which in turn can be used for pure or blended with other fibers to weave alight-weight, warm, soft, and comfortable high-grade fabric.

Examples

[0050] The technical solutions of the present invention will be described in detail below through the examples, but the protection scope of the present invention is not limited to the examples.

Example 1

[0051] Example 1 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric. The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 2

[0052] Example 2 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and N, N-dimethylformamide in a stainless steel reaction reactor under mechanically stirring, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric. The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 3

[0053] Example 3 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 4

[0054] Example 4 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 2 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 5

[0055] Example 5 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4:1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 5.5 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 6

[0056] Example 6 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 5 times to obtain a fiber bundle of electrospun polyacrylonitrile;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 7

[0057] Example 7 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 2 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 8

[0058] Example 8 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 88°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 5 times, then 5-roll drafting in air at 135°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 10 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 9

[0059] Example 9 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 80°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 3 times, then 5-roll drafting in air at 110°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers;

twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Example 10

[0060] Example 10 provided a method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, comprising the following steps:

dissolving a polyacrylonitrile raw material and a mixed solvent composed of N, N-dimethylformamide and N, N-dimethylacetamide in a stainless steel reaction reactor under mechanically stirring, and the mass ratio of N, N-dimethylformamide and N, N-dimethylacetamide was 4: 1, the dissolution temperature was 43°C., the stirring time was 8 hours. and the mass concentration of the obtained polyacrylonitrile solution for spinning was 15%;

injecting the solution obtained in the step a into the spinning device of an electrostatic spinning machine, spraying spun in a high-voltage electric field with a DC voltage of 42 kV, and collected with a stainless steel mesh belt to obtain an electrospun polyacrylonitrile nonwoven fabric, The distance between the spinneret and the stainless steel mesh belt collector was 33 cm, the travel speed of the stainless steel mesh belt was 3 m/min, and the diameter of the spinning was 150 nm;

cutting the nonwoven fabric in the step b into slender strips with a width of 4 cm;

5-roll drafting the slender strips in the step c in a water bath at 95°C with an unwinding speed of twisting of 6 m/min

and a draft ratio of 3 times, then 5-roll drafting in air at 150°C with an unwinding speed of twisting of 6 m/min and a draft ratio of 6 times to obtain a fiber bundle of electrospun polyacrylonitrile with highly oriented internal fibers; twisting the fiber bundle in the step d to more than 2000 meters, the unwinding speed was 25 m/min, and the twist degree was 900 twist/m to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers.

Performance evaluation

[0061] The continuous filament yarns of electrospun polyacrylonitrile nanofibers obtained in examples 1 to 10 were tested for metric count, tensile strength, Young's modulus, and elongation at break.

[0062] Metric count: 1000 meters of yarn was weighed its gram weight, and the metric count = 1000/gram weight. The results were shown in Table 1.

[0063] Tensile strength, Young's modulus, elongation at break: tested with an electronic universal tensile machine. The results were shown in Table 1.

Table 1

	Metric count	Tensile strength	Young's modulus	Elongation at break
Example 1	740	15 cN/dtex	298 cN/dtex	11%
Example 2	720	14.5 cN/dtex	290 cN/dtex	12%
Example 3	660	12 cN/dtex	246 cN/dtex	19%
Example 4	760	3 cN/dtex	84 cN/dtex	12%
Example 5	700	13 cN/dtex	260 cN/dtex	14%
Example 6	450	6 cN/dtex	108 cN/dtex	32%
Example 7	520	11.5 cN/dtex	223 cN/dtex	27%
Example 8	800	8 cN/dtex	130 cN/dtex	10%
Example 9	630	10 cN/dtex	194 cN/dtex	22%
Example 10	600	9.5 cN/dtex	172 cN/dtex	17%

[0064] It can be known from the comparison of examples 1 to 10 that the method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers can produce a high count filament yarn with a length of more than 2000 meters, while improving the mechanical properties of the polyacrylonitrile fiber.

Finally, it is pointed out that the foregoing is only preferred exemplary embodiments of the present invention and is not intended to be limiting of the present invention, and any modifications, equivalent substitutions, improvements and the like within the spirit and principles of the present invention are intended to be embraced by the protection range of the present invention.

Claims

1. A method for preparing a continuous filament yarn of electrospun polyacrylonitrile nanofibers, the method comprising the following steps:

- dissolving a polyacrylonitrile raw material in a polar solvent under mechanical stirring to obtain a uniform spinning solution;
- making the polyacrylonitrile solution in the step a into a polyacrylonitrile nonwoven fabric in an electrospinning machine;
- cutting the nonwoven fabric in the step b into slender strips with a width of 0.5 to 6 cm;
- drafting the slender strips in the step c in a water bath at 80 to 95°C with a draft ratio of 2 to 5 times, then drafting in air at 110 to 150°C with a draft ratio of 3 to 10 times to obtain a fiber bundle with highly oriented internal fibers;
- twisting the fiber bundle in the step d to obtain a continuous filament yarn of electrospun polyacrylonitrile nanofibers with a length of not less than 2000 meters.

2. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** the polar solvent in the step a is selected from one or more of N, N-dimethylformamide, N-methylpyrrolidone, dimethyl sulfoxide, N, N-dimethylacetamide.
- 5 3. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** the water bath draft and the air draft in the step d are 5-roll drafts.
4. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** the unwinding speed of the water bath draft in the step d is 2 to 8 m/min.
- 10 5. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** an unwinding speed of the air draft in the step d is 3 to 8 m/min.
6. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** the fiber orientation degree of the fiber bundle in the step d is 90% to 95%.
- 15 7. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** the unwinding speed of twisting in the step e is 5 to 50 m/min.
8. The method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 1, **characterized in that** the twist degree of twisting in the step e is 500 to 1500 twist/m.
- 20 9. A continuous filament yarn of electrospun polyacrylonitrile nanofibers, which is prepared by the method for preparing the continuous filament yarn of electrospun polyacrylonitrile nanofibers according to any one of claims 1 to 8.
- 25 10. The continuous filament yarn of electrospun polyacrylonitrile nanofibers according to claim 9, which can be used for pure or blended spinning to weave a light-weight and warm high-grade fabric.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2019/106959

A. CLASSIFICATION OF SUBJECT MATTER

D02G 3/02(2006.01)i; D02G 3/26(2006.01)i; D04H 3/033(2012.01)i; D04H 3/007(2012.01)i; D01D 5/42(2006.01)i; D01D 10/02(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)

D02G;D04H;D01D

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

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

CNPAT, CNKI, WPI, EPODOC, 聚丙烯腈, 电纺, 纳米, 纤维, 非织, 无纺, 不织, 切, 割, 剪, 裁, 牵伸, 拉伸, 水浴, 空气, 蒸汽, 蒸气, 热, 加捻, 取向, PAN, polyacrylonitrile, fiber, fibre, electro+, electrospinning, electrospun, twist+, draw+, draft+, stretch+, cut+

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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Y	CN 107858780 A (BEIHANG UNIVERSITY) 30 March 2018 (2018-03-30) description, paragraphs 0005-0013	1-10
Y	CN 103132194 A (YANG, Enlong et al.) 05 June 2013 (2013-06-05) claims 3, 9	1-10
A	US 2016298265 A1 (RANDOLPH V.LEWIS et al.) 13 October 2016 (2016-10-13) claims 1-12, figure 1	1-10
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A	CN 107503000 A (DONGHUA UNIVERSITY) 22 December 2017 (2017-12-22) claims 1-9	1-10

☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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INTERNATIONAL SEARCH REPORT

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PX	CN 110079903 A (JIANGXI XIAN CAI NANOFIBERS TECHNOLOGY CO., LTD.) 02 August 2019 (2019-08-02) claims 1-10	1-10
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Form PCT/ISA/210 (second sheet) (January 2015)

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Information on patent family members

International application No.

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- CHEMICAL ABSTRACTS, 872-50-4 [0023]
- CHEMICAL ABSTRACTS, 67-68-5 [0023]
- CHEMICAL ABSTRACTS, 127-19-5 [0023]