Notice

DESCRIPTION CN120698437A

A method for preparing lignin hard carbon anode materials by Joule thermal flash evaporation

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一种焦耳热闪蒸制备木质素硬碳负极材料的方法

[0001]

Technical Field

技术领域

[n0001]

This invention belongs to the field of anode material preparation technology, specifically relating to a method for preparing lignin hard carbon anode materials by Joule thermal flash evaporation.

本发明属于负极材料制备技术领域，具体涉及一种焦耳热闪蒸制备木质素硬碳负极材料的方法。

[0003]

Background Technology

背景技术

[n0002]

The rechargeable secondary battery industry has experienced explosive growth over the past decade.

在过去的十年里，可充电二次电池行业经历了爆炸式的增长。

The anode of the battery is usually derived from the pyrolysis of a carbon source with an aromatic topology.

电池的阳极通常来源于具有芳香拓扑结构的碳源的热解。

Common precursors include natural graphite, biomass-derived, resin-based, coal-based, and organic polymers.

常见的前体包括天然石墨、生物质衍生、树脂基、煤基和有机聚合物。

Compared to the high cost of organic materials and the non-renewable nature of coal-based materials, biomass materials have significant advantages in battery anode materials due to their abundant energy and low cost.

与有机物的高成本和煤基的不可再生性相比，生物质材料的能源丰富和成本低廉在电池负极材料中发挥了重要优势。

[n0003]

Lignin is the only aromatic polymer in nature with a three-dimensional network structure, and its carbon content is as high as 60%.

木质素作为自然界中唯一具有三维网络结构的芳香族高分子聚合物，且木质素中碳含量高达60%。

In recent years, significant progress has been made in the research on the application of lignin in anode materials. However, due to the heterogeneity of lignin itself and the cohesiveness of its aromatic ring structure, the changes of lignin during heating are complex. During the carbonization process, the carbon layers tend to be ordered and graphitized, which is not conducive to the storage of sodium ions.

近几年关于木质素在负极材料中的应用研究取得了很大的进展，但是由于木质素本身的异质性和其芳香环结构的凝聚性，木质素在加热过程中的变化是复杂的，木质素在成碳过程中，碳层倾向于有序堆积和石墨化，不利于钠离子的储存。

[n0004]

To address the aforementioned problems, one potential solution is to increase the heating rate.

为了解决上述存在的问题，一个潜在的解决措施是提高加热速率。

However, the cost of achieving rapid heating in a high-temperature furnace is enormous. When heated in a high-temperature furnace, the generated small gaseous molecules are easily carried away by the flowing protective gas, thus reducing the total output. In addition, slow annealing may exacerbate the accumulation of carbon layers, and the electrical conductivity of carbon materials obtained by direct pyrolysis is not ideal, which cannot meet the requirements of high-rate charge and discharge.

然而，在高温炉中实现快速加热的成本是巨大的。 当受到高温炉加热时，产生的气态小分子容易被流动的保护气体迅速带走，从而降低了总产量。 此外，缓慢退火可能会加剧碳层的堆积，直接热解的碳材料电导率不理想，不能满足高速率充放电的要求。

[0007]

Summary of the Invention

发明内容

[n0005]

[Technical Issues]

[技术问题]

[n0006]

The present invention aims to address the problem that when preparing anode materials from lignin, the high cost and low total yield are caused by using a high-temperature furnace for rapid heating during the carbonization process of lignin, and the potential for increased carbon layer accumulation during slow annealing. Furthermore, the conductivity of carbon materials obtained by direct pyrolysis is not ideal and cannot meet the requirements of high-rate charge and discharge.

本发明要解决的是以木质素制备负极材料时，木质素在成碳过程中，使用高温炉实现快速加热时成本高、总产量低，且在缓慢退火可能会加剧碳层的堆积，直接热解的碳材料电导率不理想，不能满足高速率充放电的要求。

[n0007]

[Technical Solution]

[技术方案]

[n0008]

To address the aforementioned problems, this invention provides a method for preparing lignin hard carbon anode materials by Joule thermal flash evaporation. This invention utilizes Joule heating to achieve rapid temperature rise, resulting in a fast reaction time and high reaction temperature.

为了解决上述问题，本发明提供了一种焦耳热闪蒸制备木质素硬碳负极材料的方法，本发明利用焦耳加热来实现快速升温，该方法反应时间快，反应温度高。

Under the influence of an electric field, lignin recombine and stacks into vortex carbon layers, forming a three-dimensional continuous carbon network.

在电场的作用下，木质素重组堆叠成涡层碳层，形成三维连续的碳网络。

This ultrafast method can alleviate the carbon layer buildup phenomenon during lignin pyrolysis.

这种超快方法可以缓解木质素热解过程中的碳层堆积现象。

[n0009]

This invention provides a method for preparing lignin hard carbon anode materials by Joule thermal flash evaporation, which mainly includes the following steps:

本发明提供了一种焦耳热闪蒸制备木质素硬碳负极材料的方法，主要包括如下步骤：

[n0010]

(1) React lignin with alkaline solution, then add acid, and wash with water and dry to obtain dried lignin;

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(1)将木质素与碱液反应，再加入酸，经过水洗干燥得到干燥木质素；

[n0011]

(2) The dried lignin obtained in step (1) is preheated in a tube furnace to obtain a hard carbon precursor;

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(2)将步骤(1)所得到的干燥木质素在管式炉中预加热，得到硬碳前驱体；

[n0012]

(3) The hard carbon precursor obtained in step (2) is placed in a graphite mold and carbonized by Joule heat flash evaporation to obtain lignin hard carbon anode material.

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(3)将步骤(2)中所得硬碳前驱体放入石墨模具中，通过焦耳热闪蒸进行碳化，得到木质素硬碳负极材料。

[n0013]

In one embodiment of the present invention, in step (1), the lignin is one or more of alkali lignin, enzymatically hydrolyzed lignin, organic solvent lignin, and sulfate lignin.

在本发明的一种实施方式中，步骤(1)中，所述木质素为碱木质素、酶解木质素、有机溶剂木质素、硫酸盐木质素的一种或多种。

[n0014]

In one embodiment of the present invention, in step (1), the mass concentration of alkali in the alkaline solution is 10-30%, and the alkali is one or more of NaOH and KOH.

在本发明的一种实施方式中，步骤(1)中，碱液中碱的质量浓度为10～30%，碱为NaOH、KOH中的一种或多种。

[n0015]

In one embodiment of the present invention, in step (1), the acid is one or more of HCl and H₂SO₄, and the mass concentration of the acid is 5-20%.

在本发明的一种实施方式中，步骤(1)中，所述酸为HCl、H₂SO₄中的一种或多种，酸的质量浓度为5～20%。

[n0016]

In one embodiment of the present invention, in step (2), the preheating temperature of the tubular furnace is 700-900°C.

在本发明的一种实施方式中，步骤(2)中，管式炉预加热温度为700～900°C。

[n0017]

In one embodiment of the present invention, in step (2), the preheating time of the tubular furnace is 1 to 4 hours; the heating rate is 2 to 10 °C/min.

在本发明的一种实施方式中，步骤(2)中，管式炉预加热的时间为1～4h；升温速率为2～10°C/min。

[n0018]

In one embodiment of the present invention, in step (3), the temperature for carbonization by Joule flash evaporation is 1000-1600°C; the heating rate is 100-140°C/min; and the time is 1-5 min.

在本发明的一种实施方式中，步骤(3)中，焦耳热闪蒸进行碳化的温度为1000～1600°C；加热速度为100～140°C/min，时间为1～5min。

[n0019]

In one embodiment of the present invention, in step (3), the voltage for carbonization by Joule flash evaporation is 3-5V, the current is 300-500A, the output power is 1.5-4.5kW, and the pressure is 20-35MPa.

在本发明的一种实施方式中，步骤(3)中，焦耳热闪蒸进行碳化的电压为3～5v，电流为300～500A，输出功率在1.5-4.5kw，压力在20～35MPa。

[n0020]

In one embodiment of the present invention, in step (3), Joule thermal flash evaporation is performed for carbonization cycles 2 to 6 times.

在本发明的一种实施方式中，步骤(3)中，焦耳热闪蒸进行碳化循环2～6次。

[n0021]

The present invention provides a lignin hard carbon anode material prepared by the method described above.

本发明提供上述所述方法制备得到的木质素硬碳负极材料。

[n0022]

This invention provides the application of the lignin hard carbon anode material described above in sodium-ion batteries.

本发明提供上述所述的木质素硬碳负极材料在钠离子电池中的应用。

[n0023]

[Beneficial Effects]

[有益效果]

[n0024]

(1) This invention utilizes the abundant carbon content on lignin to remove non-carbon components on lignin through Joule flash evaporation, thereby reconstructing and regulating the hard carbon structure of lignin to form advanced carbon materials with high crystallinity and structural order.

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(1)本发明利用木质素上丰富的碳含量，通过焦耳闪蒸方法去除木质素上的非碳成分，重建和调控木质素硬碳结构，形成结晶度和结构有序度强的高级碳材料。

[n0025]

(2) The present invention utilizes the control of the number of Joule heating cycles to control the carbon structure, and the prepared lignin hard carbon anode material exhibits the control of the graphite order of the carbon layer.

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(2)本发明利用对焦耳加热循环次数的调控，对碳结构进行调控，所制备的木质素硬碳负极材料表现为碳层的石墨有序度的调控。

[n0026]

(3) The present invention adjusts the carbon structure by the number of Joule heating cycles, resulting in a short reaction time and increased efficiency.

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(3)本发明通过焦耳加热的循环次数调节碳结构，反应时间短，表现为效率增加。

[n0027]

(4) This technology uses Joule heating to achieve rapid heating. This method has a fast reaction time and a high reaction temperature. Compared with the radiation cooling method of traditional high-temperature furnaces, blackbody radiation can release most of the heat within milliseconds. Under the action of the electric field, lignin is reorganized and stacked into vortex carbon layers, forming a three-dimensional continuous carbon network. This ultrafast method can alleviate the carbon layer accumulation phenomenon in the lignin pyrolysis process. The lignin hard carbon anode material prepared by Joule thermal flash evaporation in this invention can promote its application in energy conversion and open up a new path for the high-value conversion of traditional fossil fuels.

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(4)本技术采用焦耳加热来实现快速升温，该方法反应时间快，反应温度高，与传统高温炉的辐射冷却方式相比，黑体辐射可以在毫秒内释放大部分热量，在电场的作用下，木质素重组堆叠成涡层碳层，形成三维连续的碳网络，这种超快方法可以缓解木质素热解过程中的碳层堆积现象，本发明通过焦耳热闪蒸制备的木质素硬碳负极材料可以促进其在能源转化中的应用，为传统化石燃料的高价值转化开辟了新的道路。

[0031]

Attached Figure Description

附图说明

[n0028]

Figure 1 is a scanning electron microscope image of the lignin hard carbon anode material of Example 1.

图1为实施例1的木质素硬碳负极材料的扫描电镜图。

[0033]

Detailed Implementation

具体实施方式

[n0029]

Material characterization of this invention:

本发明材料表征：

[n0030]

The microstructure of the material was observed using a transmission electron microscope (JEOL, JEM-2100).

采用透射电子显微镜(JEOL,JEM-2100)观察材料的微观结构。

[n0031]

Conductivity was measured using a four-point probe (RM 3000+, Jandel Engineering, UK).

电导率使用四点探针仪(RM 3000+,Jandel Engineering，U.K.)

Measurement.

测定。

[n0032]

The method for preparing lignin used in this invention is as follows:

本发明所用到的木质素的制备方法如下所示：

[n0033]

Extraction method of alkali lignin: Add wood chips and 15% NaOH solution to a rotary cooker and heat at 160°C for 120 min with stirring.

碱木质素的提取方法：将木片与15%NaOH溶液加入到旋转蒸煮锅中，在160°C下搅拌加热120min。

After the cooking pot has cooled to room temperature, the solid residue is removed by filtration and washed with deionized water. The washing liquid is mixed with the filtrate and poured into 5000 mL of distilled acid water (pH=2) to precipitate lignin.

待蒸煮锅冷却至室温，通过过滤除出固体残渣并用去离子水洗涤，将洗涤液与滤液混合后倒入5000mL蒸馏酸水中(pH＝2)以沉淀木质素。

After the precipitate precipitates out, the solid is obtained by filtration. The precipitate is collected by filtration and washed with a large amount of deionized water until the filtrate is neutral. Then, it is freeze-dried to obtain brown lignin.

待沉淀析出，抽滤得到固体，通过过滤收集沉淀并用大量去离子水洗涤，直到滤液为中性，随后冷冻干燥以获得棕色木质素。

[n0034]

Extraction method of enzymatic hydrolysis lignin: Weigh 60g of enzymatic hydrolysis residue (purchased from Costson Biotechnology, Enzymatic lignin) and pulverize it to 100 mesh using a micro pulverizer. Dry it under vacuum at 100°C for 12h. Then add it to a stainless steel high-pressure reactor. Add 1.2L of 50% ethanol solution at a solid-liquid ratio of 1:20 (i.e., 1g of solid raw material to 20mL of solution). Add 6g of NaOH as a catalyst. After mixing, heat to 220°C and react at 300r/min for 2h.

酶解木质素的提取方法：称取60g酶解残渣(购买于科斯特森合生物，酶解木质素Enzymatic)用微型粉碎机粉碎至100目，100°C下真空干燥12h；然后将其加入不锈钢高压反应釜中，按照固液比1：20(即1g固体原料加入20mL溶液)加入1.2L体积分数为50%的乙醇溶液，再加入6g NaOH作为催化剂，混匀后加热至220°C，在300r/min下反应2h。

After the reaction was completed, the solution was cooled to room temperature and filtered to separate the lignin solution from the insoluble matter. 5% H₂SO₄ was added to the filtrate, and a solid was precipitated. The filtrate was separated and subjected to vacuum distillation to recover ethanol. The solid was washed with water until neutral and then dried at 100°C to obtain lignin.

反应结束后，降至室温，过滤，将木质素溶液与不溶物分离，滤液中加入质量分数5%的H₂SO₄，析出固体，分离出滤液进行减压蒸馏回收乙醇，固体水洗至中性后，于100°C干燥制得木质素。

[n0035]

Extraction method of lignin by organic solvent: Add wood chips and ethanol solution (ethanol: water 7:3) into a rotary cooker and heat at 70°C with stirring for 120 min.

有机溶剂木质素的提取方法：将木片与乙醇溶液(乙醇：水7：3)加入到旋转蒸煮锅中，在70°C下搅拌加热120min。

The following steps are the same as the alkali lignin extraction method.

以下步骤同碱木质素提取方法。

[n0036]

Extraction method of sulfate lignin: Wood chips and cooking liquor prepared by 18% sodium hydroxide (Na₂O), 20% sodium sulfide and deionized water are loaded into a cooking tank, and then the cooking tank is placed into a rotary cooker for cooking.

硫酸盐木质素的提取方法：将木片与由18%氢氧化钠(Na₂O计)、20%硫化钠和去离子水配制而成的蒸煮液装入蒸煮罐中，再将蒸煮罐装入旋转蒸煮锅中进行蒸煮。

The following steps are the same as the alkali lignin extraction method.

以下步骤同碱木质素提取方法。

[n0037]

The Joule heating device is from Hefei In-situ Technology Co., Ltd., CIS-JH3.2.

焦耳加热装置来自合肥原位科技有限公司，CIS-JH3.2。

[n0038]

Example 1

实施例1

[n0039]

(1) Weigh 10g of alkali lignin and place it in a three-necked flask. Add 50mL of deionized water and adjust the pH to 10 using 20wt% sodium hydroxide solution. Stir at 75°C until the lignin is completely dissolved. Adjust the pH to 2 using 10wt% hydrochloric acid. Wash with deionized water 3 times and freeze dry to obtain dried lignin.

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(1)称取10g碱木质素置于三口烧瓶中，加入50mL去离子水，使用20wt%的氢氧化钠溶液调节pH为10，在75°C下搅拌直至木质素完全溶解，使用10wt%的盐酸调节pH为2，用去离子水洗涤3次，冷冻干燥得到干燥木质素；

[n0040]

(2) The dried lignin obtained in step (1) is heated in a tube furnace at 800°C with a heating rate of 5°C/min and a heating time of 2h to obtain lignin carbon precursor.

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(2)将步骤(1)得到的干燥木质素在管式炉中800°C温度下加热，升温速率为5°C/min，加热时间为2h，得到木质素碳前驱体；

[n0041]

(3) The lignin carbon precursor (5.2g) obtained in step (2) is placed in a graphite mold and carbonized in a Joule heating device. The carbonization temperature is 1200°C; the heating rate is 120°C/min; the holding time is 3min; the Joule heating voltage is 5V; the current is

350A; the output power is 3.5kw; and the pressure is 25MPa. The Joule thermal carbonization cycle is repeated 3 times.

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(3)将步骤(2)得到的木质素碳前驱体(5.2g)放入石墨模具中，在焦耳加热装置进行碳化，碳化温度为1200℃；加热速度为120℃/min，保温时间为3min，焦耳加热电压为5v，电流为350A，输出功率在3.5kw，压力在25MPa；焦耳热碳化循环次数3次。

As shown in the scanning electron microscope in Figure 1, the carbon layer is dispersed with little accumulation, indicating that the Joule heating flash evaporation method for preparing lignin hard carbon materials can effectively improve heating efficiency, reduce carbon layer accumulation, and thus increase electrical conductivity (conductivity of 3.45×10^3 S/m).

如图1扫描电镜所示，出现碳层分散，堆积现象少的情况，说明了焦耳加热闪蒸制备木质素硬碳材料的方法可以有效的提高加热效率，减少碳层堆积进而增加导电效率(导电率为3.45×10^3S/m)。

[n0042]

Example 2

实施例2

[n0043]

(1) Weigh 10g of enzymatically hydrolyzed lignin and place it in a three-necked flask. Add 50mL of deionized water and adjust the pH to 10 using 15wt% potassium hydroxide solution. Stir at 75°C until the lignin is completely dissolved. Adjust the pH to 3 using 10wt% hydrochloric acid. Wash twice with deionized water and freeze dry to obtain dried lignin.

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(1)称取10g酶解木质素置于三口烧瓶中，加入50mL去离子水，使用15wt%的氢氧化钾溶液调节pH为10，在75°C下搅拌直至木质素完全溶解，使用10wt%的盐酸调节pH为3，用去离子水洗涤2次，冷冻干燥得到干燥木质素；

[n0044]

(2) The dried lignin obtained in step (1) is heated in a tube furnace at 700°C with a heating rate of 5°C/min and a heating time of 3h to obtain lignin carbon precursor.

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(2)将步骤(1)得到的干燥木质素在管式炉中700°C温度下加热，升温速率为5°C/min，加热时间为3h，得到木质素碳前驱体；

[n0045]

(3) The lignin carbon precursor (5.4g) obtained in step (2) is placed in a graphite mold and subjected to Joule heating carbonization at a carbonization temperature of 1000°C, a heating

rate of 130°C/min, a holding time of 4min, a Joule heating voltage of 5V, a current of 350A, an output power of 3.5kw, and a pressure of 30MPa; the Joule heating carbonization cycle is repeated 3 times.

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(3)将步骤(2)得到的木质素碳前驱体(5.4g)放入石墨模具中，进行焦耳加热碳化，碳化温度为1000°C；加热速度为130°C/min，保温时间为4min，焦耳加热电压为5v，电流为350A，输出功率在3.5kw，压力在30MPa；焦耳热碳化循环次数3次。

[n0046]

Example 3

实施例3

[n0047]

(1) Weigh 10g of organic solvent lignin and place it in a three-necked flask. Add 50mL of deionized water and adjust the pH to 10 using 15wt% sodium hydroxide solution. Stir at 75°C until the lignin is completely dissolved. Adjust the pH to 2 using 15wt% sulfuric acid. Wash with deionized water 4 times and freeze dry to obtain dried lignin.

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(1)称取10g有机溶剂木质素置于三口烧瓶中，加入50mL去离子水，使用15wt%的氢氧化钠溶液调节pH为10，在75°C下搅拌直至木质素完全溶解，使用15wt%的硫酸调节pH为2，用去离子水洗涤4次，冷冻干燥得到干燥木质素；

[n0048]

(2) The dried lignin obtained in step (1) is heated in a tube furnace at 900°C with a heating rate of 8°C/min and a heating time of 3h to obtain lignin carbon precursor.

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(2)将步骤(1)得到的干燥木质素在管式炉中900°C温度下加热，升温速率为8°C/min，加热时间为3h，得到木质素碳前驱体；

[n0049]

(3) The lignin carbon precursor (5.0g) obtained in step (2) is placed in a graphite mold and subjected to Joule heating carbonization at a temperature of 1400°C, a heating rate of 120°C /min, a holding time of 4min, a Joule heating voltage of 5V, a current of 400A, an output power of 4.5kW, and a pressure of 30MPa. The Joule heating carbonization cycle is repeated 5 times.

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(3)将步骤(2)得到的木质素碳前驱体(5.0g)放入石墨模具中，进行焦耳加热碳化，碳化温度为1400°C；加热速度为120°C/min，保温时间为4min，焦耳加热电压为5v，电流为400A，输出功率在4.5kw，压力在30MPa；焦耳热碳化循环次数5次。

[n0050]

Example 4

实施例4

[n0051]

(1) Weigh 10g of sulfate lignin and place it in a three-necked flask. Add 50mL of deionized water and adjust the pH to 10 using 15wt% sodium hydroxide solution. Stir at 75°C until the lignin is completely dissolved. Adjust the pH to 3 using 10wt% hydrochloric acid. Wash with deionized water 5 times and freeze dry to obtain dried lignin.

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(1)称取10g硫酸盐木质素置于三口烧瓶中，加入50mL去离子水，使用15wt%的氢氧化钠溶液调节pH为10，在75°C下搅拌直至木质素完全溶解，使用10wt%的盐酸调节pH为3，用去离子水洗涤5次，冷冻干燥得到干燥木质素；

[n0052]

(2) The dried lignin obtained in step (1) is heated in a tube furnace at 800°C with a heating rate of 6°C/min and a heating time of 4h to obtain lignin carbon precursor.

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(2)将步骤(1)得到的干燥木质素在管式炉中800°C温度下加热，升温速率为6°C/min，加热时间为4h，得到木质素碳前驱体。

[n0053]

(3) The lignin carbon precursor (5.1g) obtained in step (2) is placed in a graphite mold and subjected to Joule heating carbonization at a carbonization temperature of 1000°C, a heating rate of 140°C/min, a holding time of 3min, a Joule heating voltage of 5V, a current of 350A, an output power of 2.5kW, and a pressure of 20MPa; the Joule heating carbonization cycle is repeated twice.

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(3)将步骤(2)得到的木质素碳前驱体(5.1g)放入石墨模具中，进行焦耳加热碳化，碳化温度为1000°C；加热速度为140°C/min，保温时间为3min，焦耳加热电压为5v，电流为350A，输出功率在2.5kw，压力在20MPa；焦耳热碳化循环次数2次。

[n0054]

Example 5

实施例5

[n0055]

(1) Weigh 10g of alkali lignin and place it in a three-necked flask. Add 50mL of deionized water and adjust the pH to 10 using 20wt% sodium hydroxide solution. Stir at 75°C until the lignin is completely dissolved. Adjust the pH to 2 using 10wt% hydrochloric acid. Wash with deionized water 3 times and freeze dry to obtain dried lignin.

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(1)称取10g碱木质素置于三口烧瓶中，加入50mL去离子水，使用20wt%的氢氧化钠溶液调节pH为10，在75°C下搅拌直至木质素完全溶解，使用10wt%的盐酸调节pH为2，用去离子水洗涤3次，冷冻干燥得到干燥木质素；

[n0056]

(2) The dried lignin obtained in step (1) is heated in a tube furnace at 800°C with a heating rate of 5°C/min and a heating time of 2h to obtain lignin carbon precursor.

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(2)将步骤(1)得到的干燥木质素在管式炉中800°C温度下加热，升温速率为5°C/min，加热时间为2h，得到木质素碳前驱体；

[n0057]

(3) The lignin carbon precursor (5.2g) obtained in step (2) is placed in a graphite mold and subjected to Joule heating carbonization at a temperature of 1300°C, a heating rate of 130°C /min, a holding time of 2min, a Joule heating voltage of 5V, a current of 350A, an output power of 3.5kW, and a pressure of 25MPa; the Joule heating carbonization cycle is repeated twice.

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(3)将步骤(2)得到的木质素碳前驱体(5.2g)放入石墨模具中，进行焦耳加热碳化，碳化温度为1300°C；加热速度为130°C/min，保温时间为2min，焦耳加热电压为5v，电流为350A，输出功率在3.5kw，压力在25MPa；焦耳热碳化循环次数2次。

[n0058]

Example 6

实施例6

[n0059]

(1) Weigh 10g of alkali lignin and place it in a three-necked flask. Add 50mL of deionized water and adjust the pH to 10 using 20wt% sodium hydroxide solution. Stir at 75°C until the lignin is completely dissolved.

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(1)称取10g碱木质素置于三口烧瓶中，加入50mL去离子水，使用20wt%的氢氧化钠溶液调节pH为10，在75°C下搅拌直至木质素完全溶解。

The pH was adjusted to 2 using 10 wt% hydrochloric acid, and the mixture was washed three times with deionized water and then freeze-dried to obtain dried lignin.

使用10wt%的盐酸调节pH为2，用去离子水洗涤3次，冷冻干燥得到干燥木质素。

[n0060]

(2) The dried lignin obtained in step (1) is heated in a tube furnace at 800°C with a heating rate of 5°C/min for 2 hours to obtain lignin carbon precursor.

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(2)将步骤(1)得到的干燥木质素在管式炉中800°C温度下加热，升温速率为5°C/min，加热时间为2h，得到木质素碳前驱体。

[n0061]

(3) The lignin carbon precursor (5.2g) obtained in step (2) is placed in a graphite mold and subjected to Joule heating carbonization at a temperature of 1400°C, a heating rate of 140°C/min, a holding time of 5min, a Joule heating voltage of 5V, a current of 350A, an output power of 3.5kW, and a pressure of 25MPa. The Joule heating carbonization cycle is repeated 5 times.

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(3)将步骤(2)得到的木质素碳前驱体(5.2g)放入石墨模具中，进行焦耳加热碳化，碳化温度为1400°C；加热速度为140°C/min，保温时间为5min，焦耳加热电压为5v，电流为350A，输出功率在3.5kw，压力在25MPa；焦耳热碳化循环次数5次。

[n0062]

The embodiments provided above are not intended to limit the scope of the present invention, nor are the described steps intended to limit the order of their execution.

以上所提供的实施例并非用以限制本发明所涵盖的范围，所描述的步骤也不是用以限制其执行顺序。

Any obvious modifications made to this invention by those skilled in the art based on existing common knowledge also fall within the scope of protection defined by the claims of this invention.

本领域技术人员结合现有公知常识对本发明做显而易见的改进，亦落入本发明权利要求书所界定的保护范围之内。