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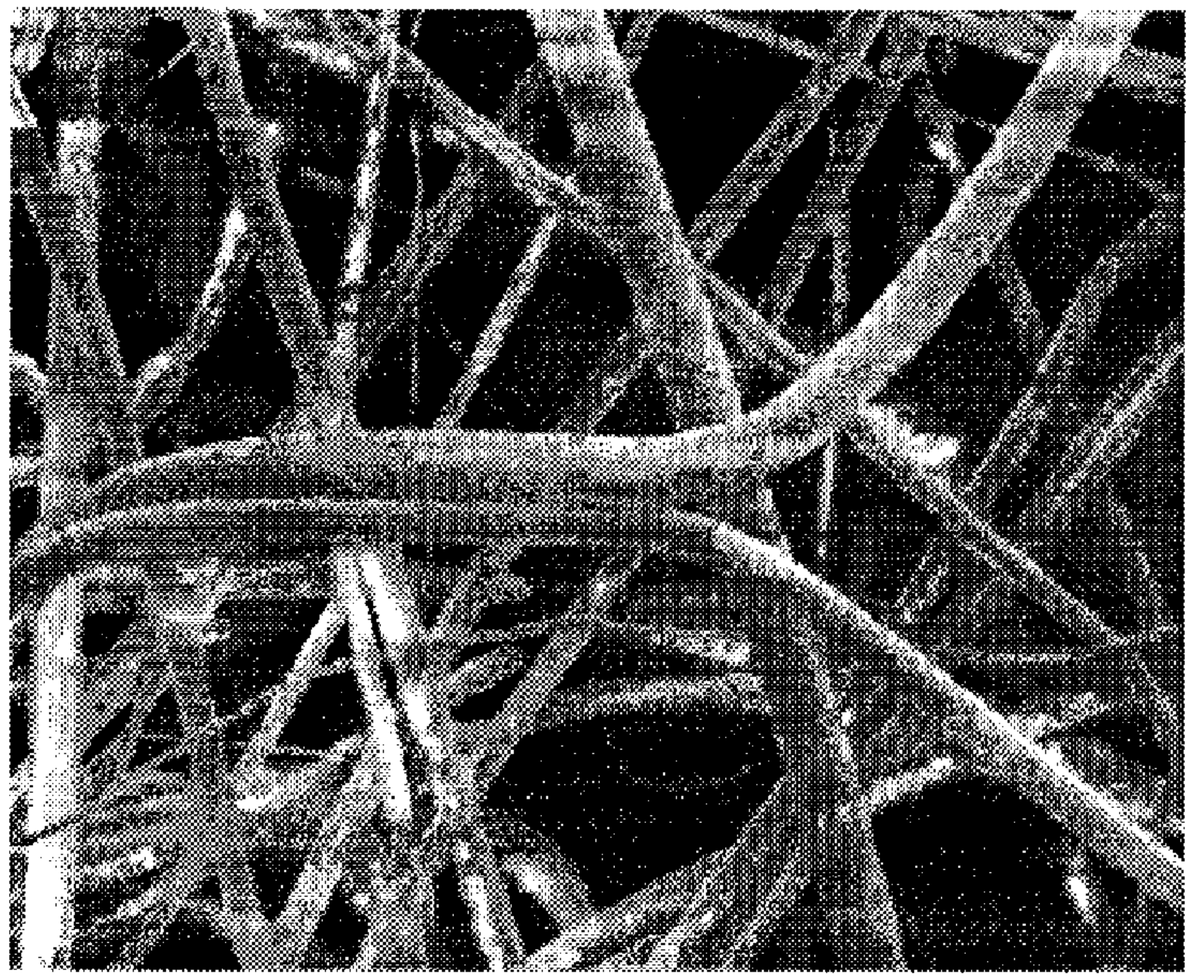
(54) Titre : BANDES DE NANOFIBRES ELECTROFILEES DESTINEES AU RENFORCEMENT DE MEMBRANE DANS LES PILES A COMBUSTIBLE

(54) Title: ELECTROSPUN NANOFIBER WEBS FOR MEMBRANE REINFORCEMENT IN FUEL CELLS



(57) Abrégé/Abstract:
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(57) Abrégé(suite)/Abstract(continued):
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ABSTRACT

Nanofiber webs prepared by electrospinning technology can be used as a reinforcement matrix in the proton exchange membrane of a solid polymer electrolyte fuel cell. Certain membrane and/or fuel cell properties can be improved by welding the nanofibers together where they intersect and/or by impregnating the nanofibers with useful additives that diffuse out over time. The mechanical and chemical stabilities as well as the durability of the membrane can be significantly improved.

Docket No.: 2016P03884CA

ELECTROSPUN NANOFIBER WEBS FOR MEMBRANE REINFORCEMENT IN FUEL CELLS

BACKGROUND

Field of the Invention

This invention relates to reinforced proton exchange membranes (PEMs) for use in solid polymer electrolyte fuel cells. In particular, it relates to PEMs reinforced with electrospun nanofiber webs in which the nanofibers have been welded together and/or impregnated with useful additives.

Description of the Related Art

Proton exchange membrane fuel cells (PEMFCs) generally employ a proton exchange membrane (PEM) between two electrodes, namely a cathode and an anode. The structure comprising a proton exchange membrane sandwiched between two electrodes is known as a membrane electrode assembly (MEA). The PEM serves as a separator to prevent mixing of reactant gases as well as an electrolyte for transporting protons from anode to cathode. Polymer electrolyte membranes for PEMFC applications should possess good performance and durability (e.g. high protonic conductivity, good chemical stability, good mechanical stability); low permeability to reactants; low swelling in water; and should be as low cost as possible for the development and commercialization of fuel cell systems in either stationary or transportation applications.

In automotive applications for instance, a PEM may be required to demonstrate durability of about 6,000 hours. To improve PEM durability, reinforcement technologies have been widely used in the preparation of PEMs for such fuel cells. Reinforcement technology can improve PEM mechanical properties and control PEM swelling in water. A suitable reinforcement matrix can be either a porous e-PTFE web (such as disclosed in WO1997041168A1, US20140261983A1, and US8795923B2) or a nanofiber web (such as disclosed in WO2014104785A1 and US20150303505A1).

To further improve PEM durability, Toyota researchers mixed inorganic free radical scavengers (e.g. CeO_2, $CePO_4$) with PTFE powder to prepare e-PTFE webs and reinforced PEMs comprising these scavengers (see for instance US20100233571A1). The purpose of this was to suppress leaching of the free radical scavengers and to improve PEM chemical stability. However, disadvantages of incorporating these

inorganic free radical scavengers in e-PTFE include making a trade-off between PEM durability and performance, and because these inorganic free radical scavengers are incorporated in the membrane in the form of particles, pinholes can form in the membrane after the free radical scavenger particles are leached out.

In commonly owned US patent serial numbers 9,172,107 and 9,391,337, organic and metal ion complex additives for PEMs are disclosed. Compared to the free radical scavengers used in US20100233571A1, these additives can significantly improve PEM durability without an impact on PEM performance. Furthermore, these additives can be soluble in solvents. However, fuel cells comprising MEAs with such additives need longer conditioning times (~16 hours) to achieve full performance (henceforth referred to as a "key-on issue").

There remains a continuing need to develop reinforcement matrix for PEM in solid polymer electrolyte fuel cells. This invention fulfills these needs and provides further related advantages.

SUMMARY

The present invention relates to new reinforcement matrices for fabrication of reinforced PEMs for solid polymer electrolyte fuel cells. In one embodiment, a new reinforcement matrix comprises an electro-spun nanofiber web in which the nanofibers are welded together where the fibres intersect, thereby forming a plurality of welded joints. In another embodiment, a new reinforcement matrix comprises an electro-spun nanofiber web in which the nanofibres have been impregnated with useful additives that diffuse out over time (e.g. a retarded release). In yet another embodiment, a new reinforcement matrix comprises an electro-spun nanofiber web comprising both welded joints and an impregnated additive. PEMs reinforced with these new types of matrices can have improved mechanical properties, chemical stability, and durability over those reinforced with a conventional nanofiber web.

Compared to conventional nanofiber webs which had previously been disclosed for use as reinforced matrices for the PEMs in fuel cells (e.g. as in US20150303505A1) or the webs used as air and water filters (e.g. as in US20120137885), in one embodiment of the invention the nanofiber web has "welded" joints. Welded joints in the web (in which fused joints are formed where fibers in the web intersect) make the nanofiber web stronger and stiffer such that the mechanical strength and hydration stability of the PEM can be significantly improved once the PEM has been reinforced with the inventive nanofiber web. When creating the joints using a solvent, the formation of the welded joints can be controlled by selection of

solvent and solvent evaporation rate. Alternatively, welded (melted) joints can be created using a post heat treatment at the temperature close to or just higher than the polymer melting point.

In another embodiment of the invention, nanofiber webs comprising impregnated additives are prepared using electro-spinning technology from a polymer solution which additionally comprises the desired additive or additives in solution. In a fuel cell using a PEM with a reinforcement matrix comprising such a nanofiber web with a suitable incorporated additive, the chemical stability of PEM can be increased as the additive is slowly released from inside the nanofiber matrix.

Specifically, the invention includes an electrospun nanofiber web in which a plurality of the fibers in the web have been welded together at their intersection points thereby forming a plurality of welded joints. The invention also includes an electrospun nanofiber web comprising an additive impregnated into the fibers of the web. In either embodiment, the web fibers can comprise polyvinylidene fluoride, polybenzimidizole, polyimide, polysulfone, polyamideimide, or polypropylene.

Suitable additives for use in the invention include a free radical scavenging inorganic material or a hydrogen peroxide decomposition catalyst, e.g. CeO_2, RuO_2, WO_3, $CePO_4$, $Ce(NO_3)_3$, CeF_3, $Ce(SO_4)_2$, MnO_2, Mn_2O_3, MnO, $MnSO_4$, $MnCl_2$, $Mn(CH_3COO)_2{\cdot}4H_2O$, $Mn(NO_3)_2$, $CoCl_2$, $Co(NO_3)_2$, $CoBr_2$, $Co_3(PO_4)_2$, $Co(CH_3COO)_2$, $CoSO_4$, $Co(H_2PO_4)_2$, Pt, and Pt-Co alloy. Suitable additives also include an organic molecule or polymer comprising a free radical scavenging antioxidant, a hydrogen peroxide decomposition catalyst, or a Fenton metal ion chelating agent, e.g. 1,10-phenanthroline, bathophenanthroline, 1,10-phenanthroline-5-amino, 8-hydroxyquinoline, and N,N'-Bis(salicylidene) ethylenediamine. Further, suitable additives also include complex additives comprising a metal component and an organic component. Such complex additives can be denoted as (metal)(ligand) where (metal) refers to a metal component and (ligand) refers to an organic component. The metal component can for instance be those cited above for use as a free radical scavenging inorganic material or a hydrogen peroxide decomposition catalyst. The ligand component can for instance be those cited above for use as an organic molecule, a free radical scavenging antioxidant polymer, a hydrogen peroxide decomposition catalyst, or a Fenton metal ion chelating agent.

In another aspect, the invention includes a proton exchange membrane for a fuel cell comprising a reinforcement matrix comprising the aforementioned electrospun nanofiber web. In such a reinforced PEM, the proton exchange material can comprise perfluorosulfonic acid ionomer or hydrocarbon type ionomer.

The invention also includes a solid polymer electrolyte fuel cell comprising the aforementioned proton exchange membrane.

These and other aspects of the invention are evident upon reference to the attached Figures and following detailed description.

BRIEF DESCRIPTION OF THE DRAWINGS

Figures 1a and 1b show magnified electron microscope images respectively of a conventional nanofiber web and of a similar nanofiber web except comprising welded joints.

Figure 2 schematically illustrates the controlled release of an additive from inside a reinforcing nanofiber web into the surrounding ionomer of the PEM.

Figure 3 shows the chemical structure of suitable exemplary bipyridine type ligand additives.

Figure 4 shows the chemical structure of suitable exemplary bipyridine type polymer additives.

Figures 5a to 5c show the chemical structure of several suitable exemplary heterocycle quinoline ligands and salen additives.

Figures 6a to 6e show the chemical structure of several suitable exemplary metal complex additives.

Figure 7 shows the UV-vis absorption spectra of a Nafion membrane in the Examples before and at various times after contacting it with a PVDF film comprising impregnated additive in water at 50 °C.

Figure 8 shows the UV-vis absorption strength at 250 nm versus time of the Nafion membrane of Figure 7 before and after contacting it with a PVDF film comprising impregnated additive.

Figure 9 shows the UV-vis absorption spectra of the PVDF film comprising impregnated additive before and after contacting it with the Nafion membrane of Figure 7.

Figures 10 shows the UV-vis absorption strength at 250 nm versus time of the PVDF film comprising impregnated additive of Figure 9. The dashed line indicates the absorption of a similar PVDF film but without any impregnated additive.

DETAILED DESCRIPTION

Herein, an electrospun nanofiber web refers to either a woven or non-woven web formed from nanofibers that have been prepared by electrospinning a suitable polymer solution or dispersion.

In one embodiment, the reinforcement matrix webs of the present invention are made of electrospun nanofibers comprising "welded" joints and thus differ from the conventional nanofiber webs previously reported as reinforcing matrices for PEMs in fuel cells (e.g. as in WO2014104785A1 and US20150303505A1) or the webs used as air and water filters (e.g. as in US20120137885). Welded joints in the web make the nanofiber reinforcement matrix stronger and stiffer so that the mechanical strength and hydration stability of the PEM will be significantly improved after having been reinforced with the novel nanofiber web. The formation of welded joints in the web can be controlled by appropriate selection of a dissolving solvent and the solvent evaporation rate or alternatively a post heat treatment at a temperature close to or higher than the polymer melting point can be employed. Figures 1a and 1b show magnified SEM images of a conventional nanofiber web and a nanofiber with welded joints respectively.

In WO2014104785A1 of Kolon Industries Inc,, a PEM reinforced with conventional nanofiber web lasts less than 250 hours in hydration operation. Obviously, PEMs reinforced with such conventional nanofiber webs need further improvement in durability.

In the present case, the nanofiber web formed from a suitable polymer solution can be a continuous web having porosity from 50% to 90%. The area weight can be from 1 to 5 gram per square meter. The fiber diameter can be from 5 nm to 2000 nm. The fibers form either a woven or non-woven porous web. Suitable polymers for use in fabricating nanofiber webs can be selected from the group consisting of polyvinylidene fluoride (PVDF), polybenzimidizole (PBI), polyimide (PI), polysulfone (PS), polyamideimide (PAI), and polypropylene (PP).

In other embodiments, additives are incorporated into the reinforcing nanofiber webs. These additives can be either co-dissolved or dispersed in the polymer solution used to electrospin the web fibers. The solution or dispersion can then be electrospun to prepare nanofibers with incorporated additive and a web is then

formed using these fibers. Alternatively, additives can be also incorporated into the nanofibers by a post treatment method, e.g. by soaking a conventional nanofiber web in a solution containing dissolved additive. In a PEM comprising such a nanofiber web, the gradual release of a suitable incorporated additive from the nanofiber web into the surrounding ionomer layers can be used to increase the chemical stability of PEM. For instance, additives that act as radical scavengers, antioxidants or chelating Fenton ions can improve the chemical stability and durability of the PEM. Figure 2 schematically illustrates the controlled release (retarded release) of an additive from inside a reinforcing nanofiber web and into the surrounding ionomer layers 1, 2 of the PEM.

The additive concentration in the nanofibers can be from 1 wt% to 50 wt% to that of the nanofiber polymer. By incorporating additive in the nanofiber web, less total additive can be used to obtain the same results as those obtained when the additive is incorporated instead into the PEM layers 1 and 2 directly. Because less additive is used, it is expected that there will be less of a "key-on issue" or adverse effect on conditioning time in fuel cells made with such PEMs, without compromising durability.

Suitable additives for use in the present invention include:

1) certain inorganic materials such as a free radical scavenger or a hydrogen peroxide decomposition catalyst, such as CeO_2, RuO_2, WO_3, $CePO_4$, $Ce(NO_3)_3$, CeF_3, $Ce(SO_4)_2$, MnO_2, Mn_2O_3, MnO, $MnSO_4$, $MnCl_2$, $Mn(CH_3COO)_2{\cdot}4H_2O$, $Mn(NO_3)_2$, $CoCl_2$, $Co(NO_3)_2$, $CoBr_2$, $Co_3(PO_4)_2$, $Co(CH_3COO)_2$, $CoSO_4$, $Co(H_2PO_4)_2$, Pt, and Pt-Co alloy;
2) certain organic molecules or polymers such as a free radical scavenger antioxidant, a hydrogen peroxide decomposition catalyst, or a Fenton metal ion chelating agent, such as 1,10-phenanthroline, bathophenanthroline, 1,10-phenanthroline-5-amino, 8-hydroxyquinoline, N,N'-Bis(salicylidene) ethylenediamine (salen) (Figures 3, 4 and 5 show the chemical structure of suitable exemplary bipyridine type ligand additives, bipyridine type polymer additives, and heterocycle quinoline ligands and salen additives respectively); and/or
3) complex additives denoted as (metal)(ligand) where (metal) refers to a metal component such as those in 1) above and (ligand) refers to an organic component such as those in 2) above (Figure 6 shows the chemical structure of several suitable exemplary metal complex additives).

Compared to conventional e-PTFE or nanofiber webs used as a reinforcement matrix for fuel cells, the nanofiber webs of the invention comprising welded joints and/or impregnated additives can provide several advantages including:

1) Electrospun nanofiber webs with welded joints and/or additives can improve both PEM mechanical properties (mechanical reinforcement) and chemical stability (via release of additives from the nanofiber);
2) A conventional e-PTFE web has a hydrophobic surface and thus is not suited for use as a reinforcement matrix for a hydrocarbon ionomer; but an electrospun hydrocarbon nanofiber web can be used for hydrocarbon ionomer reinforcement;
3) The typical additive used to improve PEM chemical stability is mobile in the membrane during fuel cell operation. Additives incorporated directly into the membrane can thus be washed out or redistributed in the membrane, thereby resulting in a decrease in PEM durability. But for instance, hydrocarbon nanofiber can be electrospun from a solution comprising an additive. The electrospun hydrocarbon nanofiber web made therefrom contains additive inside the fibers, which is less mobile and, with a controlled release rate into the PEM ionomer layer, can improve PEM chemical stability;
4) Organic ligand or complex additives can significantly improve membrane durability without compromising performance (as disclosed in US 9,172,107 and US 9,391,337), but MEAs comprising such additives typically need longer conditioning times (i.e. the "key-on issue"). Using electrospun nanofiber webs as the additive carrier can mitigate the key-on issue to reduce conditioning time;
5) The cost of a conventional e-PTFE reinforcement matrix is high. The electrospun hydrocarbon nanofiber web of the invention can be a lower cost option for a reinforcement matrix;
6) A conventional fluoropolymer e-PTFE web has high reactant (hydrogen and oxygen) crossover. However, an electrospun hydrocarbon nanofiber web has lower reactant crossover compared to e-PTFE.

EXAMPLES

A complex additive of cerium and 8-hydroxyquinoline (i.e. as shown in Figure 6d) was synthesized as described in L. Li, F.G. Yuan, T.T Li, Y. Zhou, M.M. Zhang, Inorganica Chimica Acta, 397, 69-74(2013). The complex additive was dissolved in methyl ethyl ketone with PVDF polymer in a weight ratio of 1 to 12, and then cast into a 20 μm film, thereby forming a PVDF film impregnated with additive.

The film was then sandwiched between two Nafion (NRE211) membranes, held tightly in a jig, and placed in hot water (50 °C) to allow the additive to gradually release from the impregnated PVDF film into the Nafion membranes. This release was measured using a UV-Vis technique. The cerium and 8-hydroxyquinoline complex shows absorption in the UV-Vis spectrum starting from 400 nm and a peak at

around 250 nm of wavelength. The intensity of this peak was used to analyze the additive concentration in both the Nafion and the PVDF films over time.

The experiment was carried out for 100 hours in total. Nafion and PVDF film samples were taken for UV-Vis spectroscope analysis before creating the sandwich (i.e. 0 hours) and also after intervals of 3, 6, 27, 54 and 100 hours of exposure to the hot water. The UV-Vis absorption spectra of the Nafion membrane samples at the various time intervals are shown in Figure 7. Figure 8 shows the UV-vis absorption strength versus time. Before contacting the Nafion with the impregnated PVDF film (i.e. at 0 hour), no absorption is seen at the 250 nm wavelength in the Nafion membrane. A substantial increase in absorption intensity after 27 hours indicates that there is a quick release of the additive from the impregnated PVDF film to the Nafion membrane, and thereafter the additive release slows down with the increase in additive concentration in the Nafion membrane.

On the other hand, the additive impregnated PVDF film shows the strongest absorption at 0 hour. The absorption due to the presence of additive in the PVDF film decreases with the release of additive into the Nafion membrane. This is evidenced in the UV-vis absorption spectra and UV-vis absorption strength versus time data shown in Figures 9 and 10 respectively. The release of additive isn't complete at 100 hours. This is evident when comparing the UV-Vis spectrum of the 100 hour sample to that of a PVDF film with no additive (which is also plotted in Figure 10). A certain amount of additive thus still remained in the PVDF film after 100 hours. The release of additive from the Nafion membrane into water is very slow due to the strong interaction between the sulfonic acid groups of the Nafion and the complex additive, as well as due to the poor solubility of the complex additive in water. UV-Vis spectroscopy was unable to detect the additive concentration in the hot water in this experiment.

While particular elements, embodiments and applications of the present invention have been shown and described, it will be understood, of course, that the invention is not limited thereto since modifications may be made by those skilled in the art without departing from the spirit and scope of the present disclosure, particularly in light of the foregoing teachings.

What is claimed is:

1. An electrospun nanofiber web wherein a plurality of the fibers in the web have been welded together at their intersection points thereby forming a plurality of welded joints.

2. An electrospun nanofiber web comprising an additive impregnated into the fibers of the web.

3. The electrospun nanofiber web of claims 1 or 2 wherein the fibers of the web comprise polyvinylidene fluoride, polybenzimidizole, polyimide, polysulfone, polyamideimide, or polypropylene.

4. The electrospun nanofiber web of claim 2 wherein the additive is a free radical scavenging inorganic material or a hydrogen peroxide decomposition catalyst.

5. The electrospun nanofiber web of claim 4 wherein the additive is selected from the group consisting of CeO_2, RuO_2, WO_3, $CePO_4$, $Ce(NO_3)_3$, CeF_3, $Ce(SO_4)_2$, MnO_2, Mn_2O_3, MnO, $MnSO_4$, $MnCl_2$, $Mn(CH_3COO)_2 \cdot 4H_2O$, $Mn(NO_3)_2$, $CoCl_2$, $Co(NO_3)_2$, $CoBr_2$, $Co_3(PO_4)_2$, $Co(CH_3COO)_2$, $CoSO_4$, $Co(H_2PO_4)_2$, Pt, and Pt-Co alloy.

6. The electrospun nanofiber web of claim 2 wherein the additive is an organic molecule or polymer comprising a free radical scavenging antioxidant, a hydrogen peroxide decomposition catalyst, or a Fenton metal ion chelating agent.

7. The electrospun nanofiber web of claim 6 wherein the additive is selected from the group consisting of 1,10-phenanthroline, bathophenanthroline, 1,10-phenanthroline-5-amino, 8-hydroxyquinoline, and N,N'-Bis(salicylidene) ethylenediamine.

8. The electrospun nanofiber web of claim 2 wherein the additive is a complex additive denoted as (metal)(ligand) where (metal) refers to a metal component like those in claim 5 and (ligand) refers to an organic compound like those in claim 7.

9. A proton exchange membrane for a fuel cell comprising a reinforcement matrix comprising the electrospun nanofiber web of claims 1 or 2.

10. The proton exchange membrane of claim 9 wherein the proton exchange material comprises perfluorosulfonic acid ionomer or hydrocarbon type ionomer.

11. A solid polymer electrolyte fuel cell comprising the proton exchange membrane of claim 9.

Docket No. 2016P03884CA
Title: Electrospun Nanofiber Webs For Membrane Reinforcement In Fuel Cells
Inventors: J. Li et al.

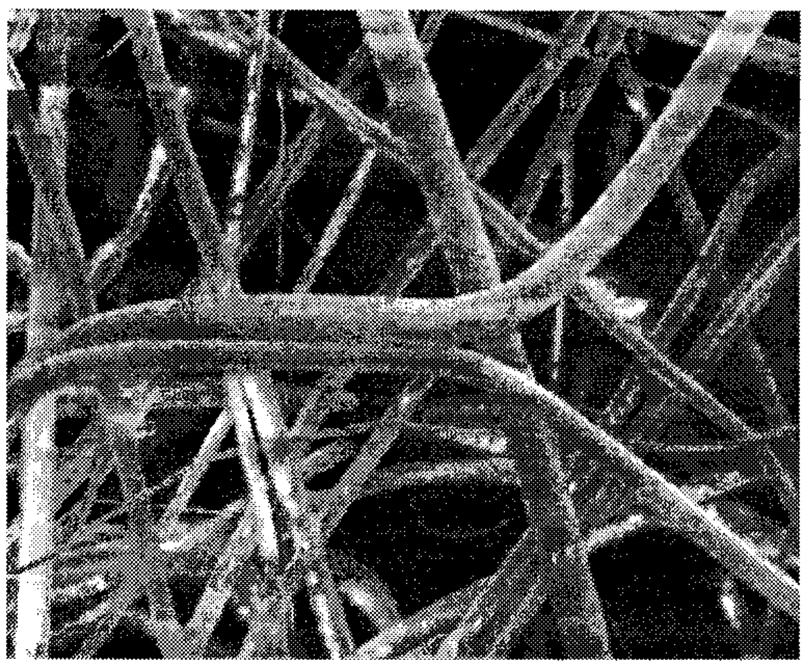


FIG. 1a

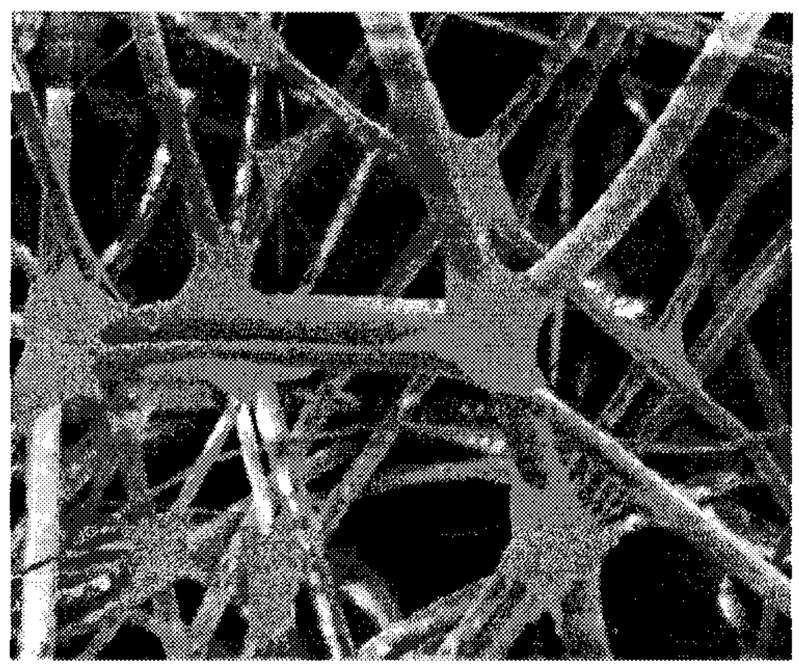


FIG. 1b

Docket No. 2016P03884CA
Title: Electrospun Nanofiber Webs For Membrane Reinforcement In Fuel Cells
Inventors: J. Li et al.

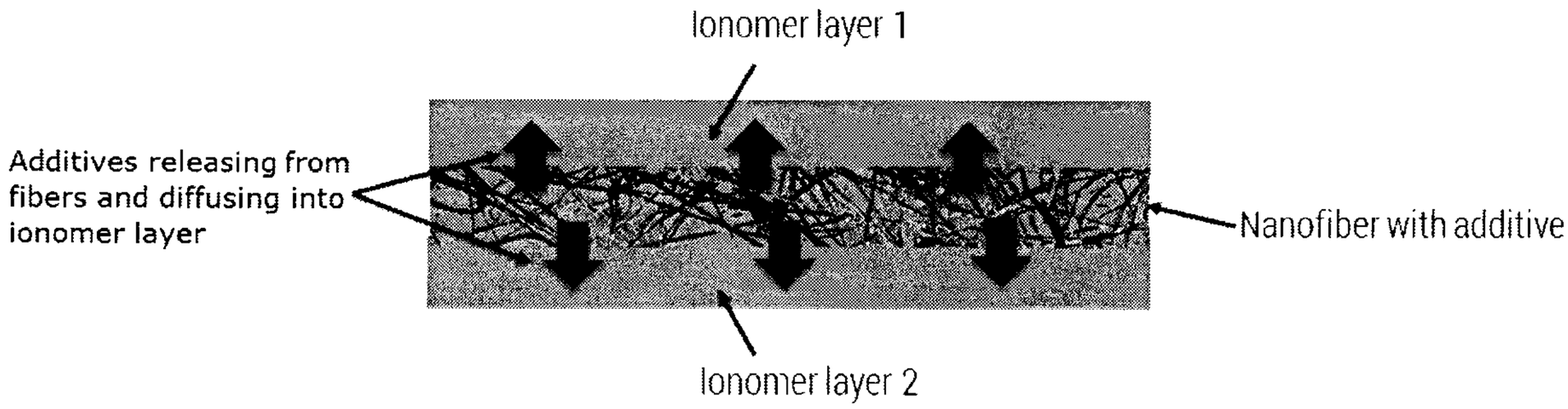
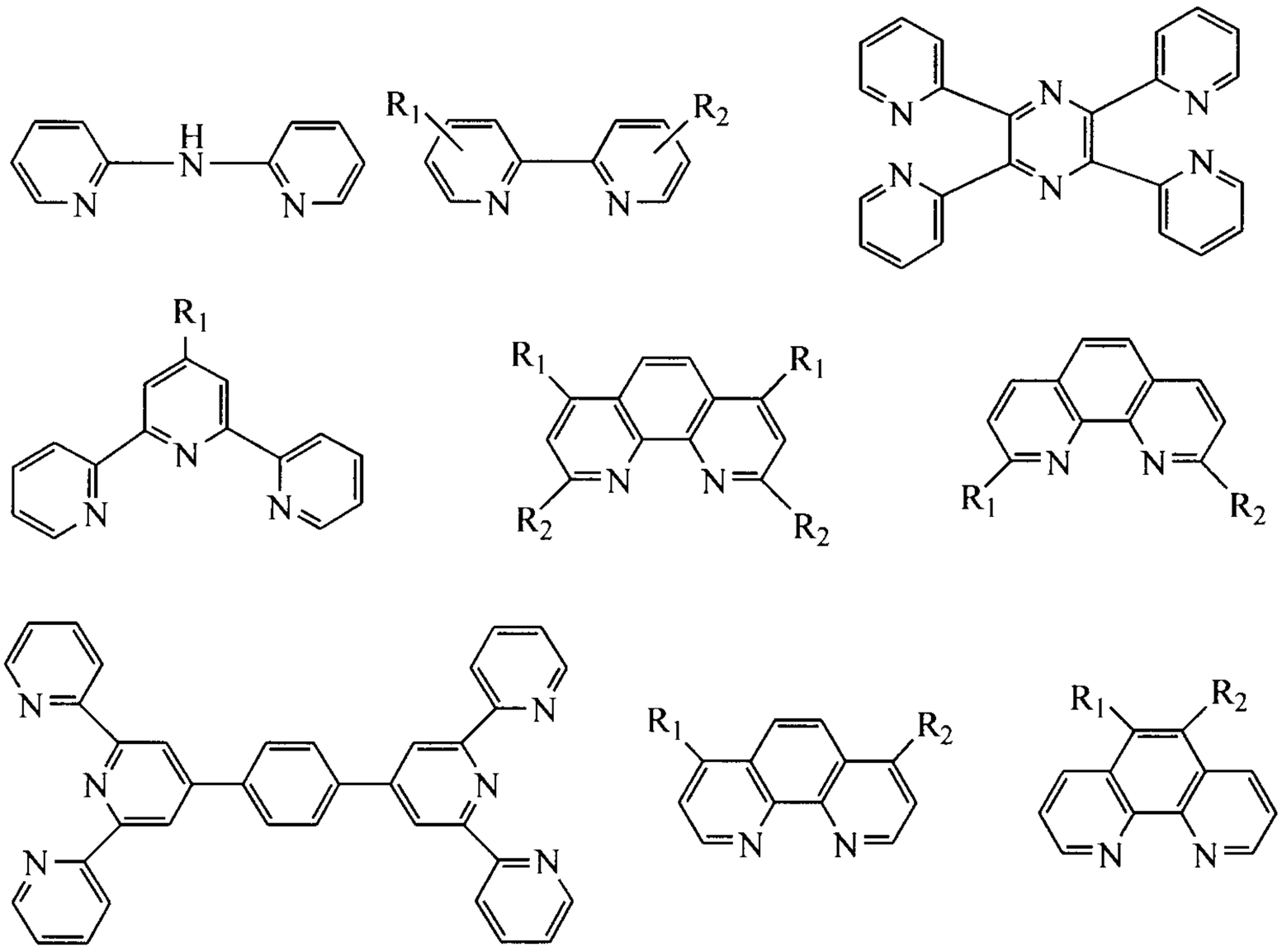
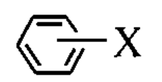


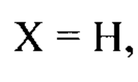
FIG. 2



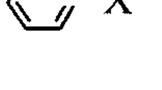
R_1 = H, $CH_3(CH_2)_n$, $CH_3(CH_2)_nO$, COOH, $PO(OH)_2$, SO_3H, NH_2, OH,



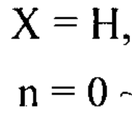
X X = H, COOH, $PO(OH)_2$, SO_3H,



R_2 = H, $CH_3(CH_2)_n$, $CH_3(CH_2)_nO$, COOH, $PO(OH)_2$, SO_3H, NH_2, OH,



X X = H, COOH, $PO(OH)_2$, SO_3H,



n = 0 ~ 10

FIG. 3

Docket No. 2016P03884CA
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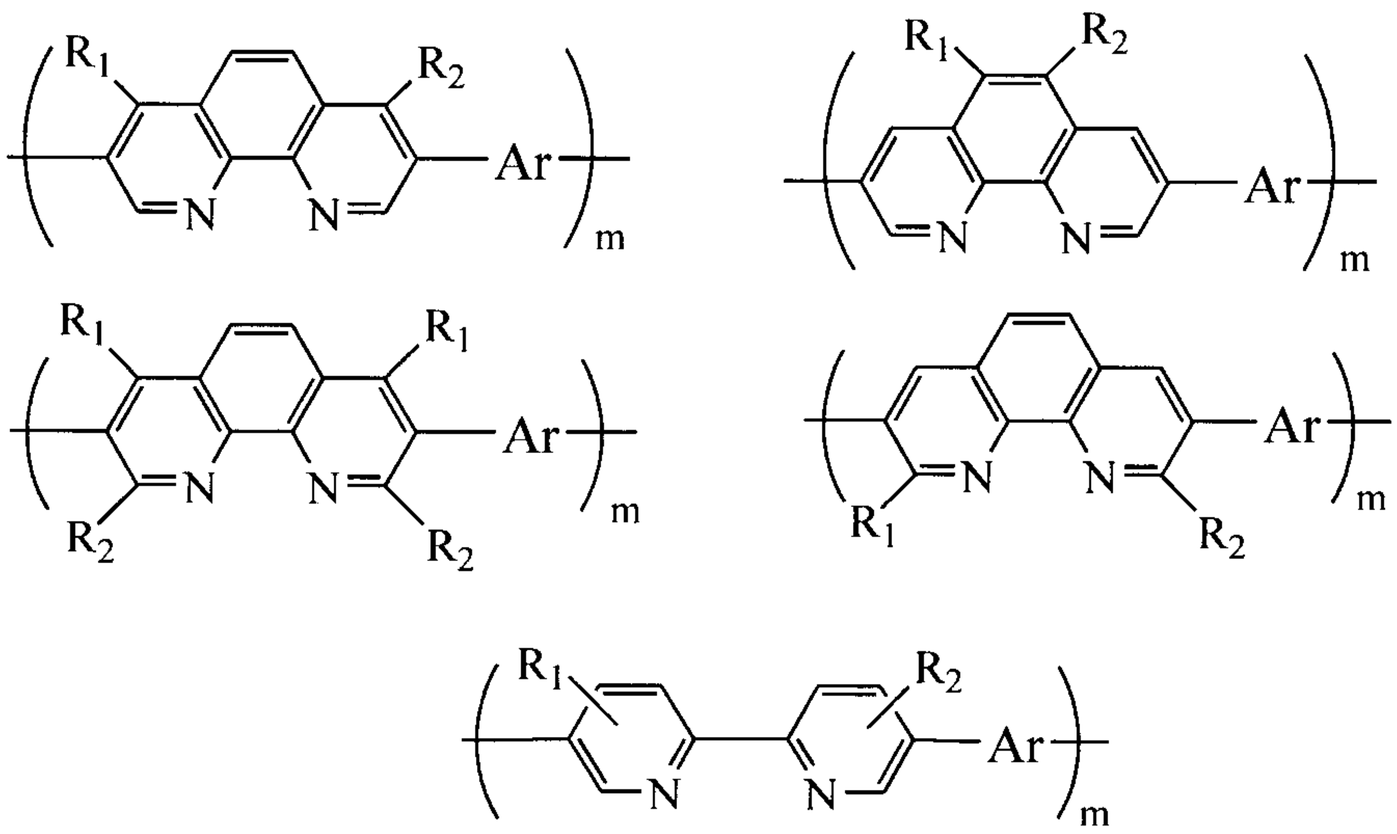
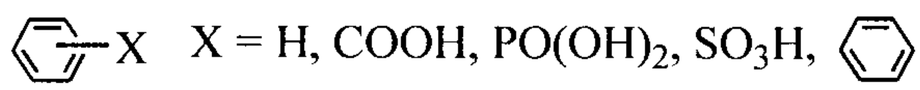
Inventors: J. Li et al.



R_1 = H, $CH_3(CH_2)_n$, $CH_3(CH_2)_nO$, COOH, $PO(OH)_2$, SO_3H, NH_2, OH,

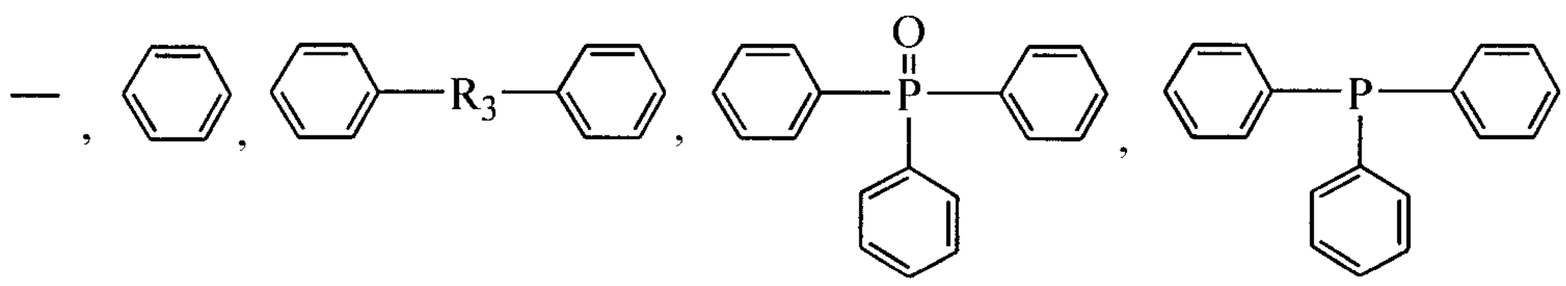
X = H, COOH, $PO(OH)_2$, SO_3H,

R_2 = H, $CH_3(CH_2)_n$, $CH_3(CH_2)_nO$, COOH, $PO(OH)_2$, SO_3H, NH_2, OH,



X = H, COOH, $PO(OH)_2$, SO_3H,

Ar:



R_3 = $(CH_2)_k$, $(CF_2)_k$, O, S, $C(CF_3)_2$, $C(CH_3)_2$,

n = 0 ~ 10; m = 2 ~ 100; k = 0 ~ 10

FIG. 4

Docket No. 2016P03884CA
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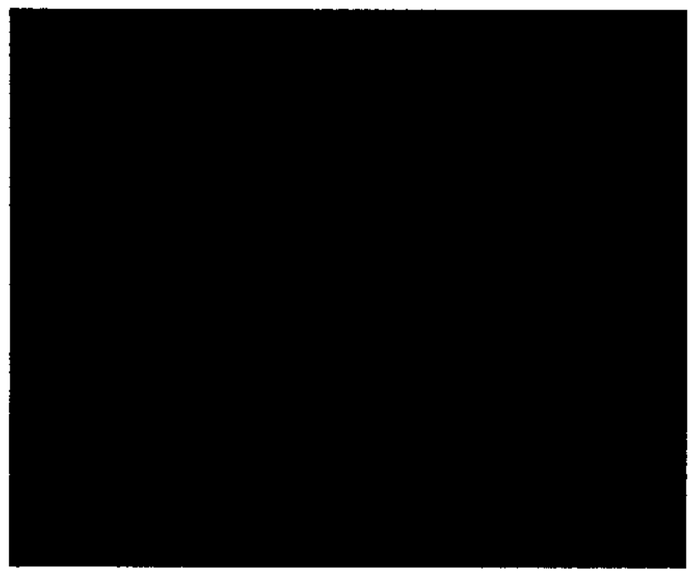
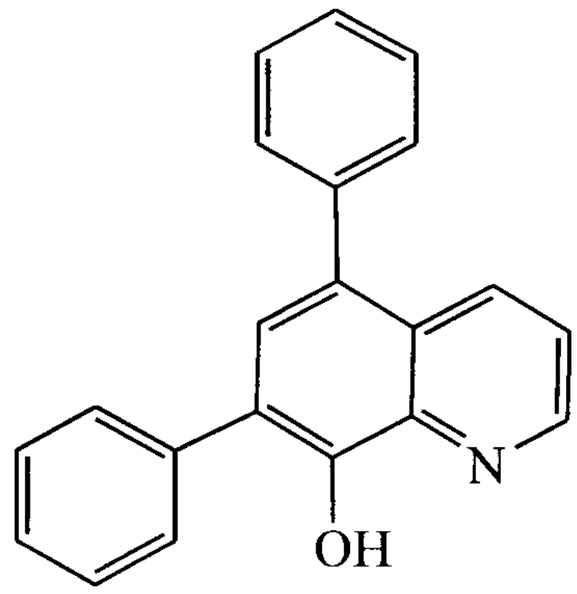
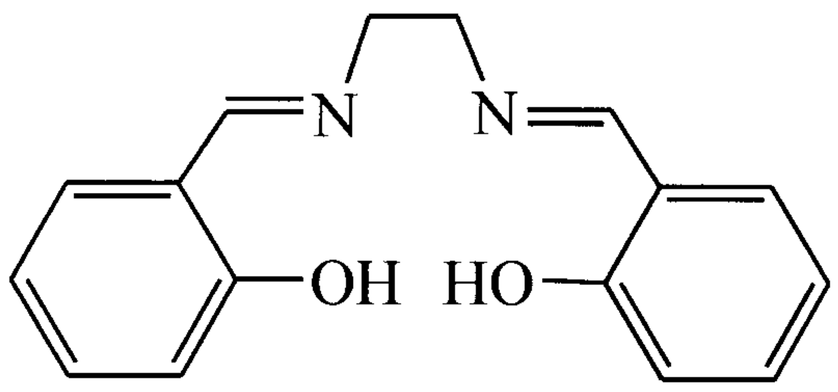
Inventors: J. Li et al.



(a)

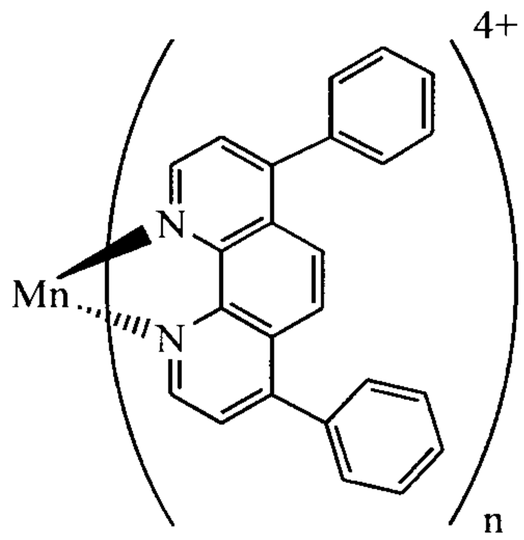


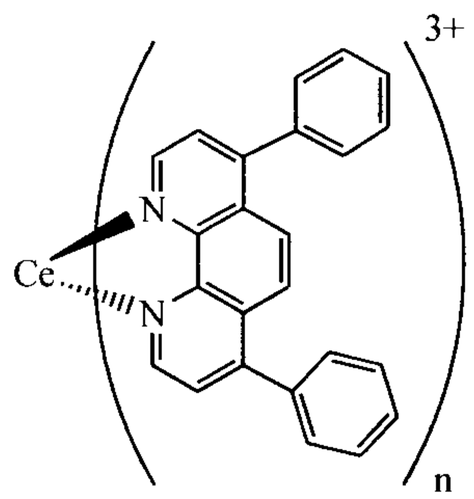
(b)



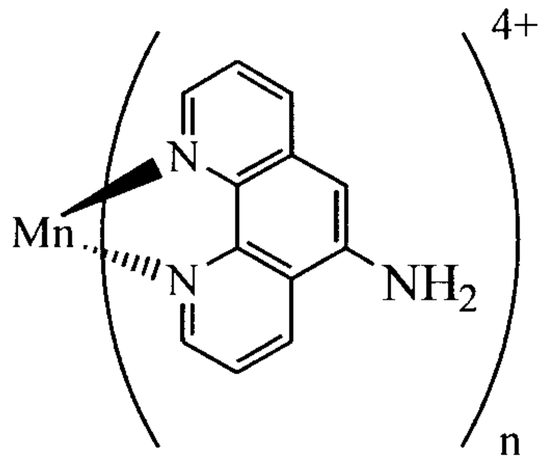
(c)

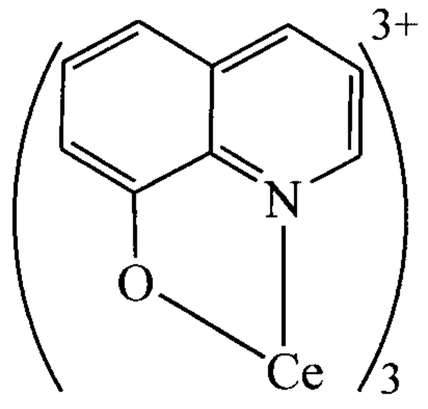
FIG. 5



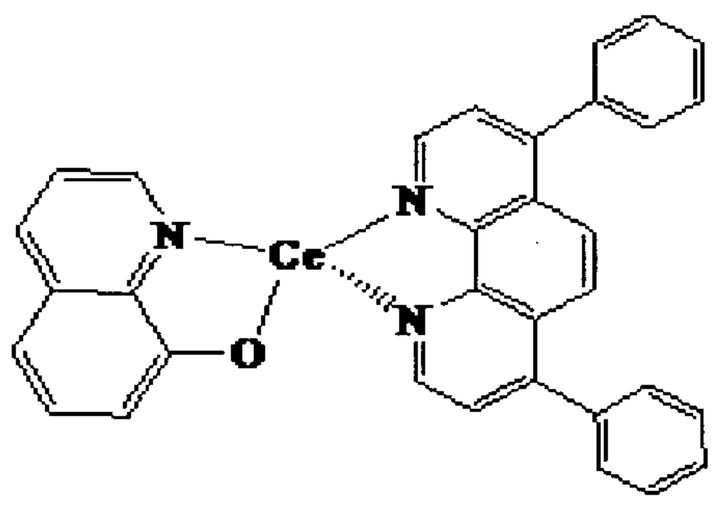


(a) **(b)**





(c) **(d)**



(e)

FIG. 6

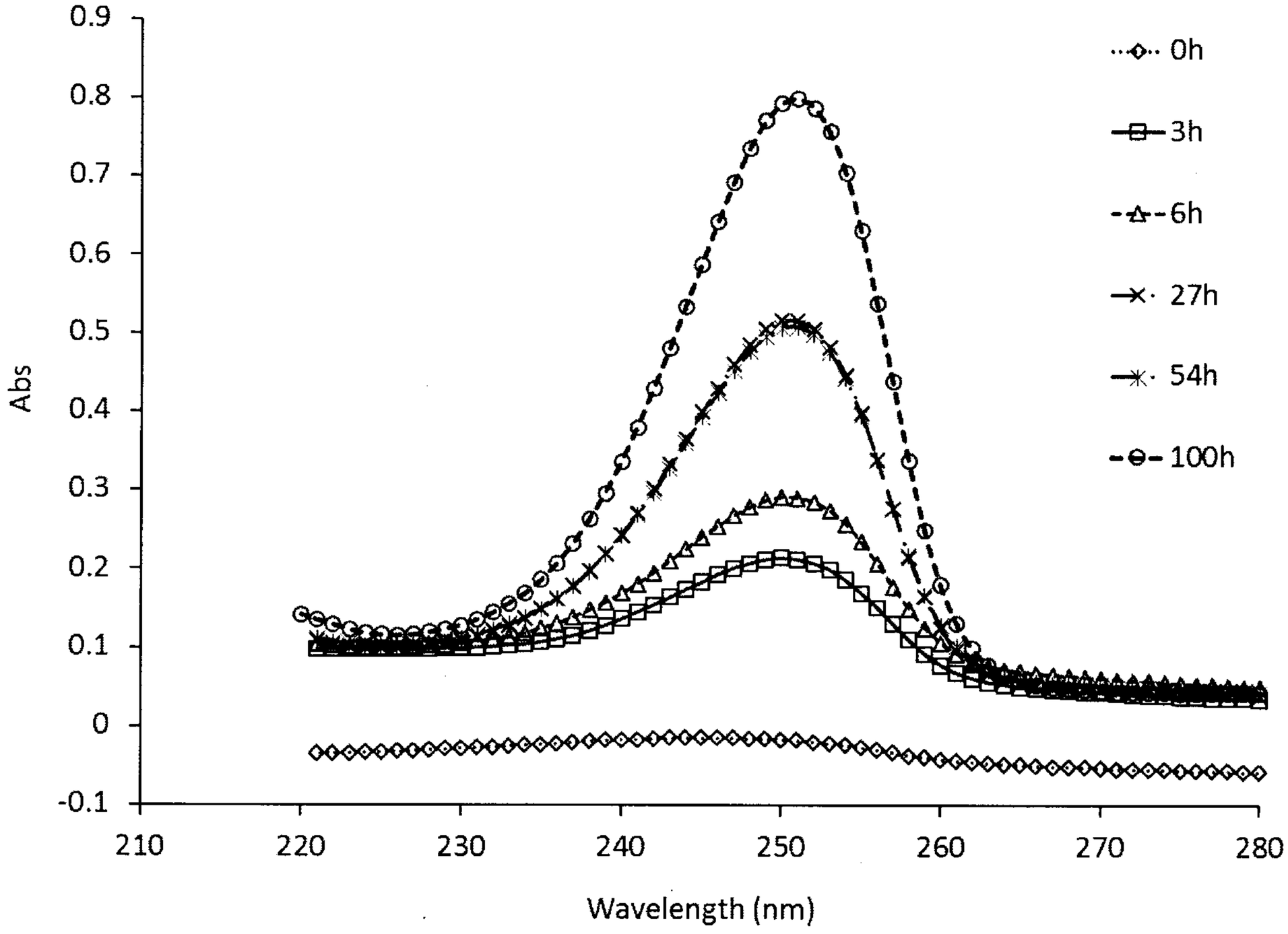

0h
3h
6h
27h
54h
100h
Abs
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
-0.1
210
220
230
240
250
260
270
280
Wavelength (nm)

FIG. 7

Docket No. 2016P03884CA
Title: Electrospun Nanofiber Webs For Membrane Reinforcement In Fuel Cells
Inventors: J. Li et al.

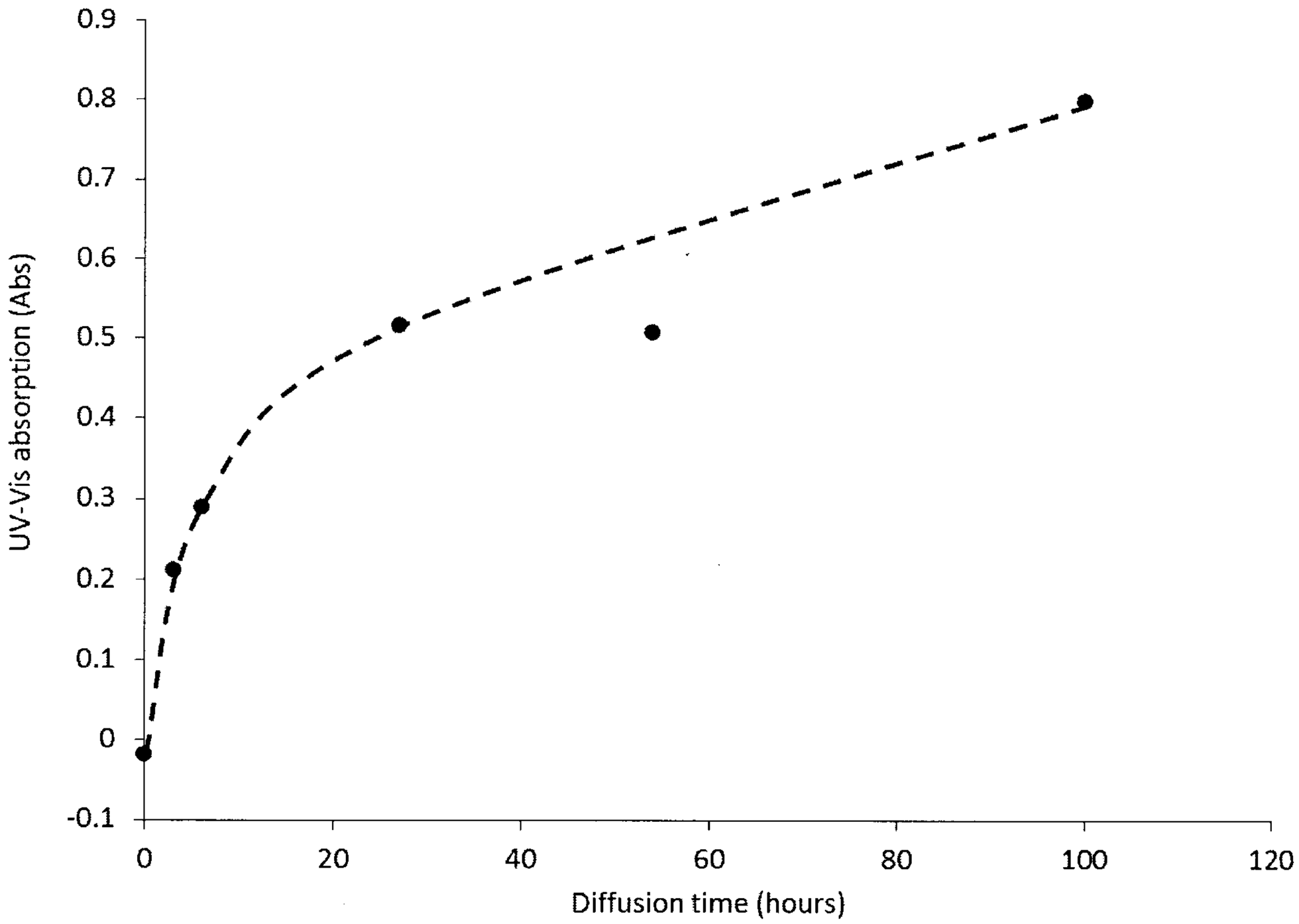


FIG. 8

Docket No. 2016P03884CA
Title: Electrospun Nanofiber Webs For Membrane Reinforcement In Fuel Cells
Inventors: J. Li et al.

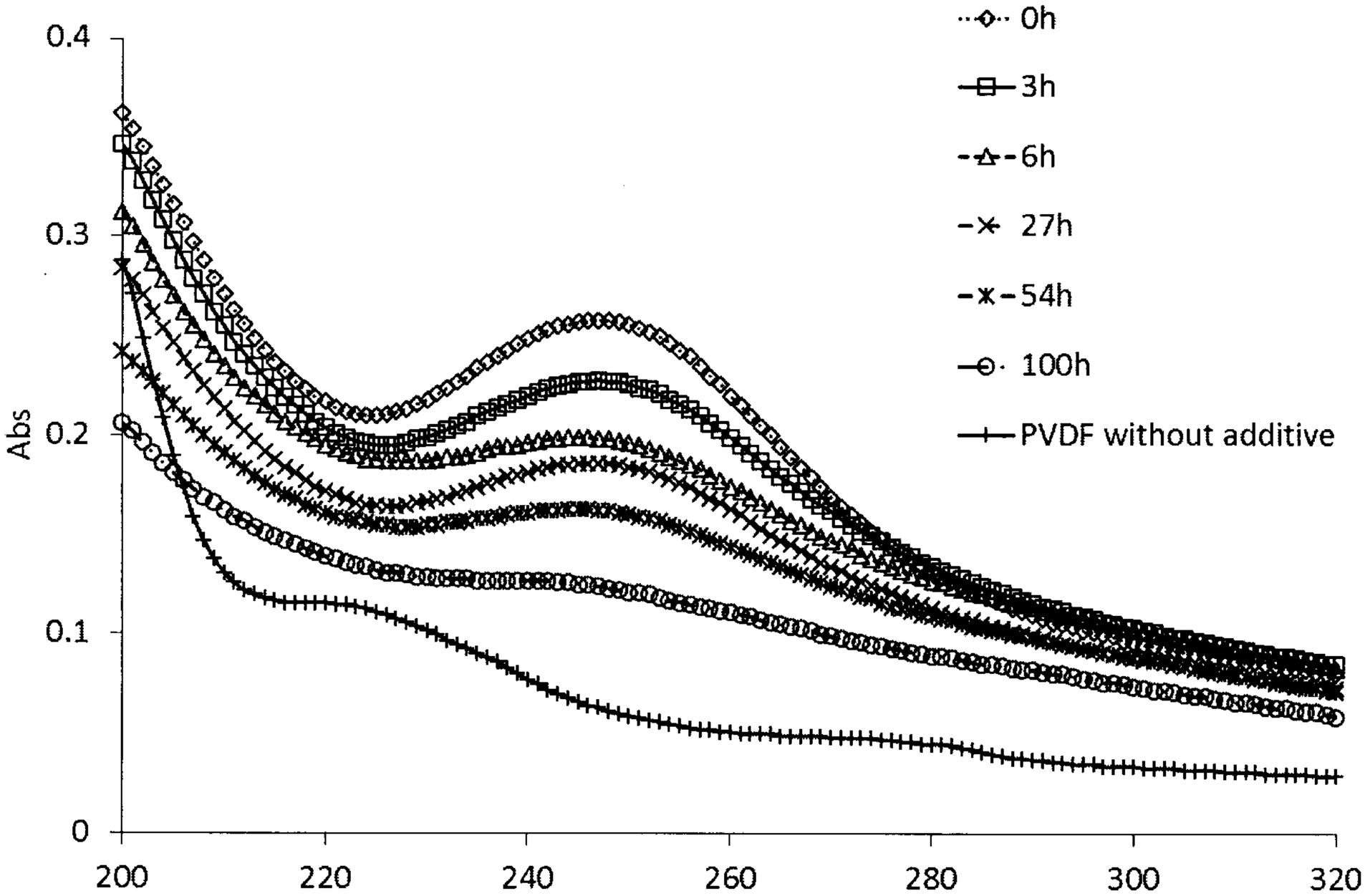


FIG. 9

Docket No. 2016P03884CA
Title: Electrospun Nanofiber Webs For Membrane Reinforcement In Fuel Cells
Inventors: J. Li et al.

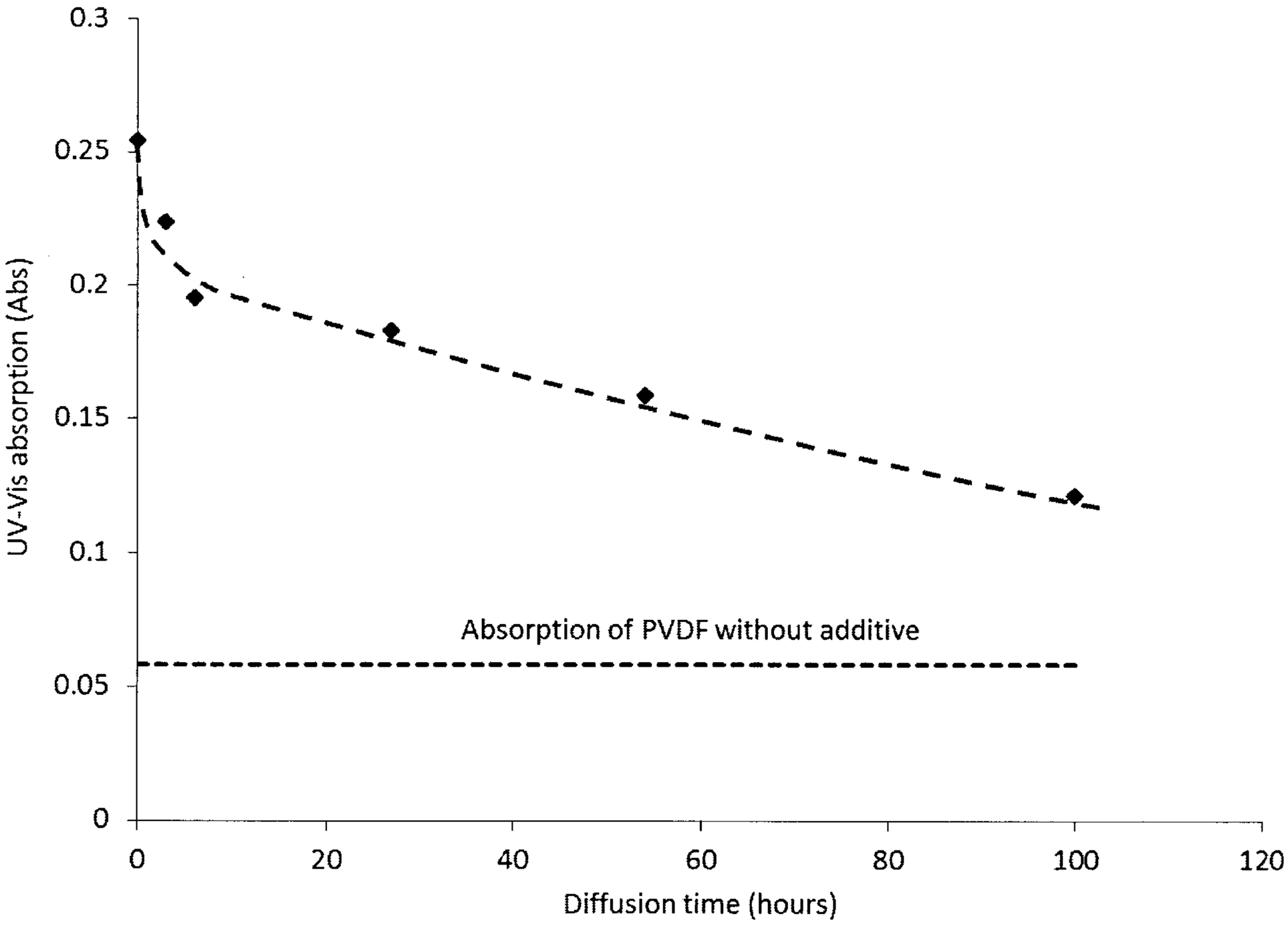


FIG. 10