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Published:

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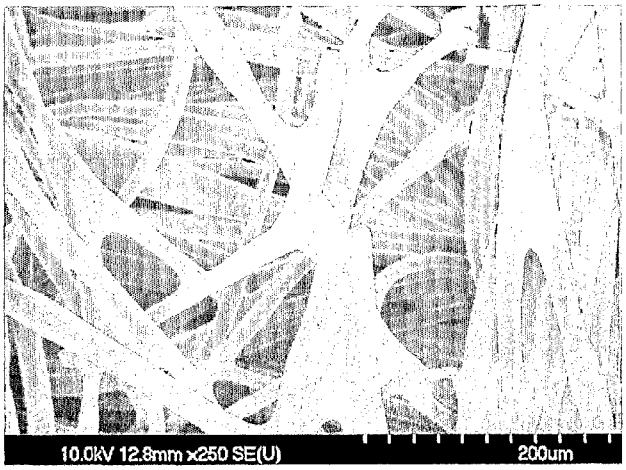
(54) Title: FABRICATION METHOD FOR CAPACITOR ELECTRODE

[Fig. 1]



(57) Abstract: A fabrication method for a capacitor electrode is disclosed. The fabrication method for a capacitor electrode comprises forming a base polymer layer on an upper surface of the electrode; forming a cation exchanger or an anion exchanger on the base polymer layer; and drying the electrode. The base polymer layer may be formed by any one selected from a group consisting of a doctor blade coating method, a dipping method, a flooding method, a spin coating method, a spraying method, a brushing method, and a painting method. When being used in the water treatment process, the capacitor electrode fabricated using the method according to the present invention can enhance the ion removal rate and reduce power consumption.

WO 2010/101338 A1

Description

FABRICATION METHOD FOR CAPACITOR ELECTRODE

Technical Field

[1] The present invention relates to a fabrication method for a capacitor electrode that can increase an ion removal rate in a water treatment process and reduce power consumption.

Background Art

[2] Capacitive deionization (CDI) process is a water treatment process that applies voltage to a porous carbon electrode to remove ions. During the CDI process, an anion moves to an anodic capacitor electrode and a cation moves to a cathodic capacitor electrode, thereby being absorbed. During the CDI process, the ion deionized during a regeneration period of the capacitor electrode is moved to a capacitor electrode on an opposite side to be re-absorbed, causing problems of degradation in an electrode regeneration rate and an ion removal rate.

[3] In order to solve such problems, membrane capacitive deionization (MCDI) process has been attempted. A capacitor electrode used in the MCDI process is in a form where a membrane (ion exchange membrane) is coupled to the capacitor electrode. In other words, an anion exchange membrane is coupled to an anodic capacitor electrode and a cation exchange membrane is coupled to a cathodic capacitor electrode. During a water treatment process, the anion and the cation selectively pass through the ion exchange membrane to be absorbed by the electrode. There has been reported a study that as a result, a phenomenon that the deionized ion is re-absorbed can be prevented at the time of regeneration of the electrode, such that the ion removal rate is increased. However, the resistance of the entire system has been increased due to an ion migration resistance (Rm) of the ion exchange membrane itself and a contact resistance (Rc) caused by a generation of an interface between the ion exchange membrane and the capacitor electrode. As a result, current I (I=V/R) that is calculated according to ohm s law is reduced, causing a problem that the ion removal rate is actually lowered.

Disclosure of Invention

Technical Problem

[4] Accordingly, it is an object of the present invention to provide a fabrication method for a capacitor electrode that can increase an ion removal rate in a water treatment process and reduce power consumption.

Technical Solution

[5] In order to accomplish the object of the present invention, there is provided a fabrication method for a capacitor electrode comprising: forming a base polymer layer on

an upper surface of the electrode; forming a cation exchanger or an anion exchanger on the base polymer layer; and drying the electrode.

[6] Preferably, the base polymer layer may be formed by any one selected from a group consisting of a doctor blade coating method, a dipping method, a flooding method, a spin coating method, a spraying method, a brushing method, and a painting method.

Advantageous Effects

[7] The present invention forms the base polymer layer using the spraying method, making it possible to make the thickness of the base polymer layer thin. The capacitor electrode including the base polymer layer having the thin thickness generates little resistance, thereby having little power consumption.

[8] Further, the capacitor electrode fabricated according to the present invention includes the base polymer layer on which the ion exchanger is formed, thereby making it possible to increase the ion removal rate in the water treatment process.

Brief Description of Drawings

[9] The above and other objects, features and advantages of the present invention will become apparent from the following description of preferred embodiments given in conjunction with the accompanying drawings, in which:

[10] FIG. 1 is a SEM photograph showing a capacitor electrode (A) of the present invention when a cation exchanger of a sulfonic acid group is formed on a base polymer layer of comparative example 1;

[11] FIG. 2 is a SEM photograph showing a capacitor electrode (B) of the present invention when a cation exchanger is not formed on the base polymer layer of comparative example 1;

[12] FIG. 3 is a unit cell structure diagram of ECDI using the capacitor electrode of the present invention;

[13] FIG. 4 is a unit cell structure diagram of CDI;

[14] FIG. 5 is a unit cell structure diagram of MCDI;

[15] FIG. 6 is a graph showing comparison of conductivities in the ECDI, CDI, and MCDI processes; and

[16] FIG. 7 is a graph showing comparison of current values in the ECDI, CDI, and MCDI processes.

Best Mode for Carrying out the Invention

[17] The present invention describes a fabrication method for a capacitor electrode. The fabrication method for the capacitor electrode comprises: forming a base polymer layer on an upper surface of the electrode; forming a cation exchanger or an anion exchanger on the base polymer layer; and drying the electrode, wherein the base polymer layer is formed by any one selected from a group consisting of a doctor blade coating method,

a dipping method, a flooding method, a spin coating method, a spraying method, a brushing method, and a painting method.

[18] The electrode may include any one selected from a group consisting of carbon, a mixture of carbon and metal, a compound containing carbon and metal, a mixture of carbon and a binder, and a mixture of carbon and functional substance. The carbon, which is any one porous carbon selected from a group consisting of aerogel, activated carbon, graphite, recticulated vitreous carbon (RVC), and carbon fiber, may include carbon in the form of paper, fiber, cloth, AC composite or a felt, having conductivity. The functional substance may be an ion exchange fiber. The binder may be a binder that is commonly used.

[19] The base polymer layer may include hydrocarbon-based polymer, fluorinated polymer or semifluorinated polymer.

[20] The hydrocarbon-based polymer may include one or more mixture selected from a group consisting of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide) (BPPO), polystyrene, styrene butadiene, polyethylene, polypropylene, polyarylene ether, polyimide, polyetherimide, polyketone, polyetherketone, polysulfone, polyimidazole, and polybenzimidazole. Further, the hydrocarbon-based polymer may include a copolymer that is obtained by copolymerizing one or more selected from a group consisting of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide) (BPPO), polystyrene, styrene butadiene, polyethylene, polypropylene, polyarylene ether, polyimide, polyetherimide, polyketone, polyetherketone, polysulfone, polyimidazole, and polybenzimidazole.

[21] The semifluorinated polymer may include one or more mixture selected from a group consisting of polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), ethylene tetrafluoroethylene (ETFE), tetrafluoroethylene- perfluoroalkylvinyl ether copolymer (poly(perfluoroalkyl acrylate), PFA), tetrafluoroethylene-hexafluoropropylene copolymer (fluorinated ethylene propylene, FEP), and polyvinyl fluoride (PVF). In addition, the semifluorinated polymer may include a copolymer that is obtained by copolymerizing one or more selected from a group consisting of polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), ethylene tetrafluoroethylene (ETFE), tetrafluoroethylene-perfluoroalkylvinyl ether copolymer (poly(perfluoroalkyl acrylate), PFA), tetrafluoroethylene-hexafluoropropylene copolymer (fluorinated ethylene propylene, FEP), and polyvinyl fluoride (PVF).

[22] The cation exchanger may be any one of a sulfonic acid group, a phosphoric acid group, and a carboxylic acid group.

[23] The anion exchanger may be any one of a primary amine group, a secondary amine group, a tertiary amine group, a quaternary ammonium group, a poly ethylene imine group, and a phosphonium group.

[24] The capacitor electrode of the present invention provides advantages in view of a high ion removal rate in the water treatment process and low power consumption, etc. Further, the capacitor electrode of the present invention provides advantages in view of a rapid ion capacitive ability, a low resistance loss, etc., such that it can also be applied to a super capacitor, etc.

[25] Hereinafter, the present invention will be described in detail with reference to Examples and Comparative Examples. The present invention may, however, be embodied in many different forms and should not be construed as being limited to the Examples set forth herein.

[26] **Example 1: Fabrication of capacitor electrode having a sulfonic acid group as a cation exchanger**

[27] 1g of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide)(BPPO) was put in 5ml of N-methylpyrrolidone (NMP) and then was sufficiently mixed at room temperature for 1 day, thereby making a mixed solution. The mixed solution was put into a centrifugal separator and was centrifuged at a temperature of 25℃ for 30 minutes at 13,000 rpm. The solid obtained by centrifuging the mixed solution was cleaned with distilled water four times or more and then was put in a vacuum state at a temperature of 40℃ for 12 hours or more, thereby obtaining pure BPPO.

[28] 5ml of NMP was put for each 1g of the pure BPPO obtained as above and then was sufficiently mixed at room temperature for 1 day, thereby making a BPPO solution. 0.02 to 20ml of BPPO solution was sprayed on a porous carbon electrode having a size of 0.05 to 50 cm^2. Then, the porous carbon electrode to which the BPPO solution was sprayed was dried at a temperature of 40℃ for 12 hours or more. As a result, the porous carbon electrode where the BPPO is formed as a base polymer layer was fabricated.

[29] The porous carbon electrode where the BPPO is formed as the base polymer layer was dipped in 100% sulfuric acid solution for 20 minutes and then was dipped in 80% sulfuric acid solution, 50% sulfuric acid solution, and 30% sulfuric acid solution each for 1 minute. Thereafter, the porous carbon electrode was cleaned with distilled water. As a result, a capacitor electrode where the sulfonic acid group is formed on the base polymer layer as a cation exchanger was obtained.

[30] **Example 2: Fabrication of capacitor electrode having a quaternary amine group as an anion exchanger**

[31] 1g of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide)(BPPO) was put in 5ml of N-methylpyrrolidone (NMP) and then was sufficiently mixed at room temperature for 1 day, thereby making a mixed solution. The mixed solution was put into a centrifugal separator and was centrifuged at a temperature of 25℃ for 30 minutes at 13,000 rpm. The solid obtained by centrifuging the mixed solution was cleaned with

distilled water four times or more and then was put in a vacuum state at a temperature of 40°C for 12 hours or more, thereby obtaining pure BPPO.

[32] 5ml of NMP was put for each 1g of the pure BPPO obtained as above and then was sufficiently mixed at room temperature for 1 day, thereby making a BPPO solution. 0.02 to 20ml of BPPO solution was sprayed on a porous carbon electrode having a size of 0.05 to 50 cm^2. Then, the porous carbon electrode to which the BPPO solution was sprayed was dried at a temperature of 40°C for 12 hours or more. As a result, the porous carbon electrode where the BPPO is formed as a base polymer layer was fabricated.

[33] The porous carbon electrode where the BPPO is formed as the base polymer layer was dipped in trimethylamine (TMA) solution for 20 minutes and was cleaned with distilled water. As a result, a capacitor electrode where the quaternary amine group is formed on the base polymer layer as an anion exchanger was obtained.

[34] **Comparative Example 1**

[35] A capacitor electrode (A) where a sulfonic acid group of a cation exchanger is formed on a base polymer layer was compared with a capacitor electrode (B) configured of pure carbon fiber. The capacitor electrode fabricated in Example 1 was used as the capacitor electrode (A).

[36] The capacitor electrode (B) was fabricated through the following process.

[37] 1g of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide)(BPPO) was put in 5ml of N-methylpyrrolidone (NMP) and then was sufficiently mixed at room temperature for 1 day, thereby making a mixed solution. The mixed solution was put into a centrifugal separator and was centrifuged at a temperature of 25°C for 30 minutes at 13,000 rpm. The solid obtained by centrifuging the mixed solution was cleaned with distilled water four times or more and then was put in a vacuum state at a temperature of 40°C for 12 hours or more, thereby obtaining pure BPPO.

[38] 5ml of NMP was put for each 1g of the pure BPPO obtained as above and then was sufficiently mixed at room temperature for 1 day, thereby making a BPPO solution. 0.02 to 20ml of BPPO solution was sprayed to a porous carbon electrode having a size of 0.05 to 50 cm^2. Then, the porous carbon electrode to which the BPPO solution was sprayed was dried at a temperature of 40°C for 12 hours or more. As a result, the porous carbon electrode (B) where the BPPO is formed as a base polymer layer was fabricated.

[39] The SEM photographs of the capacitor electrode (A) of the present invention and the capacitor electrode (B) as described above are shown in FIGS. 1 and 2, respectively. Differently from the capacitor electrode (B), a gap between the respective electrodes was filled with the cation exchanger of the sulfonic acid in the capacitor electrode (A). As a result, the capacitor electrode (A) reduces a contact resistance, thereby increasing

conductivity.

[40] **Comparative Example 2**

[41] As a water treatment process of removing ions using a capacitor electrode where a cation exchanger is formed on a base polymer layer of the present invention and a capacitor electrode where an anion exchanger is formed on the base polymer layer thereof, a unit cell structure diagram of embedded capacitive deionization (ECDI) is shown in FIG. 3. A current collection plate is coupled to the lower surface of the capacitor electrode.

[42] Compared therewith, as a water treatment process of removing ions using the capacitor electrode coupled to the current collection plate, a unit cell structure diagram of capacitive deionization (CDI) process is shown in FIG. 4.

[43] Further, as a water treatment process of removing ions using a capacitor electrode where a cation exchange membrane is formed and a capacitor electrode where an anion exchange membrane is formed, a unit cell structure diagram of membrane capacitive deionization (MCDI) is shown in FIG. 5. A current collection plate is coupled to the lower surface of the capacitor electrode.

[44] **Comparative Example 3: Ion removal rate**

[45] As shown in Comparative Example 2, in order to check ion removal performance when the capacitor electrode of the present invention is used in the ECDI process (see FIG. 3), conductivity while the process is performed was reviewed. The process under the operation conditions of voltage of 1.8V and flow rate of 4ml/min was performed for 30 minutes.

[46] As the comparative example of the ECDI process, as shown in Comparative Example 2, conductivities when the capacitor electrode configured of pure carbon fiber is used in the CDI process (see FIG. 4) and when the capacitor electrode where the ion exchange membrane is formed is used in the MCDI process (see FIG. 5) were reviewed.

[47] FIG. 6 is a graph showing comparison of conductivities in the ECDI, CDI, and MCDI processes.

[48] In the case of the ECDI process of the present invention, the conductivity fell from 188.9μS/cm to 31.5μS/cm. From the change in the conductivity, it could be appreciated that the ion removal rate in the ECDI process was as high as 83.4%. Ions were selectively absorbed due to the action of the ion exchanger of the capacitor electrode used in the ECDI process, such that the ions were efficiently removed.

[49] In the case of the CDI process, the conductivity fell from 190.8μS/cm to 39.9μS/cm. From the change in the conductivity, the ion removal rate in the CDI process was calculated as 79.1%.

[50] In the case of the MCDI process, the conductivity fell from 185.1μS/cm to

168μS/cm. From the change in the conductivity, the ion removal rate in the MCDI process was calculated as 9.23%. With the voltage under the same condition as the ECDI process of the present invention, the ion removal rate in the MCDI process was significantly lower.

[51] **Comparative Example 4: Power consumption**

[52] As shown in Comparative Example 2, in order to check power consumption when the capacitor electrode of the present invention is used in the ECDI process (see FIG. 3), current values while the process is performed were examined. The process under the operation conditions of voltage of 1.8V and flow rate of 4ml/min was performed for 30 minutes.

[53] As the comparative example of the ECDI process, as shown in Comparative Example 2, current values when the capacitor electrode without ion exchanger on the base polymer layer is used in the CDI process (see FIG. 4) and when the capacitor electrode where the ion exchanger is formed on the base polymer layer is used in the MCDI process (see FIG. 5) were compared.

[54] FIG. 7 is a graph showing comparison of current values in the ECDI, CDI, and MCDI processes.

[55] The average value of current while the ECDI process of the present invention is performed for 30 minutes was as low as 0.02564A. As a result, the power consumption calculated using the average value of current was merely 0.02308Wh (0.02564A x 1.8V x 0.5 = 0.02308Wh).

[56] The average value of current while the CDI process of the present invention is performed for 30 minutes was 0.2357A. The power consumption calculated using the average value of current was 0.2121Wh (0.2357A x 1.8V x 0.5 = 0.2121Wh).

[57] The average value of current while the MCDI process of the present invention is performed for 30 minutes was 0.002575A. The power consumption calculated using the average value of current was 0.002318Wh (0.002575A x 1.8V x 0.5 = 0.002318Wh). Although the power consumption in the MCDI process was very low, the ion removal rate at this time was merely 9.23% (see Comparative Example 3). Therefore, the object of the MCDI process, removal of dissolved ions, can not be accomplished.

[58] Through the results of Comparative Example 3 and Comparative Example 4, it can be confirmed that it is the most preferable to perform the water treatment using the ECDI process using the capacitor electrode of the present invention. In other words, when performing the water treatment using the capacitor electrode of the present invention, the ion removal rate can be enhanced and at the same time, the power consumption can also be significantly reduced compared to the CDI process.

[59] While the present invention has been described with reference to the preferred em-

bodiments, it will be understood by those skilled in the related art that various modifications and variations may be made therein without departing from the scope of the present invention as defined by the appended claims.

Claims

[1] A fabrication method for a capacitor electrode comprising:
forming a base polymer layer on an upper surface of the electrode;
forming a cation exchanger or an anion exchanger on the base polymer layer; and drying the electrode,
wherein, the base polymer layer is formed by any one selected from a group consisting of a doctor blade coating method, a dipping method, a flooding method, a spin coating method, a spraying method, a brushing method, and a painting method.

[2] The fabrication method according to claim 1, wherein the electrode includes any one selected from a group consisting of carbon, a mixture of carbon and metal, a compound containing carbon and metal, a mixture of carbon and a binder, a mixture of carbon and functional substance, and the carbon is any one conductive porous carbon selected from a group consisting of aerogel, activated carbon, graphite, recticulated vitreous carbon (RVC), and carbon fiber.

[3] The fabrication method according to claim 2, wherein the functional substance is ion exchange fiber.

[4] The fabrication method according to claim 1, wherein the base polymer layer includes any one of hydrocarbon-based polymer, fluorinated polymer, and semi-fluorinated polymer.

[5] The fabrication method according to claim 4, wherein the hydrocarbon-based polymer includes one or more mixture selected from a group consisting of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide) (BPPO), polystyrene, styrene butadiene, polyethylene, polypropylene, polyarylene ether, polyimide, polyetherimide, polyketone, polyetherketone, polysulfone, polyimidazole, and polybenzimidazole.

[6] The fabrication method according to claim 4, wherein the hydrocarbon-based polymer includes a copolymer that is obtained by copolymerizing one or more selected from a group consisting of bromomethylated poly(2,6-dimethyl-1,4-phenylene oxide) (BPPO), polystyrene, styrene butadiene, polyethylene, polypropylene, polyarylene ether, polyimide, polyetherimide, polyketone, polyetherketone, polysulfone, polyimidazole, and polybenzimidazole.

[7] The fabrication method according to claim 4, wherein the semifluorinated polymer includes one or more mixture selected from a group consisting of polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), ethylene tetrafluoroethylene (ETFE), tetrafluoroethylene-perfluoroalkylvinyl ether

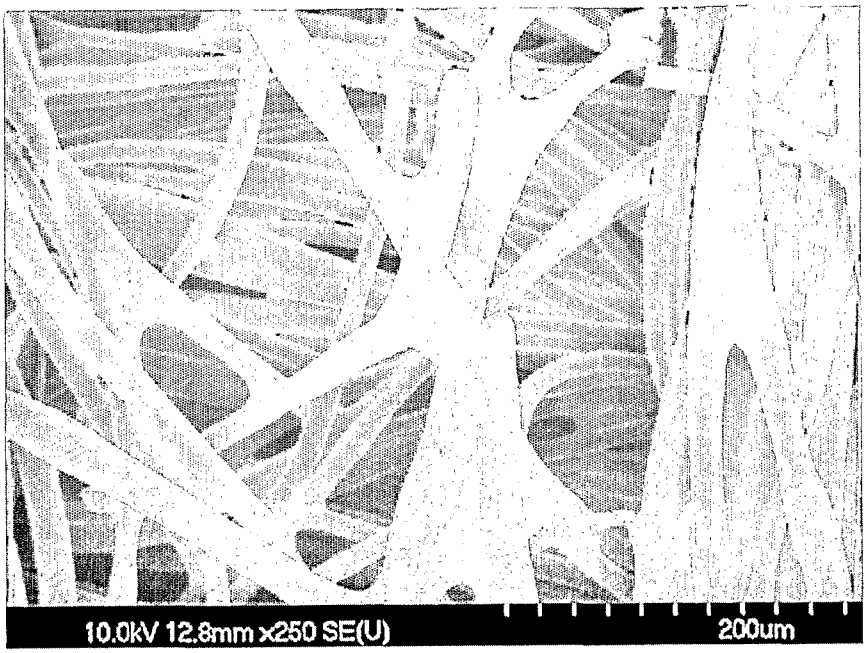
copolymer (poly(perfluoroalkyl acrylate), PFA), tetrafluoroethylene-hexafluoropropylene copolymer (fluorinated ethylene propylene, FEP), and polyvinyl fluoride (PVF).

[8] The fabrication method according to claim 4, wherein the semifluorinated polymer includes a copolymer that is obtained by copolymerizing one or more selected from a group consisting of polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE), ethylene tetrafluoroethylene (ETFE), tetrafluoroethylene-perfluoroalkylvinyl ether copolymer (poly(perfluoroalkyl acrylate), PFA), tetrafluoroethylene-hexafluoropropylene copolymer (fluorinated ethylene propylene, FEP), and polyvinyl fluoride (PVF).

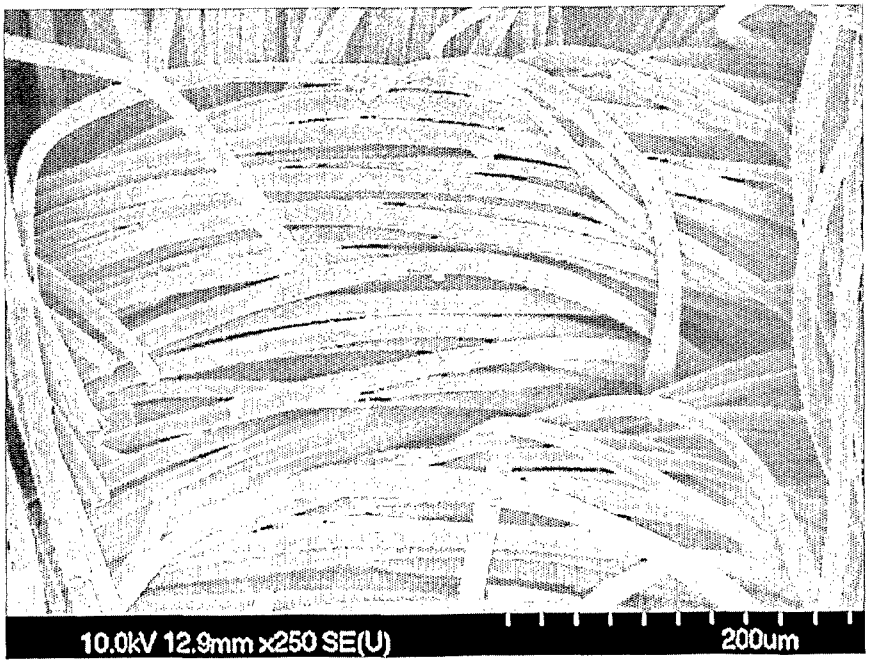
[9] The fabrication method according to claim 1, wherein the cation exchanger is any one of a sulfonic acid group, a phosphoric acid group, and a carboxylic acid group.

[10] The fabrication method according to claim 1, wherein the anion exchanger is any one of a primary amine group, a secondary amine group, a tertiary amine group, a quaternary ammonium group, a poly ethylene imine group, and a phosphonium group.

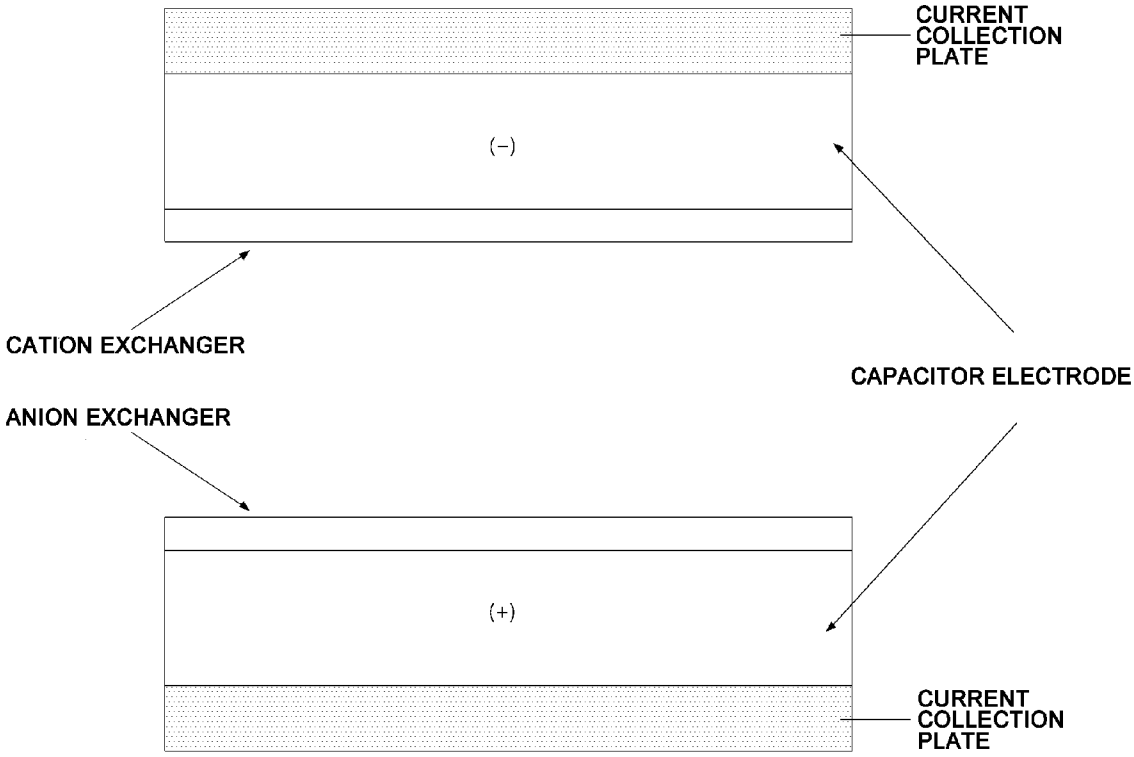
[Fig. 1]



[Fig. 2]



[Fig. 3]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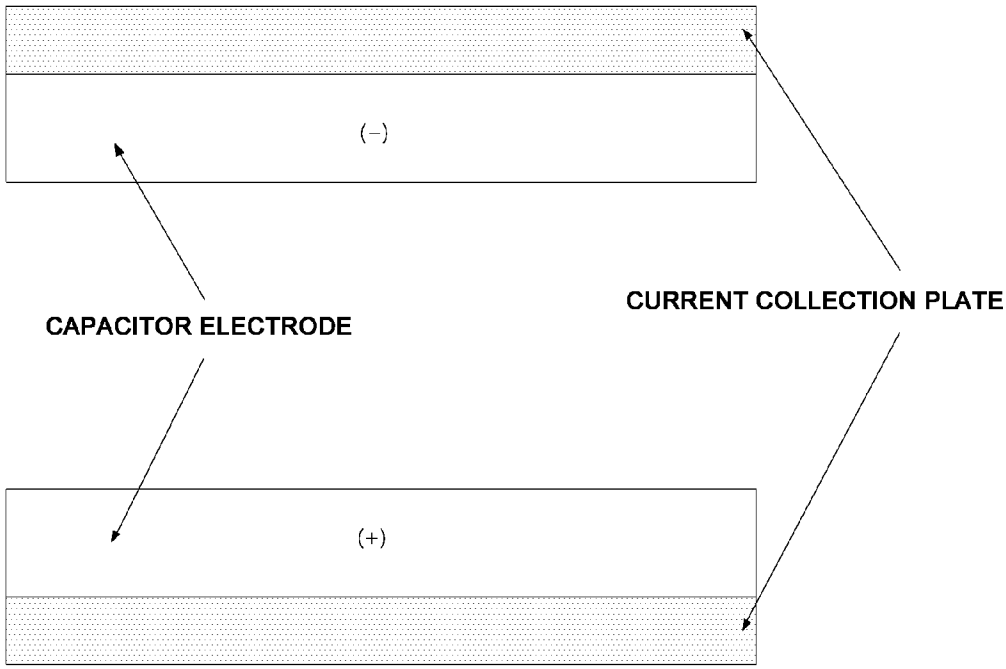
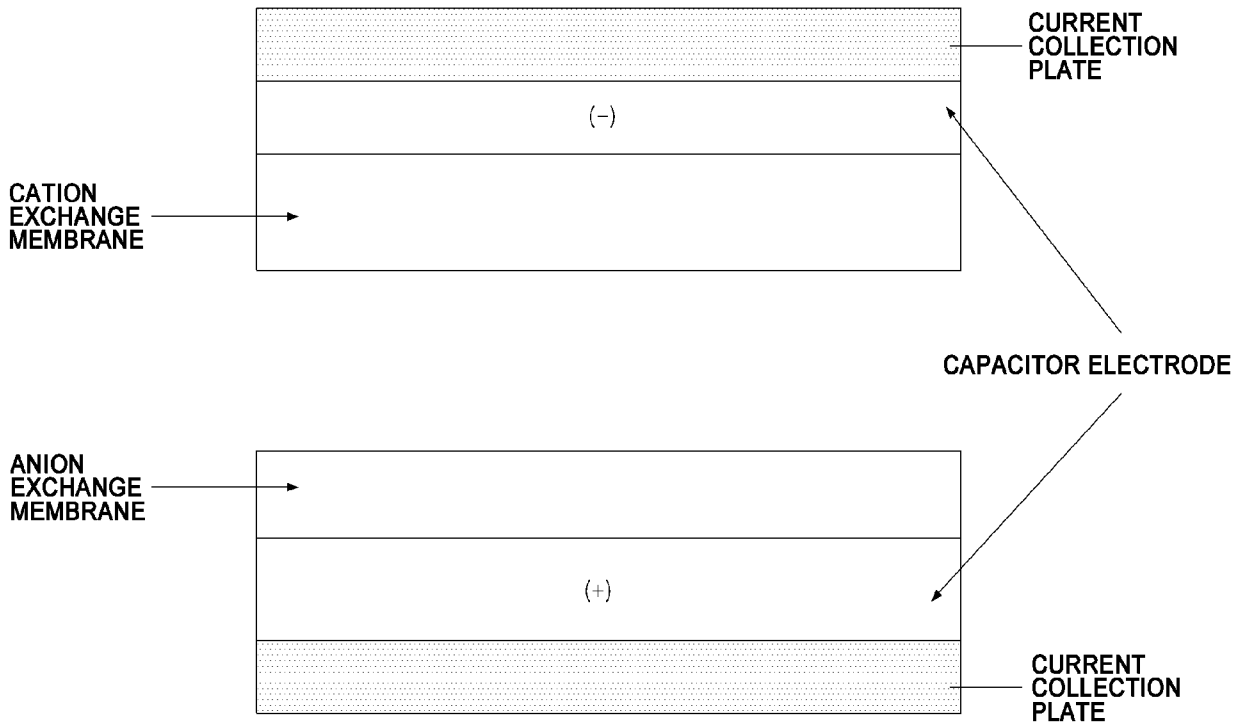
CAPACITOR ELECTRODE
ANION EXCHANGER
(+)
CURRENT COLLECTION PLATE

[Fig. 4]


(-)
CAPACITOR ELECTRODE
CURRENT COLLECTION PLATE
(+)

[Fig. 5]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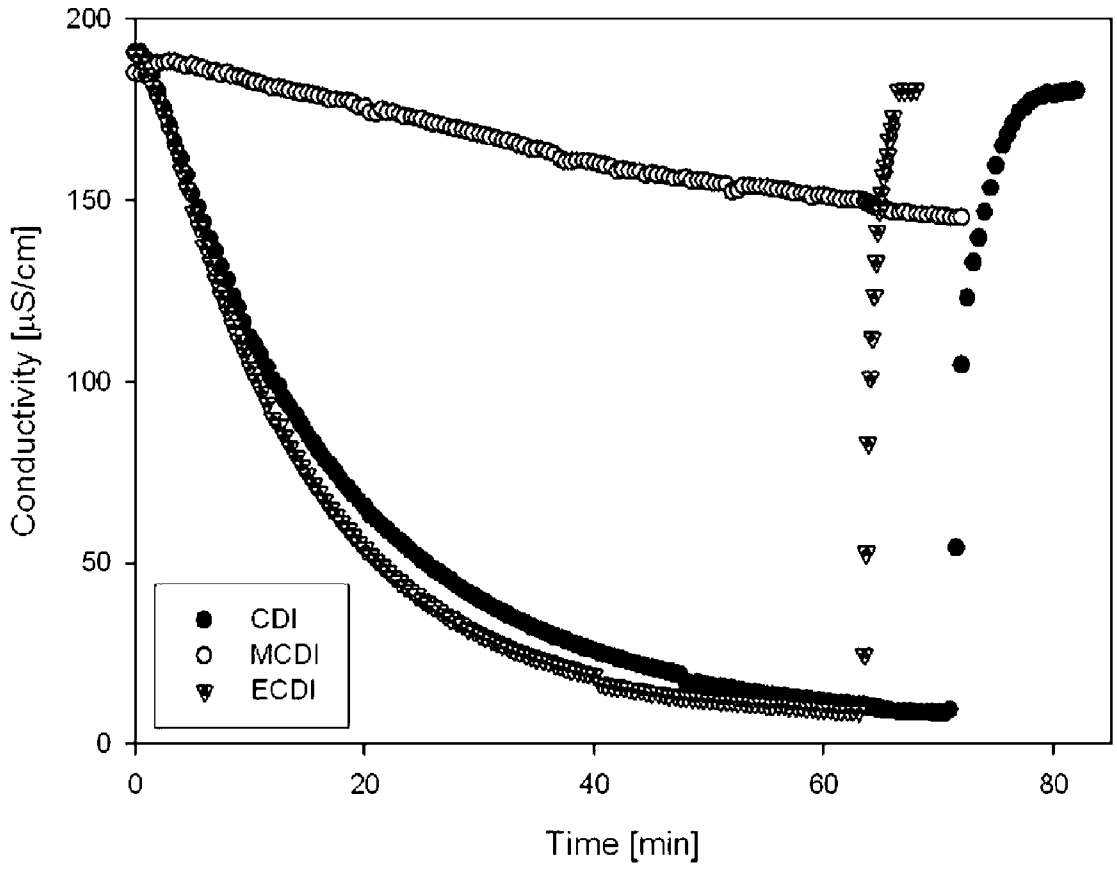
CATION EXCHANGE MEMBRANE
CAPACITOR ELECTRODE
ANION EXCHANGE MEMBRANE
(+)
CURRENT COLLECTION PLATE

[Fig. 6]


Conductivity [μS/cm]
Time [min]
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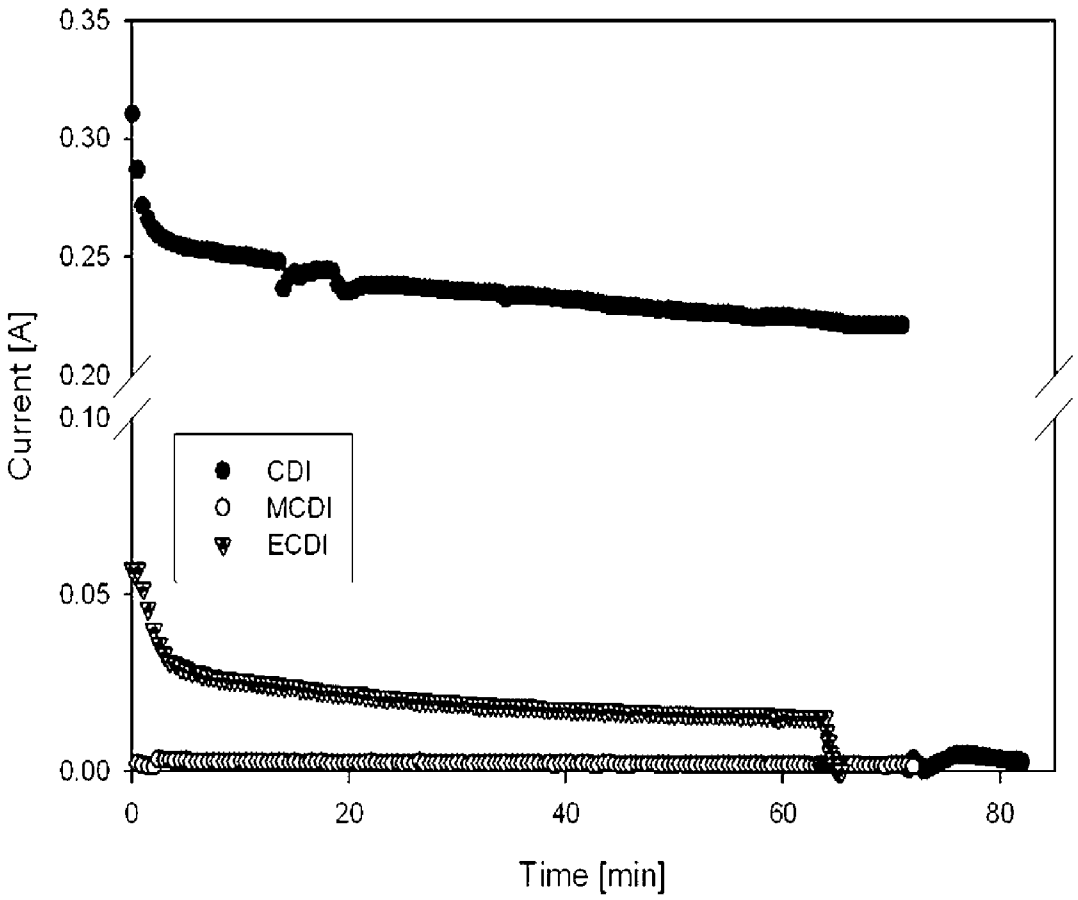
MCDI
ECDI
0
50
100
150
200
0
20
40
60
80

[Fig. 7]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MCDI
ECDI
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20
40
60
80
Time [min]

INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2009/004261

A. CLASSIFICATION OF SUBJECT MATTER

H01G 9/058(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)

IPC: H01G 9/058

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

KOREAN UTILITY MODELS AND APPLICATIONS FOR UTILITY MODELS SINCE 1975

JAPANESE UTILITY MODELS AND APPLICATIONS FOR UTILITY MODELS SINCE 1975

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

eKOMPASS, PAJ; capacitor, electrode, polymer layer, exchanger

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 2008/0078672 A1 (ATLAS, R.D.) 03 April 2008. see abstract; paragraphs [0031]-[0035]; figures 1-4; claim 1	1-10
A	US 2008/0198531 A1 (SHIUE, L. et al.) 21 August 2008. see abstract; paragraphs [0022]-[0027]; figures 1-3; claim 1	1-10
A	US 2002/0167782 A1 (ANDELMAN, M.D. et al.) 14 November 2002. see abstract; paragraphs [0046]-[0060]; figures 1-8; claim 1	1-10
A	US 6426863 B1 (MUNSHI, M.Z.A.) 30 July 2002. see abstract; column 14 line 40-column 17 line 17; figures 1-3; claim 1	1-10

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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29 MARCH 2010 (29.03.2010)	**30 MARCH 2010 (30.03.2010)**

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Information on patent family members

International application No.

PCT/KR2009/004261

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