BIOCHAR-BASED CATALYST SUPPORT LAYER

Technical Field

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

Background of the Utility Model

Advanced energy materials from biochar are being actively pursued by researchers for the development of new components for energy conversion and storage. This approach makes use of the unique structures and functionalities of biomass as well as its effects on transport and electrochemical processes during applications. Water hyacinth was utilized in the laboratory of Fuel Cell R&D and Testing Center of ITDI to develop carbon film as electrode support.

Water hyacinth (*Eichhornia crassipes*) is a free-floating aquatic plant native to the Amazon basin. It possesses long stems, purple flowers, and broad leaves that form dense mats on the water surface, obstructing sunlight penetration. This plant grows rapidly which led to its classification as an invasive species in many regions, including the Philippines, where it infests waterways like the Pasig River and Laguna de Bay. The dense mats formed by water hyacinths disrupt aquatic ecosystems by limiting sunlight, oxygen levels, and water flow, significantly impacting the livelihoods of local fisherfolk. As a readily available biomass waste and its abundance, water hyacinth was used for carbonization to produce carbon material suitable for further processing.

The present utility model utilizes the water hyacinth collected from the Laguna de Bay to produce biochar for carbon ink formulation of carbon electrode support for catalyst loading. There were two temperatures, 670°C and 800°C, used

during carbonization under N_2 atmosphere in the tube furnace to investigate the effects on the quality of biochar produced. In addition, subsequent processing of carbon material for carbon ink formulation was studied with varying ratio of carbon to binder such as 60:40, 50:50, and 40:60 to optimize processing conditions that will produce the desired properties. Initial trials were conducted to utilize the carbon film for Platinum catalyst layer deposition to investigate the suitability for the integration process into electrode catalyst.

The objective of this utility model is to provide a carbon film derived from water hyacinth-based biochar for use as a support layer in fuel cell electrodes. The carbon-to-binder ratio is optimized to achieve a uniform, stable carbon film. The resulting structure is characterized to confirm its suitability for proton exchange membrane fuel cell (PEMFC) applications.

Allam et al. (2020) characterized and evaluated water hyacinth biochar (WHB), produced via pyrolysis at 900 °C, for its catalytic activity in the oxygen reduction reaction (ORR). The study showed that WHB exhibited ORR activity based on its surface properties and achieved a higher power density than Pt/C in a microbial fuel cell. These findings demonstrate the potential of WHB as a low-cost ORR catalyst in microbial fuel cell applications.

CN111054423A discloses the development of a nitrogen doped porous carbon catalyst using biomass aquatic algae, followed by pyrolysis in an inert atmosphere. The resulting carbon film contains carbon, nitrogen, and transition metals such as iron, magnesium, or copper. It exhibits mechanical stability capable of supporting catalyst in fuel cells.

CN101780952A discloses a method for preparing porous carbon loaded with functional oxides from biomass. The process involves carbonizing the biomass at 600-800°C under inert atmosphere, altering the microstructure of the biochar. After drying and

pyrolysis at 600-800°C, a porous carbon material is obtained. The resulting material retains the biomass-derived pore structure and offers functional properties for use in water treatment, hydrogen storage, photocatalysis, fuel cells, and related applications.

Summary of the Utility Model

The utility model relates to a sustainable and cost-effective catalyst support layer composition for use in proton exchange membrane fuel cells (PEMFCs). The catalyst support layer comprises a carbon film derived from biochar made by carbonizing water hyacinth at 800°C under nitrogen atmosphere. The biochar, sieved to approximately 250 micrometers, is blended with a PVDF binder in a 50:50 weight ratio to form a carbon ink solution. The resulting composition exhibits favorable thickness, hydrophobicity, and disordered carbon structure suitable for catalyst support, enabling the utilization of water hyacinth biomass in clean energy applications.

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 FTIR analysis of Water hyacinth under different conditions (Dried Water hyacinth, 670 Water hyacinth Biochar, 800 Water hyacinth Biochar).

FIG. 2 shows XRD analysis of water hyacinth under different conditions a.) 670 Water hyacinth Biochar b.) 800 Water hyacinth Biochar.

FIG. 3 shows FTIR analysis of Carbon film with variation of carbon ink — binder solution at a.) 60/40 b.) 50/50 c.) 40/60

Detailed Description of the Utility Model

The present utility model concerns the composition of a catalyst support layer applicable to proton exchange membrane fuel cells (PEMFCs), incorporating a carbon film derived from invasive biomass waste, specifically water hyacinth (*Eichhornia crassipes*).

Water hyacinth stems are washed, sun-dried, and subjected to thermal carbonization at 800°C in a nitrogen atmosphere using a tube furnace. The resulting biochar is sieved to a particle size of approximately 250 micrometers to ensure uniformity.

The biochar is mixed with polyvinylidene fluoride (PVDF) binder in a 50:50 weight ratio using N-methyl-2-pyrrolidone (NMP) as a solvent to form a carbon ink. The ink is homogenized and coated onto a substrate to form a carbon film, which is subsequently dried under vacuum and mild heating. The carbon film displays a mean thickness of approximately 0.19 millimeters and a surface area of at least 40 cm^2. It also demonstrates hydrophobic surface properties, with a water contact angle of about 120°, favorable for water management in fuel cells.

FTIR analysis confirms the successful transformation of lignocellulosic biomass into carbon-rich material, with characteristic transmittance peaks related to C–O, C–H, and O–H groups. X-ray diffraction analysis reveals broad peaks at 28.3°, 40.5°, and 50.16°, confirming the amorphous, disordered carbon structure of the biochar.

This composition presents a low-cost, renewable alternative to commercial catalyst supports, utilizing an abundant and invasive biomass resource while achieving the structural and chemical properties necessary for efficient PEMFC operation.

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

Examples

Example 1: Biochar production from water hyacinth

Initially, the stems were thoroughly washed with water and sprayed with ethanol to remove surface contaminants and inhibit microbial activity. The cleaned stems were sun-dried until completely dehydrated. The dried biomass was then placed in a ceramic boat and preheated at 100°C for at least one hour to ensure the complete removal of remaining moisture. Following this pretreatment, the material was subjected to pyrolysis in a tube furnace. The furnace was programmed according to the temperature profile outlined in Table 1, ramping up to 670°C and 800°C, and maintaining this temperature for one hour under an inert nitrogen (N_2) atmosphere. After the pyrolysis process and subsequent cooling, the charred material was collected and mechanically sieved to achieve a uniform particle size of 250 µm, ensuring consistency and suitability for further applications.

Table 1. Temperature profile for tube furnace operation

Sample #	Present value (°C)	Set value (°C)	Ramp time (hr)	Maintained time (hr)
Sample 1: Water hyacinth biochar 670°C	Room temperature	500 °C	01:35	0:15
	500 °C	670 °C	00:34	01:00
Sample 2: Water hyacinth biochar 800°C	Room temperature	500 °C	01:35	0:15
	500 °C	800 °C	01:00	01:00

Example 2: Carbon film from biochar as support for catalyst layer

A carbon solution was prepared by dissolving the biochar product in N-methyl-2-pyrrolidone (NMP) (12 wt.% solution) and homogenizing it at a speed of 10,000 rpm for 20 minutes to ensure uniform dispersion. Separately, a binder solution was prepared by manually dissolving polyvinylidene fluoride (PVDF) powder in NMP (10 wt.% solution). This binder solution was then added dropwise to the carbon solution while continuously stirring. The resulting mixture was stirred for 2 hours to achieve homogeneity. Subsequently, the homogeneous mixture was sprayed evenly onto a fabricated glass plate with dimensions of 10 x 10 cm. The coated plate was subjected to full vacuum treatment for 8 hours, followed by another 8 hours of vacuum heating at 70°C with negative lbar set pressure. This process yielded a biochar film.

Table 2. Final formulation of polymer blend solution

Factor	Factor Range	Final Formulation
Water hyacinth biochar carbonization temperature, °C	670°C and 800°C	800°C
Carbon-binder ratio	60/40, 50/50, 40/60	50/50

Example 3: Characterization of biochar film

The biochar was characterized for confirmation of chemical composition of carbon material using Fourier Transform Infrared (FTIR) spectroscopy with Attenuated Total Reflectance (ATR) accessory and X-ray Diffraction (XRD) analysis to compare with carbon used for electrode material in literatures. The morphology and microstructure were analyzed using scanning electron microscope (SEM) with Energy Dispersive X-ray (EDX) spectroscopy to confirm uniform microstructure. The surface wettability was determined for carbon film using water contact angle (WCA) to determine the hydrophobicity.

Biochar

A typical biomass is expected to have lignin, cellulose, hemicellulose, starch, some organic components and biomass ash. The Fourier Transform Infrared (FTIR) analysis of the pyrolyzed water hyacinth revealed spectral characteristics of successful carbonization of cellulose and lignin components at 800°C which was confirmed by the presence of distinct transmittance peaks. The C-O stretching vibrations associated with cellulose and hemicellulose were observed in the 1200-950 cm^{-1} region. Peaks in the 1380-1240 cm^{-1} and 1460 cm^{-1} regions correspond to lignin structures, while the bands at 2925 cm^{-1} and 2850 cm^{-1} were attributed to C—H stretching in polysaccharides forming the cellulose and hemicellulose framework. Additionally, a broad absorption band in the 3680-3000 cm^{-1} range was indicative of O—H stretching, representing hydroxyl and carbonyl functional groups in lignin. These spectral features confirm the transformation of the biomass into a carbon-rich material with structural similarities to commercial activated carbon, as shown in Figure 1, where the functional groups of dried water hyacinth, biochar produced at 670°C, and biochar produced at 800°C were compared and analyzed.

Figure 2 shows the XRD analysis of biochar treated at 670°C and 800°C reveals structural changes influenced by temperature. The biochar at 670°C exhibits a peak at 10.68°, which is close to the 10.8° peak reported in the literature for graphite subjected to modification processes, indicating reduced layer organization and increased interlayer spacing. However, this peak is absent in the 800°C biochar, suggesting further structural rearrangement or degradation of the amorphous regions. Both biochar exhibit peaks at approximately 28.3°, 40.5°, 50.16°, and higher-index reflections, which are characteristic of disordered carbon structures. The crystallinity index (CI) of 52.23% for 670°C and 52.10% for 800°C

suggests that increasing the temperature does not significantly enhance crystallinity, aligning with findings that biochar retains a largely amorphous structure with limited graphitic ordering. These results highlight that while biochar undergoes some structural evolution with temperature, it does not develop the highly ordered graphite-like features on extensively processed carbon materials.

Carbon film

The FTIR spectra of carbon film with varying carbon-to-binder ratios (60/40, 50/50, and 40/60) indicate no significant changes in the functional groups present across the different compositions as shown in Figure 3. The characteristic peak at 1369 cm^{-1} is attributed to C-F stretching vibrations from PVDF, which is consistent with the typical absorption range of 1400-1000 cm^{-1} for strong C-F bonds. Additionally, the peak at 1020 cm^{-1} corresponds to C-O stretching or Si-O-Si vibrations, suggesting the possible presence of silica-based impurities from biochar. A broad absorption band observed between 3000-3600 cm^{-1} corresponds to 0-H stretching, which can be attributed to residual hydroxyl (-OH) groups within the biochar matrix. The absence of new peaks or peak shifts confirms that the fundamental chemical structure of the composite remains unchanged, indicating that biochar incorporation does not alter the primary functional groups of the PVDF binder.

SEM images of the carbon film with a ratio of 50/50 for carbon to binder solution. Arrays of carbon nanostructures were observed in the SEM image. An amorphous carbon film with no structure was formed on the substrate, the film thickness was 0.194 micrometers.

Table 3. Water contact angle data for various carbon film formulations using an optical tensiometer

Formulation	Trial 1	Trial 2	Trial 3	Trial 4	Mean	Standard deviation
60/40	113°	122°	131°	122°	122°	±7.35
50/50	115°	119°	121°	123°	120°	±2.97
40/60	100°	112°	113°	109°	109°	±5.92

Table 4. Thickness data for various carbon film formulations measured using a thickness gauge meter

Formulation	Trial 1	Trial 2	Trial 3	Trial 4	Trial 5	Mean ± s.d. (mm)
60/40	0.27 mm	0.26 mm	0.29 mm	0.21 mm	0.22 mm	0.25±0.034
50/50	0.19 mm	0.19 mm	0.17 mm	0.21 mm	0.21 mm	0.19±0.017
40/60	0.10 mm	0.11 mm	0.12 mm	0.11 mm	0.11 mm	0.11±0.007

The data in Table 3 indicate an increase in water contact angle, while Table 4 shows an increase in thickness as the carbon content increases.