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3,461,082 METHOD FOR PRODUCING CARBONIZED LIGNIN FIBER

Sugio Otani, Kiryu-shi, and Yoshihiko Fukuoka, Bunjiro Igarashi, and Kesao Sasaki, Takasaki-shi, Japan, assignors to Nippon Kayaku Kabushiki Kaisha, Tokyo, Japan, a corporation of Japan

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ABSTRACT OF THE DISCLOSURE

A method is provided for producing carbonized fiber. A fiber produced from lignin obtained by the chemical treatment of woody material is subjected to carbonizing treatment.

This invention relates to a method for producing carbonized fiber by carbonizing fiber produced from lignin which is obtained by the chemical treatment of woody material of softwood, hardwood, vegetation, etc.

The production of carbonized fiber by the carbonization of cellulose fiber or acrylonitrile fiber while retaining its original form has conventionally been carried out. However, such production has a disadvantage in that the cost of producing carbonized fiber is high due to the use of 30 expensive raw materials.

Lignin employed as a raw material according to this invention is recovered as a byproduct in the paper-manufacturing industry and is available as a very low-priced material. However, this has had no practical uses. 35 It has been found that a fiber can be produced from lignin, and that by carbonizing the thus produced fiber, carbonized fiber is obtained. The carbonized fiber obtained according to this invention has the features that it is produced at low cost and, when activated, has a 40 much higher adsorptive power in comparison with conventional fibers.

This invention relates to a method for producing carbonized fiber which comprises heating fiber (referred to as "lignin fiber" hereinafter) produced from alkali-lignin, 45 thiolignin or ligninsulfonate which is produced by the chemical treatment of woody material, at from 400° C. to a temperature at which substantial graphitization takes place.

Lignin fiber used as a raw material may be in the form of a continuous monofilament, short-length or staple fiber, yarn or woven webs, or in any other suitable fiber forms.

The conventional methods for spinning a high molecular weight material, that is, the melt spinning method, the dry spinning method and the wet spinning method can be used in order to produce lignin fiber from alkali-lignin, thiolignin or ligninsulfonate obtained by the chemical treatment of woody material.

When the melt spinning method is employed to produce lignin fiber, alkali-lignin or thiolignin is charged into the melting apparatus and made molten by rapid heating to a suitable temperature between 100° C. and 400° C., preferably between 150° C. and 200° C. The melt is spun while passing inert gas such as N₂ or CO₂ to the surface of the melt, if necessary. For spinning, one of the following methods may be employed, namely, the method for producing filaments by continuously spinning from a small nozzle, the method for producing short-length fibers by 70 passing the molten lignin through a blower of air or inert gas, and the method for producing short-length fibers by

2

the dropping of the melt on a turning disk. When filament is produced, the raw material should be purified before this is charged into the melting apparatus to remove the foreign matters included and to prevent the snapping of yarn. It is desirable for preventing the change of the property of the melt due to the oxidation during the melting procedure to cover the surface of the melt with an inert gas.

When lignin fiber is produced by the dry spinning method, alkali-lignin, thiolignin or ligninsulfonate is dissolved in a suitable solvent capable of dissolving it, for example, water, alkaline solution of sodium hydroxide, potassium hydroxide, etc., acid solution of sulfuric acid, hydrochloric acid, etc., dimethyl sulfoxide, glycerine, etc.

This solution is extruded from a small nozzle and then dried at a suitable temperature to obtain lignin fiber. When alkali-lignin or thiolignin is used, it is necessary to employ a solvent other than water and acid solution mentioned above because said two lignins are insoluble in water and the acid solution. On the other hand, when ligninsulfonate is used, any of said solvents may be employed. The suitable amount of each solvent per 100 parts of lignin is as follows:

	Parts
Acid solution (concentration less than 20%)	50-150
Alkaline solution (concentration 5-20%)	10-200
Water	30-100
Dimethyl sulfoxide	100-200
Glycerine	10–20

In producing lignin fiber by dissolving lignin in a solvent, the addition of polyvinyl alcohol, polyacrylonitrile or viscose soluble in said solvents in large amounts renders the lignin fiber stronger. However, carbonized fiber produced by carbonizing lignin fiber which is produced with the addition of large amounts of a high molecular weight material is weaker in its strength than that produced from lignin alone. Therefore, the addition of a larger amount of the high molecular weight material is not desirable. Accordingly, the amount should be not more than the amount of lignin.

When lignin fiber is produced by the wet spinning method, a mixture of a suitable solvent in which alkalilignin, thiolignin or ligninsulfonate is dissolved and a solvent in which an appropriate amount of viscose is dissolved is spun into a non-solvent and is desolvented to produce lignin fiber.

Thus obtained lignin fiber is weak in its strength and is not of practical use. However, when carbonized, this lignin fiber yields carbonized fiber having a practical strength. Such characteristic is not found in the conventional cellulose fiber or acrylonitrile fiber whose strength is decreased by the carbonization.

The production of carbonized fiber is effected as follows: Lignin fiber is pretreated in an oxidizing atmosphere such as air or ozone at 50° C.-400° C., or in a closed vessel at 100° C.-400° C., and then treated in an inert gas at a temperature increasing at the rate of less than 50° C./minute. Thus a flame resistant fiber is produced at about 400° C., which becomes carbon fiber, having a practical strength, at a temperature higher than 700° C. This carbon fiber becomes graphite fiber when subjected to a graphitizing treatment at a temperature higher than 2000° C.

In case the carbonized fiber of lignin is produced as mentioned above by using fiber produced from alkalilignin, thiolignin or ligninsulfonate without adding any high molecular weight material, the pretreatment in air or ozone at 50° C.—400° C. and subsequent carbonization in an inert gas result in a stronger carbonized fiber than the pretreatment in a closed vessel at 100° C.—400° C.

3

On the other hand, when carbonized fiber is produced with the addition of polyvinyl alcohol, polyacrylonitrile or viscose to alkali-lignin, thiolignin or ligninsulfonate, a stronger carbonized fiber is obtained by effecting pretreatment in a closed vessel of 100° C.-400° C. and subsequent carbonization in an inert gas than by pretreatment in air or ozone at 50° C.-400° C.

One of the features of this invention is that highly activated carbonized fiber is produced; for this purpose one has only to use the activating processes by chemicals or gases alone or in combination. Thus, the activating process by chemicals can be applied to various methods for producing lignin fiber. When lignin fiber is produced from alkali-lignin or thiolignin by the melt spinning method, sulfur, zinc chloride, etc. is incorporated as an 15 activating agent in the molten lignin in an amount less than 30 parts per 100 parts of lignin. Then the melt is spun while covering the surface of the melt with an inert gas such as N₂ or argon, if necessary. Thus obtained lignin fiber is pretreated as mentioned above and carbonized at a temperature of 600° C.-1000° C. at a rate of less than 50° C./min.

Regarding the dry spinning methods and the wet spinning, an alkaline activating agent such as sodium hydroxide, potassium hydroxide, etc. which are useful as 25 an activating agent or an acid activating agent such as sulfuric acid may be used in the solvent in which lignin is dissolved.

The activating process by gases is useful for producing carbonized fibers which should be activated. Thus, in 30 carbonizing lignin fiber containing no high molecular weight material, the fiber is pretreated by heating it in air or ozone to 50° C.-400° C., then heated to 600° C.-1000° C. at a rate of less than 50° C./min. in a closed vessel or an inert gas, and furthermore an activating gas 35 such as air, oxygen, steam, etc. is blown thereinto in an amount of 100-2000 cc. per gram of lignin fiber, whereby activated carbonized fiber is produced. In carbonizing lignin fiber produced with the addition of polyvinyl alcohol, polyacrylonitrile or viscose, activated 40 carbonized fiber is obtained by the same method, that is, pretreating lignin fiber, heating it to 600° C.–1000° C. in a closed vessel, and blowing an activating gas thereinto, or by a method comprising heating lignin fiber to 600° C.-1000° C. at a rate of less than 50° C./min. in a closed vessel without effecting the pretreatment, and blowing thereinto an activating gas such as air, oxygen, steam, etc. in an amount of 100-2000 cc. per gram of the fiber. However, the direct activation by the activating gas without the pretreatment is preferable because this yields the 50stronger activated carbonized fiber.

The carbonized fiber according to this invention has chemical resistance, heat resistance, electrical conductivity and textile flexibility. Therefore, this fiber has various uses such as filtrating material for acid and alkali, material for removing dusts from high temperature gas, filler for plastics, electrical resistance material, electric ribbon, electrode, packing material, etc.

Furthermore, the activated carbonized fiber has a high adsorptive power besides said properties and therefore is useful as gas adsorbent, decoloring agent, etc.

This invention is illustrated by the following examples.

Example 1

Twenty grams of thiolignin produced by the chemical 65 treatment of hardwood was rapidly heated to 170° C. in a steel vessel. CO₂ gas was passed to the surface of the melt. The melt was spun by winding on a bobbin at a rate of 5-10 m./min. through the inert gas layer to produce filamentary lignin fiber of 20-30 μ . This fiber 70 was pretreated by allowing it to stand at 150° C. for 10 hours in air. Thus treated fiber was placed in a porcelain tube. After air was replaced by N₂ gas, the fiber in the tube was heated up to 700° C. at the rate of 5° C./min. in an electric furnace and kept at that tem-75

4

perature for 10 minutes. Thereafter, this was allowed to stand at room temperature to produce 12 g. of carbonized fiber. The fiber showed black color in appearance and had a tensile strength of 8×10^3 kg./cm.² and an electric specific resistance of 10×10^{-3} Ω cm.

Example 2

Thirty grams of a mixture consisting of one part by weight of alkali-lignin produced by the chemical treatment of softwood and one part by weight of alkali-lignin produced by the chemical treatment of hardwood was rapidly heated to 170° C. in a steel vessel. The molten lignin was jetted by means of an air blower to produce a short-lenfth fiber. This fiber was kept at 80° C. for 7 hours in an ozone atmosphere and placed in a porcelain tube. The fiber in the tube was heated up to 100° C. at the rate of 2° C./min. in an electric furnace, and then to 150° C. at the rate of 0.5° C./min. After maintaining the temperature at 150° C. for about 10 minutes, the fiber was transferred to a graphite crucible, which was placed in a Kryptol electric furnace. After air was replaced by N₂ gas, the fiber in the crucible was heated to 2000° C. at the rate of 50° C./min. and then allowed to stand at room temperature, whereby 8 g. of graphite fiber was obtained. This graphite fiber had a strength of 7×10^3 kg./cm.² and an electric specific resistance of $1 \times 10^{-3} \Omega \text{cm}$.

Example 3

A mixture of 100 g. of alkali-lignin produced by chemically treating softwood and 15 g. of sulfur were rapidly heated to 200° C. in a rotary cylinder containing a heating apparatus. The mixture was thus molten. The melt was spun from a hole of 1.0 mm. diameter provided at the cylinder by the centrifugal force to produce lignin fiber. This fiber was pretreated for 10 hours in air of 200° C. Thus treated fiber was placed in a porcelain tube and, after air was replaced by N₂ gas, was heated to 800° C. in an electric furnace. The fiber was kept at that temperature for 1 hour and then allowed to stand at room temperature to produce 40 g. of activated carbonized fiber. This carbonized fiber had a Methylene Blue adsorbtive power of 8 cc. and a tensile strength of 3×10^3 kg./cm.²

When the above process was repeated except that air was blown into the fiber heated to 800° C. while keeping this temperature, 3 g. of activated carbonized fiber was obtained, which had a Methylene Blue adsorbtive power of 15 cc. and a tensile strength of 2.5×10^3 kg./cm.²

The Methylene Blue adsorbtive power is measured as follows: An amount of 0.12% aqueous solution of Methylene Blue is dropped onto 0.1 g. of carbon to decolor the Methylene Blue in five minutes. Such amount (in cc.) of Methylene Blue dropped represents the adsorbtive power.

Example 4

A mixture of 100 g. of thiolignin produced by the chemical treatment of hardwood and 25 g. of powdered zinc chloride was rapidly heated to 400° C. in a rotary cylinder containing a heating apparatus to molten state. The melt was spun by centrifugal force from a hole of 1.0 mm. diameter provided at the rotary cylinder to produce lignin fiber. This fiber was kept at 270° C. for 10 hours in air, then placed in a porcelain tube and, after air was replaced by N₂ gas, heated to 800° C. at the rate of 2° C./min. in an electric furnace and kept at that temperature for 1 hour. This fiber was, thereafter, allowed to stand at room temperature to yield 42 g. of activated carbonized fiber, which had a Methylene Blue adsorbtive power of 10 cc. and a tensile strength of 3×10³ kg./cm.².

Example 5

Thirty grams of a mixture comprising 1 part by weight of thiolignin obtained by the chemical treatment of softwood and 1 part by weight of thiolignin produced by the chemical treatment of hardwood was rapidly heated to 170° C. in a steel vessel. To the surface of molten lignin

5

argon gas was passed. The molten lignin was spun from a nozzle at a rate of 5-10 m./min. and wound on a bobbin. For pretreatment, the resultant fiber was kept at 150° C. for 10 hours in air, then placed in a porcelain tube. The tube was closed and placed in the electric furnace. The fiber in the closed vessel was heated to 900° C. at the rate of 5° C./min. and, after 1500 cc./g. of air was blown thereinto while keeping said temperature, kept at the same temperature for 1 hour and allowed to stand at room temperature. Six grams of carbonized fiber obtained had a Methylene Blue adsorbtive power of 18 cc. and a tensile strength of 2.5×10^3 kg./cm.².

Example 6

Fifty grams of a mixture comprising 100 parts by 15 weight of thiolignin produced by the chemical treatment of softwood and 10 parts by weight of glycerine was heated to 150° C. in a steel vessel, extruded at that temperature under the pressure of 0.5 kg./cm.2 from a nozzle of 1.0 mm. diameter and then wound at the rate of 100 20 m./min. to produce lignin fiber. This lignin fiber was allowed to stand at 80° C. for 10 hours in ozone atmosphere and then heated at 250° C. for 5 hours in air. Furthermore, thus treated lignin fiber was placed in a porcelain tube. After air was replaced by N₂ gas, the fiber in 25 the tube was heated to 1000° C. at the rate of 1° C./min. in the electric furnace and kept at that temperature for 1 hour and thereafter allowed to stand at room temperature to produce 15 g. of carbonized fiber. This fiber had a tensile strength of 5×10^3 kg./cm.² and an electric specific 30 resistance of $8 \times 10^{-3} \Omega \text{cm}$.

Example 7

Thirty parts by weight of calcium ligninsulfonate was dissolved in 15 parts by weight of water. On the other 35 hand, 30 parts by weight of polyvinyl alcohol was dissolved in 100 parts by weight of water with heating. The two were mixed at a temperature of 80°-85° C. One hundred grams of the mixture thus obtained was extruded from a nozzle of 0.3 mm. diameter at 70° C. under the pressure of 1.0 kg./cm.2 and was wound on a bobbin at the rate of 70 m./min. while drying it. Lignin fiber having 20μ diameter was produced. This fiber was placed in a porcelain tube. The tube was closed and placed in an electric furnace. The fiber in the closed vessel was heated 45 at 200° C. for 10 hours for pretreatment. After N₂ gas was blown thereinto, the fiber was heated to 1500° C. at the rate of 30° C./min. and thereafter allowed to stand at room temperature to produce 35 g. of carbonized fiber which had a tensile strength of 3×10^3 kg./cm.² and an 50 electric specific resistance of $3 \times 10^{-3} \Omega \text{cm}$.

Example 8

Thirty parts by weight of alkali-lignin produced by 55 the chemical treatment of softwood was mixed with and dissolved in 30 parts by weight of 15% aqueous solution of sodium hydroxide by heating the mixture to 80° C. On the other hand, 30 parts by weight of polyvinyl alcohol was dissolved by heating in 110 parts by weight 60 of water. The two were mixed at a temperature higher than 75° C. One hundred grams of the mixture obtained was extruded at 85° C. under the pressure of 1.5 kg./cm.² from a nozzle of 0.2 mm. diameter and then wound on a bobbin at the rate of 600 m./min. while drying it at 65 250° C. Lignin fiber of 15μ diameter was obtained. This fiber was placed in a porcelain tube. The tube was closed and placed in an electric furnace and the fiber was heated at 300° C. for 5 hours in the closed vessel. After N₂ gas was blown thereinto, the fiber was heated to 1500° C. 70 at the rate of 2° C./min. in N₂ atmosphere and then allowed to stand at room temperature to produce 32 g. of carbonized fiber. Thus obtained fiber had a tensile strength of 5×10³ kg./cm.² and an electric specific resistance of $5 \times 10^3 \ \Omega \text{cm}$.

6

Example 9

One hundred grams of fiber produced by following the procedure of Example 8 was placed in a porcelain tube. This tube was closed and placed in the electric furnace. After the fiber in the closed vessel was heated at 200° C. for 10 hours and then N_2 gas was blown thereinto, the fiber was heated to 400° C. and allowed to stand at room temperature. Sixty-five grams of flame proof fiber having a tensile strength of 1.5×10^3 kg./cm.² was obtained.

Example 10

One hundred grams of fiber produced by following the procedure of Example 8 was placed in a porcelain tube. This tube was closed and placed in an electric furnace. The fiber in the closed vessel was heated at 200° C. for 10 hours. The fiber was transferred to a graphite crucible, which was placed in a Kryptol electric furnace. Then N_2 gas was blown thereinto and the fiber was further heated to 2500° C. and allowed to stand at room temperature to produce 18 g. of graphite fiber. This graphite fiber had a tensile strength of 3×10^3 kg./cm.² and an electric specific resistance of 0.8×10^{-3} Ω cm.

Example 11

One hundred parts by weight of alkali-lignin produced by the chemical treatment of hardwood was added to 10 parts by weight of 15% sodium hydroxide solution and the mixture was heated to 80°-90° C. to effect dissolution. One hundred grams of the resultant mixture was extruded from a nozzle of 2.0 mm. diameter and then continuously wound on a bobbin at the spinning rate of 80 m./min. while drying it in a drying cylinder at 200° C. The thus produced fiber was pretreated at 80° C. for 7 hours in an ozone atmosphere and placed in a porcelain tube. After air was replaced by N₂ gas, the fiber in the tube was heated to 900° C. in the electric furnace and kept at that temperature for 1 hour to produce 35 g. of carbonized fiber having a Methylene Blue adsorbtive power of 10 cc. and a tensile strength of 3×10^3 kg./cm.².

Example 12

Thirty parts by weight of thiolignin produced by the chemical treatment of softwood was dissolved in 30 parts by weight of 20% sodium hydroxide solution. On the other hand, 20 parts by weight of polyvinyl alcohol was dissolved in 80 parts by weight of water. The two were mixed while keeping them at 80° C. One hundred grams of the mixture was extruded from a nozzle of 0.3 mm. diameter and, after being dried at 200° C., was wound on a bobbin to produce lignin fiber. This fiber was placed in a porcelain tube. This tube was closed and placed in an electric furnace. The fiber in the closed vessel was heated to 800° C. and kept at that temperature for one hour to produce 45 g. of carbonized fiber having a Methylene Blue adsorbtive power of 10 cc. and a tensile strength of 3×10^3 kg./cm.².

Example 13

One hundred grams of lignin fiber produced by following the procedure of Example 12 was heated at 200° C. for 10 hours in air and was placed in a porcelain tube. After air was replaced by N_2 gas, the fiber in the tube was heated to 800° C. in an electric furnace and kept at that temperature for 1 hour. Then N_2 gas was replaced by air and the fiber was treated for 15 minutes in air to produce 34 g. of carbonized fiber having a Methylene Blue adsorbtive power of 25 cc. and a tensile strength of 2×10^3 kg./cm.².

Example 14

Thirty parts by weight of sodium ligninsulfonate was dissolved in 15 parts by weight of 15% potassium hydroxide. On the other hand, 30 parts by weight of polyvinyl alcohol was dissolved in 100 parts by weight of water. The two were mixed. One hundred grams of the mixture was extruded from a nozzle of 0.2 mm. diameter

and, after being dried at 200° C., wound on a bobbin. In this manner, lignin fiber was obtained. This fiber was heated for 10 hours in air at 200° C. and placed in an electric furnace. The fiber in the closed vessel was heated to 900° C. and kept at that temperature for 1 hour to produce 38 g. of carbonized fiber having a Methylene Blue adsorptive power of 12 cc. and a tensile strength of 3×10^3 kg./cm.².

Example 15

Thirty parts by weight of calcium ligninsulfonate produced by the chemical treatment of hardwood was dissolved in 40 parts by weight of 20% sulfuric acid solution. On the other hand, 30 parts by weight of polyvinyl alcohol was dissolved in 100 parts by weight of water. The two were mixed. One hundred grams of the mixture was extruded from a nozzle of 0.2 mm. diameter, then dried at 250° C. and thereafter wound on a bobbin to produce lignin fiber. This fiber was pretreated at 200° C. for 10 hours in air and placed in a porcelain tube. After air was replaced by N₂ gas, the fiber in the tube was heated to 1000° C. in an electric furnace to produce 32 g. of carbonized fiber having a Methylene Blue adsorbtive power of 8 cc. and a tensile strength of 3×10^3 kg./cm.².

Example 16

One hundred parts by weight of alkali-cellulose was dissolved in 30 parts by weight of carbon disulfide. On the other hand, 200 parts by weight of thiolignin produced 30 by the chemical treatment of softwood was dissolved in 400 parts by weight of 15% sodium hydroxide solution. The two were mixed. One hundred grams of this mixture was extruded from a nozzle of 0.1 mm. diameter into hydrochloric acid solution while preventing gelation. The extruded mixture was solidified, spun at the spinning rate of 10 m./min. and dried. The resultant fiber was placed in a porcelain tube. The tube was closed and placed in an electric furnace and the fiber in the closed vessel was heated to 320° C. After N₂ gas was blown thereinto, the 40 fiber was heated to 1000° C. in N₂ atmosphere to obtain 42 g. of carbonized fiber having a tensile strength of 3×10³ kg./cm.² and an electric specific resistance of $8\times10^{-3} \Omega \text{cm}$.

Example 17

One hundred parts by weight of alkali-cellulose was dissolved in 30 parts by weight of carbon disulfide. On the other hand, 100 parts by weight of alkali-lignin produced by the chemical treatment of hardwood was dissolved in 200 parts by weight of 10% sodium hydroxide 50 solution. The two were mixed. One hundred grams of the mixture was extruded from a nozzle of 0.15 mm. diameter into air of 400° C.-450° C. while preventing the gelation and spun at a spinning rate of 50-100 m./ min. This fiber was placed in a porcelain tube and the 55 tube was closed and placed in an electric furnace. The fiber in the closed vessel was heated to 800° C. and at that temperature 500 cc./g. of air was blown thereinto. Thereafter, the fiber was allowed to stand at room temperature to produce 20 g. of carbonized fiber having a 60 Methylene Blue adsorptive power of 23 cc. and a tensile strength of 2×10^3 kg./cm.².

Example 18

Ten parts by weight of polyacrylonitrile was dissolved in 100 parts by weight of dimethyl sulfoxide. On the other hand, 60 parts by weight of thiolignin produced by the chemical treatment of hardwood was dissolved in 100 parts by weight of dimethylsulfoxide. The two were mixed 70 C./min. in a closed vessel, containing an activating gas. at room temperature. One hundred grams of the mixture was extruded from a nozzle of 0.15 mm. into N₂ gas of 350° C. and then wound on a bobbin at the spinning rate of 200 m./min. while drying it. The resultant lignin

placed in a porcelain tube. After air was replaced by N₂ gas, the fiber in the tube was heated to 1500° C. at the rate of 10° C./min. in an electric furnace to produce 32 g. of carbonized fiber having a tensile strength of 5×10^3 kg./cm. and an electric specific resistance of $5\times10^{-3} \Omega \text{cm}$.

What we claim is:

1. A method for producing carbonized fiber which comprises heating a lignin fiber at a temperature of from 400° C. to the temperature at which substantial graphitization of the fiber occurs said lignin fiber being initially produced by dissolving lignin selected from the group consisting of alkali-lignin, thiolignin and lignin-sulfonate in a solvent capable of dissolving said lignin, adding thereto a high molecular weight material selected from the group consisting of polyvinyl alcohol, polyacrylonitrile and viscose, and dry-spinning the mixture.

2. A method according to claim 1, in which the fiber is produced by dissolving lignin selected from the group consisting of alkali-lignin, thiolignin and lignin-sulfonate produced by the chemical treatment of woody material, in a solvent capable of dissolving said lignin, adding thereto a high molecular weight material selected from the group consisting of polyvinyl alcohol, polyacrylo-25 nitrile and viscose, and dry-spinning the resultant mixture.

3. A carbonized fiber produced in accordance with the method of claim 1.

- 4. A method for producing carbonized fiber which comprises heating a lignin fiber at a temperature of from 400° C. to the temperature at which substantial graphitization of the fiber occurs, said lignin fiber being initially produced by dissolving lignin selected from the group consisting of alkali-lignin, thiolignin and lignin sulfonate produced by the chemical treatment of woody material, in a solvent capable of dissolving said lignin, adding thereto viscose and then wet-spinning the resultant mixture.
- 5. A carbonized fiber produced in accordance with the method of claim 4.
- 6. A method for producing carbonized fiber which comprises pretreating a fiber produced from lignin selected from the group consisting of alkali-lignin, thiolignin and ligninsulfonate produced by the chemical treatment of woody material, by heating to a temperature between 50° C. and 400° C. in an oxidizing atmosphere and then heating the thus treated fiber to a temperature between 400° C. and 2500° C. at a rate of less than 50° C./min. in an inert gas.
 - 7. A method for producing carbonized fiber which comprises dissolving lignin selected from the group consisting of alkali-lignin, thiolignin and ligninsulfonate produced by the chemical treatment of woody material, in a solvent capable of dissolving said lignin, adding thereto a high molecular weight material selected from the group consisting of polyvinyl alcohol, polyacrylonitrile and viscose, spinning the mixture, pretreating the thus obtained fiber by heating the fiber to a temperature between 100° C. and 400° C. in an oxidizing atmosphere and then heating the fiber to a temperature between 400° C. and 2500° C. at the rate of less than 50° C./min. in an inert gas.
- 8. A method for producing activated carbonized fiber which comprises pretreating fiber produced from lignin selected from the group consisting of alkali-lignin, thio-65 lignin and ligninsulfonate produced by the chemical treatment of woody material, by heating the fiber to a temperature between 50° C. and 400° C. in an oxidizing atmosphere and, heating the fiber to a temperature between 600° C. and 1000° C. at a rate of less than 50°
 - 9. A method according to claim 8, in which the activating gas is selected from the group consisting of air, oxygen and steam.
- 10. A method for producing activated carbonized fiber was heated to 220° C. in a closed vessel and then 75 fiber which comprises dissolving lignin selected from the

group consisting of alkali-lignin, thiolignin and ligninsulfonate produced by the chemical treatment of woody material, in a solvent capable of dissolving said lignin, adding thereto a high molecular weight material selected from the group consisting of polyvinyl alcohol, polyacrylonitrile and viscose, spinning the mixture and heating the thus obtained fiber to a temperature between 600° C. and 1000° C. at a rate of less than 50° C./min. in a closed vessel containing an activating gas.

11. A method according to claim 10, in which the 10 activating gas is selected from the group consisting of air, oxygen and steam.

10

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