

PROCESS FOR PRODUCING CATALYST LAYER WITH BIOCHAR-BASED CARBON SUPPORT AND INTEGRATED PLATINUM

Technical Field

5 The present utility model concerns fuel cell technologies, particularly to a process for producing a catalyst layer for proton exchange membrane fuel cells.

Background of the Utility Model

10 The use of fossil fuels, particularly in transportation, has significantly contributed to pollution, global warming, and ultimately climate change. Vehicle emissions release pollutants that harm human health and contain greenhouse gases, which exacerbate climate change. As a result, there has been increasing
15 interest in finding cleaner energy alternatives to mitigate the harmful effects of fossil fuel consumption. One such solution that has been extensively explored is hydrogen technology, particularly the use of fuel cells. Fuel cells are a promising clean energy source that generates electricity from the
20 oxidation of hydrogen. In this process, electrons move through an external circuit, generating electricity. There are various types of fuel cells, including polymer electrolyte membrane fuel cells (PEMFC), direct methanol fuel cells, alkaline fuel cells, and reversible fuel cells. The present utility model focuses on
25 the use of biomass waste, particularly as a carbon support for PEMFCs.

 One of the main challenges of using fuel cells is their high cost and limited durability. The commercialization of PEMFCs, especially for vehicles, is hindered by their high cost,
30 largely due to the substantial amount of platinum required in the electrodes as a catalyst. Therefore, it is important to prioritize finding alternative materials for the components of the fuel cell, beyond just the catalyst. The catalyst is

typically supported by a layer to prevent particle agglomeration, which can lower the fuel cell's energy output. This support layer needs to be conductive, porous, and have a high surface area to enhance the stability of the catalyst.

5 Platinum-based catalysts are often deposited on carbon supports, which have been shown to improve catalytic stability.

As part of the development of fuel cell technology, biomass-derived carbons are being explored as cost-effective electrode alternatives that also improve sustainability. In this utility

10 model, water hyacinth (WH) was used as the biomass waste source for carbon in the catalyst layer. Numerous studies have shown that WH is an invasive species that negatively impacts water quality. Therefore, investigating the carbonization of WH and using it as a carbon support in the catalyst layer is highly

15 significant, as it contributes to sustainability and the advancement of fuel cell technology.

The method of incorporating platinum particles into the catalyst layer is also crucial in fuel cell synthesis. The conventional method of integrating the catalyst layer onto the

20 membrane is spray coating, where carbon ink with the catalyst (or catalyst with a solvent) is sprayed onto the membrane, as used in previous studies. In this utility model, an alternative method inspired by Lafforgue (2025) was adapted. This method involves wetting the platinum powder with carbon ink and then

25 manually casting it onto the membrane.

CN111054423A provides a nitrogen self-doped porous carbon catalyst as well as a preparation method and application thereof. The preparation method of the nitrogen self-doped porous carbon catalyst comprises the following steps of: 1) mixing biomass

30 aquatic algae with an active agent solution to obtain a mixture A, and concentrating the mixture A to obtain paste, 2) pyrolyzing the product obtained in the step 1) in an inert atmosphere, and 3) performing surface treatment on a solid product obtained by

pyrolysis by using concentrated nitric acid or aqua regia to obtain the catalyst.

CN115498199A relates to a PtCu alloy/G-bacterium modified rGO composite catalytic material which comprises a G-bacterium modified rGO substrate and PtCu alloy particles compounded on the substrate.

CN105375042A discloses a biomass carbon catalyst and a preparation method and application thereof. The precursor of the catalyst comprises biomass and metal salts, wherein the biomass includes one or more of maize straw, corncob, rice straw, cotton straw, peanut straw, peanut shell, wheat hull, rice hull, wood, sawdust, bamboo wood and weed. Through controlling addition of metal salts in different amounts, adjusting impregnation concentration of the precursor and reaction ambient and carbonization and nitriding treatment temperatures, the biomass carbon catalyst with controllable and adjustable pore structure and specific surface area can be prepared.

Summary of the Utility Model

The objective of the present utility model is to provide a simplified method for integrating platinum into a carbon support to form an efficient catalyst layer for proton exchange membrane fuel cells (PEMFCs). Another objective is to enhance the performance and durability of the catalyst layer for improved suitability in PEMFC applications. A further objective is to demonstrate the applicability of the improved catalyst layer in a multi-cell PEMFC stack by assessing its voltage output.

Accordingly, the present utility model provides a process for producing a catalyst layer for proton exchange membrane fuel cells (PEMFCs). The process utilizes biochar derived from water hyacinth (*Eichhornia crassipes*) as a carbon support material, onto which a platinum catalyst and a polymeric binder are incorporated. The method includes dispersing the biochar in a

solvent, homogenizing and sonicating the solution, integrating a platinum catalyst, and incorporating a binder to obtain a catalyst suspension. This suspension is then coated onto a substrate and dried to form the catalyst layer. The process enables uniform distribution of platinum within the carbon support, improves catalytic performance, and ensures a durable and thermally stable catalyst layer suitable for PEMFC applications.

10 **Brief Description of the Drawings**

The accompanying drawings, which are included to provide a further understanding of the present utility model, are incorporated herein to illustrate embodiments of the present utility model. Along with the description, they also explain the principle of the present utility model and are not intended to be limiting.

FIG. 1 shows a comparison of catalyst layer morphology using two different methods, (A) spray coating method, and (B) wetting method.

20 FIG. 2 shows SEM images comparing catalyst layer morphology at 100,000x magnification, (A) spray coating method, and (B) wetting method.

FIG. 3 shows the catalyst layer after 24-hour oxidative soaking in 3% hydrogen peroxide.

25 FIG. 4 shows the EDX spectra of the catalyst layer after oxidative soaking.

FIG. 5 shows the catalyst layer after 1-hour boiling test at 100°C.

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Detailed Description of the Utility Model

The present utility model provides a process for producing a catalyst layer specifically designed for proton exchange membrane fuel cells (PEMFCs).

5 In one embodiment, biochar is obtained from water hyacinth (*Eichhornia crassipes*), which is dried, carbonized, and ground to a fine powder of suitable particle size. The biochar is dispersed in a solvent, preferably N-Methyl-2-pyrrolidone (NMP), to form a carbon solution. The carbon solution is homogenized,
10 for example at a speed of about 10,000 rpm, within an ambient temperature range of 20 °C to 35 °C, to achieve uniform particle distribution.

A platinum catalyst is then integrated into the homogenized carbon solution at a controlled loading, preferably around 0.1
15 mg/cm², to ensure effective catalytic activity. The mixture is subjected to sonication to promote uniform dispersion of the platinum catalyst onto the carbon particles, forming a stable suspension.

A polymeric binder, such as polyvinylidene fluoride (PVDF),
20 is added to the suspension to enhance film-forming properties and mechanical stability. The resulting catalyst solution is then applied onto a substrate by casting or coating, thereby forming a thin film.

The coated film is subjected to drying, first under ambient
25 conditions for several hours, followed by vacuum drying at elevated temperature, such as 60 °C, to completely remove solvent residues. The drying step produces a self-supporting catalyst layer with uniform morphology, strong adhesion, and enhanced electrochemical performance.

30 The described process provides several advantages. The use of water hyacinth biochar offers a renewable and sustainable carbon support, while the integration of platinum through homogenization and sonication ensures uniform catalyst

dispersion. The addition of PVDF binder and controlled drying conditions yield a durable and thermally stable catalyst layer. This process is simple, scalable, and adaptable for PEMFC fabrication.

5 Further features of the present utility model are apparent from the examples.

Examples

10 1.1 Catalyst layer fabrication

Initially, 6.6 g of N-Methyl-2-pyrrolidone (NMP) solvent was accurately weighed in an empty vial. Subsequently, 0.89 g of biochar synthesized from water hyacinth was introduced into the solvent, forming a carbon solution. The mixture was homogenized
15 at 10,000 rpm under ambient conditions (typically, 20 °C to 35 °C) for 20 minutes to achieve uniform dispersion. Following homogenization, 25 mg of 40% platinum on Vulcan was incorporated into the solution to attain a target catalyst loading of 0.1 mg/cm² on 10x10 cm fabricated glass plates.

20 The resultant suspension was subjected to sonication in an ultrasonic bath operating in dynamic mode for 30 minutes to further enhance dispersion and interaction between components. This method of incorporating the catalyst in carbon is referred to as the wetting method.

25 In a separate beaker, a binder solution was prepared by weighing 0.75 g of polyvinylidene fluoride (PVDF) and dissolving it in 6.75 g of NMP through manual mixing until complete dissolution of the polymer, ensuring homogeneity.

The previously sonicated solution containing biochar, NMP,
30 and platinum on Vulcan was placed onto a hot plate stirrer. A 20 mm magnetic stir bar was introduced, and stirring commenced to maintain uniformity. The screw cap was removed, and the prepared binder solution was added dropwise at an approximate interval of 5 seconds per drop to facilitate gradual incorporation. Once the

complete binder solution was added, the vial was securely closed, and the mixture was left to stir continuously for 16 hours to ensure thorough blending.

5 The fully developed catalyst solution was subsequently sprayed onto a fabricated 10x10 cm glass plate using a HPLV airbrush with 1.5 mm nozzle. The coated sample underwent vacuum drying for 8 hours at ambient temperature (typically, 20 °C to 35 °C), followed by an additional 8 hours under vacuum at 60°C to eliminate residual solvents and enhance film integrity by
10 curing.

Upon completion of the drying process, the catalyst layer was carefully peeled from the glass plate, yielding a stable catalytic film for subsequent applications.

15 1.2 Integration of catalyst layer in membrane

A fabricated MEA former was opened by unscrewing the eight bolts, and protective layers were placed. The catalyst layer was then positioned followed by the membrane, which was also made in the laboratory, and another catalyst layer on top followed by
20 the top part of the MEA former. The eight bolts and nuts were tightened using a 75 N.m torque wrench to ensure proper compression. The fully assembled MEA former was then placed in a convection oven until 140°C was reached and maintained for 15 minutes to facilitate bonding. After the heating process, the
25 hot press was carefully disassembled, resulting in the successful formation of the membrane electrode assembly (MEA).

1.3 Characterization of Catalyst Layer

The morphology and microstructure of the catalyst layer
30 were analyzed using scanning electron microscopy (SEM) to evaluate its structural characteristics. The durability of the catalyst layer was assessed through oxidation testing, while Energy Dispersive X-ray (EDX) spectroscopy was used to determine

whether platinum dissolution occurred during a 24-hour oxidation soak. A subsequent boiling test was conducted to evaluate the catalyst layer's stability under accelerated conditions, ensuring its ability to withstand operation at 100°C for at least one hour. Finally, the performance of the integrated catalyst layer was assessed in a three-cell PEMFC stack by comparing its voltage output to that of a commercial three-cell PEMFC stack.

1.3.1 Morphological Characterization of Fabricated Catalyst Layers

The catalyst layers produced using two different approaches for catalyst incorporation is shown in Figure 1. Method 1 used the spray coating technique as shown in Figure 1A. In this method, catalyst ink with a binder was sprayed onto the carbon film, resulting in warping. In contrast, Method 2, which is the focus of this utility model, involved integrating platinum into the carbon support, producing a smooth and uniform film shown in Figure 1B.

The SEM images of the catalyst layers produced using the two methods, captured at 100,000x magnification with a scale bar of 100 pm is shown in Figure 2. The images illustrate that Method 2 results in a more uniform catalyst layer compared to Method 1.

1.3.2 Stability test of the Catalyst Layer

The catalyst layer was subjected to an accelerated durability test by immersing the sample in 3% hydrogen peroxide for 24 hours to simulate the oxidative environment of a fuel cell. The material remained intact after exposure to this oxidative environment, shown in Figure 3.

The EDX spectra of the sample after soaking (Figure 4) confirm the retention of platinum, indicating its stability and adherence to the substrate.

Additionally, the structural integrity was evaluated through a boiling test (Figure 5), in which the material was immersed in distilled water at 100°C for one hour. The results demonstrated its ability to withstand high operational
5 temperatures.

1.3.3 MEA using the fabricated Catalyst Layers

To assess the performance of the catalyst layers, they were integrated into a membrane electrode assembly (MEA) and tested
10 in a three-cell PEMFC stack. The testing was conducted using a HORIBA FuelCon Evaluator C Test Bench 71044, with parameters including a hydrogen flow rate of 0.3 NI/min and an air flow rate of 0.8 NI/min at room temperature. The performance of the fabricated MEA was compared to a commercial MEA purchased from
15 Fuel Cell Store (USA), along with the fuel cell stack. The fabricated MEA achieved an output voltage of 1.285 V, whereas the commercial MEA reached 2.836 V.

Although the fabricated MEA exhibited a lower output than its commercial counterpart, the results indicate promising
20 potential for an initial prototype, validating that the fabricated catalyst layer is operational. This Voltage output is also affected by the fabricated membrane which needs to be studied further. The interfacial contact and adhesion of the electrode catalyst with the membrane plays a vital effect on the
25 performance of the stack assembly.