Energy Storage Performance of Hydrogen Fuel Cells Operating in a Marine Salt Spray Environment using Experimental Evaluation

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Abstract

This work experimentally explores the influence of the sodium chloride pollution on the PEMFC performance in the marine salt spray environment by analyzing the concentration diffusion characteristics of the sodium chloride in the PEMFC membrane electrodes. Firstly, a set of experiments were carried out to determine the distribution of the sodium chloride components in the membrane electrodes, where five different salt spray environments (i.e., 100 mg/L, 200 mg/L, 300 mg/L, 400 mg/L, and 500 mg/L of the salt component, respectively) were used/employed to analyze the concentration diffusion characteristics of the sodium chloride. Then, the obtained samples were microscopically characterized and elementally analyzed by the field emission scanning electron microscopy (FESEM) and the energy spectrometry. Subsequently, a least squares-based model was proposed to predict the diffusion rate of the contaminating ions in the membrane electrodes. Lastly, the pollution of the sodium chloride was evaluated/assessed to reveal the performance degradation of the PEMFCs. The experimental results demonstrated that (1) the sodium chloride fraction existed as crystals or ions in the membrane electrodes in the marine salt spray environment; (2) the sodium chloride poisoning was founded in the proton exchange membrane in the form of sodium ions; (3) and the sodium-to-chloride ratio was proportional to the contamination time and the salt spray in the proton exchange membrane.

Experimental Evaluation of Energy Storage Performance of Hydrogen Fuel Cells Operating in a Marine Salt Spray Environment

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Abstract

In marine salt spray environments, sodium chloride contamination significantly deteriorates the performance of hydrogen fuel cells, particularly proton exchange membrane fuel cells (PEMFCs). While current evaluations of PEMFC degradation mechanisms caused by sodium chloride poisoning often focus on fluoride ion pollution, contamination of the membrane electrodes by sodium chloride remains largely unexplored. This work experimentally investigates the influence of sodium chloride pollution on PEMFC performance in marine salt spray environments by analyzing the concentration diffusion characteristics of sodium chloride in PEMFC membrane electrodes. First, a series of experiments were conducted to determine the distribution of sodium chloride components in the membrane electrodes, employing five different salt spray environments (100 mg/L, 200 mg/L, 300 mg/L, 400 mg/L, and 500 mg/L of salt component) to analyze the concentration diffusion characteristics. The obtained samples were then characterized microscopically and analyzed elementally using field emission scanning electron microscopy (FESEM) and energy dispersive spectrometry. Subsequently, a least squares-based model was proposed to predict the diffusion rate of contaminating ions in the membrane electrodes. Finally, the sodium chloride pollution was evaluated to reveal PEMFC performance degradation. The experimental results demonstrated that: (1) sodium chloride existed as both crystals and ions in the membrane electrodes within the marine salt spray environment; (2) sodium chloride poisoning was found in the proton exchange membrane in the form of sodium ions; and (3) the sodium-to-chloride ratio was proportional to both contamination time and salt spray concentration

in the proton exchange membrane.

Keywords: Hydrogen fuel cells; proton exchange membrane fuel cell; membrane electrode; salt spray

1. Introduction

The proton exchange membrane fuel cell (PEMFC) represents a typical type of hydrogen fuel cell that has emerged as a promising alternative to fossil fuels in marine power systems due to its high practicability and low emissions. However, in marine salt-spray environments, sodium and chloride concentrations are substantially higher than those in inland areas, resulting in frequent sodium chloride poisoning incidents that cause irreversible performance degradation in PEMFCs [1, 2]. The term "sodium chloride poisoning" was introduced by Mikkola [3], who discovered that sodium chloride can damage the catalyst in PEMFCs and lead to battery performance degradation. Unnikrishnan et al. [4] reported that chlorine contamination can cause performance losses of 94% and 82% on the battery anode and cathode, respectively. Therefore, investigating the mechanisms of sodium chloride poisoning in marine salt spray environments is critical for preventing battery degradation.

The durability of the proton exchange membrane serves as one of the most important indicators of cell performance, and proton exchange membrane operation is subject to contamination from various materials in the cell stack, coolant, and fuel-side contaminants [5]. Impurities from the air side enter the cathode side of the PEMFC, where they dissolve in liquid water and penetrate the cathode side under a concentration gradient, causing degradation or failure during operation [6]. Regarding sodium chlorine poisoning, when chlorine enters the PEMFC, chlorine ions are adsorbed on the Pt catalyst surface, potentially damaging the catalyst. Literature review indicates that impurities degrade cell performance by affecting the H+ content and the active specific surface area of the catalytic layer [7], with the degree of contamination typically evaluated in terms of fluoride ion release rate, atomic percentage, and impurity migration rate. Recent progress suggests that membrane cation contamination represents an important cause of battery degradation. For example, Wang et al. [7] investigated membrane electrode component contamination through immersion experiments in perionic solutions (Ca2+, Mg2+, Na+), examining the effects of contaminated solution environments on catalytic and gas diffusion layers, and found that H+ in the proton exchange membrane was more contaminated by metal cations than the catalytic layer. Jie et al. [8] found that the presence of Na+ had a significant toxic effect on Pt/C. In 2009, Strmcnik et al. [9] proposed a cluster of non-covalent interactions between Pt and surface oxides on cations (containing Na+), leading to occupation of Pt active sites in electrochemical reactions. Jayasayee et al. [10] studied the relationship between Cl- concentration and dissolution rate of PtNi alloy using energy dispersive X-ray spectroscopy (EDX) and inductively coupled plasma spectroscopy, determining that Cl- enhanced the dissolution of Pt and Ni by monitoring changes in the percentage of Pt and

Ni atoms. Uddin et al. [11] analyzed the surface and cross-section of the gas diffusion layer of the cell membrane electrode and the cross-sectional morphology of the catalytic coating membrane (CCM) using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS), with results showing that cations were not detected in the CCM, though the study indicated that chloride ions reduced the catalyst activity specific surface area.

Arruda et al. [12] studied anion adsorption competition on the Pt catalyst surface using spectra formed by XRD and EDX in a half-cell system. Mo et al. [13] analyzed the migration of corrosion product metal cations in PEMEC catalyst coating membranes with EDS images, showing that high levels of Fe cations in Nafion membranes occupied water nanochannels, reducing proton transport efficiency in PEM. Gutleben et al. [14] demonstrated that Pt degradation was caused by the electrochemical reaction of chloride in the vicinity of the membrane. Chemical degradation of the membrane primarily results from the vulnerability of SO3H, CF3, OCF2, and CF groups on the side chains of perfluoro sulfonic acid polymer to attack by (-OH) radicals and shedding, leading to destruction of the function and integrity of the proton exchange membrane [15, 16]. Hori et al. [17] studied the dissociation of F- from fluorine elements in membranes after immersion in contaminated solutions containing iron, along with the appearance of intermediate products in the membrane side chains. Zhang et al. [18] showed that the rate of fluorine ion release from membranes was linearly and positively correlated with contamination time. Kelly et al. [19] evaluated the degree of membrane contamination by determining calculated ion uptake in membranes based on cationic impurity concentration and by energy dispersive X-ray analysis through atomic percentage ratio. Inaba et al. [20] studied the rate of reaction between air and hydrogen at the fuel side to generate free radicals, comparing the fast rate of fluorine ion generation on the anode side and the cathode side. Liu et al. [21] found through in situ detection methods that free radicals produced at the anode diffused to the cathode side through concentration differences, leading to attenuation of the proton film. Kienitz et al. [22] investigated the degradation of proton exchange membrane fuel cell performance contaminated with foreign cations (Na+, Ca2+, or metal ions) in a half-cell model through numerical simulations, evaluating contaminant and proton transport through diffusion and migration by comparing fluoride ion release rates from the anode and cathode sides. Xie et al. [23] evaluated the degree of contamination by observing membrane crack expansion through electron micrographs and performed molecular simulations of weight loss and product emission rates over time to model chemical contamination degradation and mechanical deformation of the membrane. Md Aman Uddin et al. [24] investigated the effect of the gas diffusion layer (GDL) on cationic contamination of polymer electrolyte fuel cells (PEFCs) by soaking three membrane electrode assembly (MEA) structures in cationic (Ca2+) solution, with results showing that the hydrophobicity of GDL and microporous layer (MPL) served as a barrier preventing the cationic solution from reaching the CCM.

In summary, although existing literature has indicated that contamination of membrane electrode components causes PEMFC degradation, very limited work has addressed PEMFC degradation mechanisms in marine salt-spray environments. For PEMFC-powered ships, investigating the influence of sodium chloride on membrane electrodes and exploring PEMFC degradation mechanisms is inevitable. However, little work has been conducted on this topic in existing literature. To this end, this study examines the effects of sodium chloride diffusion rate and contamination in membrane electrodes to explore battery degradation in marine salt-spray environments. The evaluation of contamination degree provides a theoretical basis for addressing the adverse effects of impurity ions on the battery and offers implications for developing battery contamination mitigation strategies in marine salt-spray environments.

The remainder of this paper is organized as follows. Section 2 describes the experimental testing of sodium chloride pollution on PEMFC membrane electrodes in marine salt-spray environments. Section 3 presents the experimental results. Section 4 evaluates the contamination level of Na+ in the membranes. Section 5 summarizes conclusions and future work.

2.1 Experiment Preparation

Based on the operation of salt spray aerosol and the principle of salt spray generation, the marine environment was simulated using a standard salt spray test chamber according to marine atmospheric environment parameters. The salt spray particle size ranged between 0.1 m and 0.5 m, salt spray concentration between 100 mg/m^3 and 500 mg/m^3, temperature between 25°C and 45°C, humidity between 85%RH and 95%RH, and settling volume between 1 ml/80cm^2/h and 2 ml/80cm^2/h. Accelerated tests and natural atmospheric exposure tests were carried out by loading salt spray contents with different ratios, and referring to [25], the salt mist content ranged between 0.2 mg/m^3 and 0.66 mg/m^3. Fig. 1 [Figure 1: see original paper] illustrates the experimental test procedure, where the gas phase test was conducted to evaluate the sodium and chloride contamination effects on the battery membrane electrodes. The experimental procedure is described as follows [26-27].

First, the experimental membrane electrodes were prepared. A 5 cm × 5 cm membrane electrode was divided into five 1 cm × 5 cm membrane electrode samples. The mass of each sample was weighed with an analytical balance. Each sample was then placed in a vacuum drying oven at 80°C for 8 hours and baked until its mass became constant. Second, the salt spray test solution was prepared. Pure sodium chloride was dissolved into tertiary water in accordance with GB1266 (which specifies the configuration of chemical reagent neutral salt spray solution) and GB6682 (which specifies water specifications and experimental methods for analysis laboratories). The pH value of the obtained solution was then tested at 25°C and ensured to be between 6.5 and 7.5 to generate the neutral solution.

After that, the neutral solution was deionized with impedance of 18 Ω and mixed with NaCl powder (99% purity) to prepare five different concentrations of salt spray test solutions (100 mg/L, 200 mg/L, 300 mg/L, 400 mg/L, and 500 mg/L of NaCl). Third, the gas phase test was performed. The 100 mg/L NaCl solution was atomized and tested for 15 days. The test was repeated using different concentrations to generate 30 membrane electrode samples total for characteristic analyses. Finally, liquid nitrogen embrittlement was used to cut the electrode samples into 1 cm × 1 cm flat sections for SEM and EDS equipment.

A Quorum sputter coater was used to pre-treat the samples to ensure accurate elemental analysis. A field emission electron microscope and an energy dispersive X-ray spectrometer Smart EDX were used to analyze the CCM of the catalyst coated membranes and the surface microscopy of the gas diffusion layer (GDL). Fig. 1 provides an overview of the experimental test. In Fig. 1, the membrane electrodes (platinum loading: cathode 0.3 mg/cm^2 and anode 0.1 mg/cm^2) were produced by Wuhan Polytechnic New Energy Co., Ltd.; the sodium chloride (99% purity) was produced by Tianjin Zhiyuan Chemical Reagent Co., Ltd.; the vacuum drying oven (model DZF-6020) and electronic analytical balance (model FA2204) were provided by Shanghai Lichenbangxi Instrument Technology Co., Ltd.; the KD-60 standard salt spray chamber was provided by Zhejiang Blue Arrow Instrument Co., Ltd.; the German Zeiss field emission electron microscope (model SUPRA 55) and energy dispersive X-ray spectrometer Smart EDX were used; and the Quorum SC7620 sputter coater was supplied by Nanjing Qinshi Technology Co., Ltd.

2.2 Microscopic Morphological Characterization

The presence of sodium chloride and its distribution in the membrane electrode were analyzed through a combination of micromorphology and elemental analyses. To examine the effect of experimental time on GDL surface morphology and the microscopic effects of sodium chloride diffusion, membrane electrodes were tested at different periods. In the experimental tests, crystal formation was made independent of solution saturation precipitation by ensuring the solution concentration was much lower than the saturation points of crystallization and precipitation. Additionally, since experimental impurities were derived solely from the independent variable sodium chloride and the deionized water used for solution preparation excluded other impurity components, any crystal particles appearing in the experiments originated only from sodium chloride in the salt spray environment.

The tested results of membrane electrodes with impurities were compared with those without impurities. Fig. 2 [Figure 2: see original paper] shows the SEM surface morphology of the original GDL [28], while Fig. 3 [Figure 3: see original paper] depicts GDL morphologies after sodium chloride experimental tests. Fig. 3 presents GDL morphologies for three NaCl mass concentrations (300 mg/L, 400 mg/L, and 500 mg/L) and six testing periods (24 h, 72 h, 144 h, 216 h, 288 h, 360 h).

Comparing Fig. 2 and Fig. 3 reveals that sodium chloride crystal particles appeared in all tested GDL morphologies. As testing time increased, the number of NaCl crystal particles increased significantly and particle distribution changed from sparse to dense. The most striking feature was that sodium chloride crystals were uniformly distributed at carbon fibers and bonds, with crystal density increasing over time. With different NaCl mass concentrations, sodium chloride crystal particles were randomly distributed on the carbon fibers.

The surface morphologies of the GDLs observed through electron microscopy demonstrate that sodium chloride crystal particles were uniformly distributed in the salt spray environment. The overall shape of the crystal particles was regular, showing a typical cubic crystal structure. The distribution of crystal particles became increasingly extensive and dense with increasing experimental time at a given NaCl mass concentration, while the number of crystals varied more significantly with different NaCl mass concentrations at a given testing time.

To further investigate the concentration dispersion characteristics of sodium chloride at PEMFC membrane electrodes under salt spray conditions, one testing sample contaminated with sodium chloride was tested for 360 h. The base sample was a cross-sectional microstructure of the uncontaminated membrane electrode. The cross-sectional microstructures of the CCM layer and the membrane electrode were observed under 15 kV voltage. Fig. 4 [Figure 4: see original paper] shows the cross-sectional microstructure of the original CCM, and Fig. 5 [Figure 5: see original paper] depicts the cross-sectional microstructure of the tested CCM.

Unlike the surface morphologies of the GDL in Fig. 3, sodium chloride crystals were not observed in the cross-sectional morphologies shown in Fig. 5. No crystal attachments were present at magnifications of both 5 KX and 40 KX, and no crystal structure was found after 360 h of testing at concentrations of 300 mg/L, 400 mg/L, and 500 mg/L in the salt spray environment. It is inferred that sodium chloride may appear in the catalytic layer and the membrane in ionic form. Comparing the CCM surface morphologies before and after tests in Fig. 4 and Fig. 5 reveals that 360 h of testing did not cause any visible morphological changes in the microscopic structure.

Fig. 6 [Figure 6: see original paper] shows the elements and their distributions in the cross sections of the tested membrane electrodes. In Fig. 6(a), because the membrane electrode received no treatment, no sodium or chloride elements were found in the elemental distribution. However, the elemental distribution of membrane electrodes tested at 500 mg/L NaCl mass concentration in a salt spray environment differed significantly from Fig. 6(a). The elemental distributions of sodium and chloride in Fig. 6(b)-(d) suggest the presence of sodium in the membrane and chlorine in the catalytic layer. Sodium and chloride elements were distributed homogeneously across the proton exchange membrane and catalytic layer, but they were not co-located, instead appearing in a dispersed manner.

Fig. 6 also suggests that while sodium chloride crystal structure attached to the GDL, no crystal structure was found on the catalytic layer or the proton exchange membrane. Therefore, it is clear that sodium chloride elements appeared in two forms: sodium chloride crystal form on the gas diffusion layer and ionic form on the catalytic and proton exchange membranes.

Fig. 7 [Figure 7: see original paper] depicts the cross-sectional morphology of the CCM after 360 h testing in a salt spray environment with a NaCl mass concentration of 500 mg/L, and Fig. 8 [Figure 8: see original paper] shows the element distribution curves of the cross section. In Fig. 8(a), element F fluctuates at a distance of 5 to 20 m and forms three peaks; element Pt forms two peaks with values of approximately 100 cps at 2.6 m and 300 cps at 27.5 m; element Cl appears with three peaks between 5 m and 22.5 m with a maximum peak value of about 80 cps; and the overall distribution curve of element Na is similar to that of element Cl but with much smaller peaks.

3.1 Elements Contents

In the element analysis of the membrane microstructure, spot analysis was performed to detect the relative content of each element. A sample under the 300 h-500 mg/L condition was used to interpret the element spot position and element content spectrum, as shown in Fig. 9 [Figure 9: see original paper] and Table 2 . Elements C, O, Na, Pt, S, and Cl were detected. The C and O contents accounted for the largest percentages in the GDL, PEM, and catalytic layer, followed by F content, with Na content larger than that of Cl and S.

Table 3 shows element contents in the membrane with respect to varying time, and Table 4 displays the concentration effect. The percentage content of sodium in the membrane increased with both test time and concentration. At 24 h-500 mg/L testing, the sodium percentage content was 0.64% in the membrane, increasing to 2.92% at 360 h-500 mg/L testing in Table 3. At 100 mg/L-360 h testing, the sodium percentage content was 1.61% in the membrane, increasing to 2.92% at 500 mg/L-360 h testing in Table 4. However, chlorine content did not show any consistent pattern in the membrane with respect to testing time or solution concentration.

3.2 Evaluation of Membrane Contamination Degree

Membrane affinity for different contaminant concentrations is critical, making it important to evaluate the contaminant degree in membranes. This study adopts a two-step approach to qualify and quantify the contaminant degree. First, qualitative analysis was performed to qualify the contaminant degree based on experimental results from microscopic characterization. Tables 3 and 4 show that sodium content in the proton exchange membranes increases with increasing time and solution concentration, while chloride ions are hardly absorbed into the membrane. Consequently, the degree of membrane contamination can be evaluated by sodium content. For this reason, the sodium-to-fluorine ratio is proposed as an evaluation metric to qualify membrane contamination. The sodium-to-fluorine ratio in the membranes measured by EDX is summarized in Table 5 . The EDX results show that as NaCl mass concentration increases, sodium ion absorption in the membranes increases and the sodium-to-fluorine ratio gradually rises. Similarly, the sodium-to-fluorine ratio in the membranes gradually increases with testing time. These observations suggest that increases in both testing time and NaCl mass concentration lead to increased impurity absorption by the membrane and increased sodium-to-fluorine ratio. Therefore, a larger sodium-to-fluorine ratio indicates heavier contamination in the membrane.

Second, quantitative analysis was performed to quantify the contaminant degree by developing a diffusion rate prediction model for sodium contamination in the membrane. The prediction model, which considers testing time and NaCl mass concentration factors, is expressed in Eq. (1) [29]:

$$W = W_j(C, t) \tag{1}$$

where W is the membrane contaminant degree and W_j is a prediction function related to the NaCl mass concentration C and the testing time t in the salt spray environment.

The prediction function can be established by regressing the sodium mass percentage from experimental tests [31-33]:

$$W_j(C, t) = K_c \cdot K_t \tag{2}$$

where K_c denotes the change rate of sodium mass percentage with respect to NaCl mass concentration and K_t denotes the change rate of sodium mass percentage with respect to time.

K_c can be estimated by fitting experimental data using the least squares method. Fig. 10 [Figure 10: see original paper] shows the fitting results describing the relationship between sodium mass percentage and solution contamination concentrations. Similarly, K_t can be estimated according to Table 6 using the least squares method, with fitting results shown in Fig. 11 [Figure 11: see original paper]:

$$k_t = 0.0024 + 3.9244 \times 10^{-6} t \tag{3}$$

Combining Eq. (2) and Eq. (3) yields:

$$W_j(C, t) = (0.0024 + 3.9244 \times 10^{-6} t) \cdot k_c \tag{4}$$

Assuming the total mass in the test area of the PEMFC membrane electrode remains constant, the relationship between sodium mass percentage and total element mass can be expressed as:

$$m_{Na} + m \tag{5}$$

where m_{Na} is the sodium mass and m is the total element mass in the test area. Combining Eq. (4) and Eq. (5) yields:

$$m_{Na} = \frac{(0.0024 + 3.9244 \times 10^{-6} t) \cdot k_c}{1 - (0.0024 + 3.9244 \times 10^{-6} t) \cdot k_c} \cdot m \tag{6}$$

If the concentration is determined, the sodium diffusion rate can be obtained according to the principle of partial derivatives:

$$V_{Na} = \frac{d[m_{Na}]}{dt} \tag{7}$$

Combining Eq. (6) and Eq. (7), the sodium diffusion rate can be rewritten as:

$$V_{Na} = \frac{3.9244 \times 10^{-6} k_c m}{[1 - (0.0024 + 3.9244 \times 10^{-6} t) k_c]^2} \tag{8}$$

Consequently, the diffusion rate prediction model for sodium in the membrane was obtained to quantify the membrane contamination degree.

4. Conclusions

This work experimentally explored the influence of sodium chloride pollution on PEMFC performance in marine salt spray environments by analyzing the concentration diffusion characteristics of sodium chloride in PEMFC membrane electrodes. The experimental findings demonstrate that:

1. Sodium chloride appears on the membrane electrode in two forms: as crystals on the gas diffusion layer structure and as ions on the catalytic layer and proton exchange membrane structure. The crystals are uniformly distributed on the carbon fibers of the gas diffusion layer in the salt spray environment, with crystal distribution becoming increasingly dense over time, though crystal particle size does not grow significantly.
2. The effect of sodium chloride differs across membrane electrode structures. The percentage content of sodium atoms detected on the proton exchange membrane structure reaches up to 2.92%, with atomic percentage content positively correlated with both experimental time and concentration. The sodium element distribution curve shows a similar trend to that of the

fluorine element, with overall sodium content detected higher than that of chlorine.

3. Based on experimental test data, a quantitative relationship was established to evaluate membrane electrode contamination degree. Using linear fitting with the least squares method, the relationship between sodium element diffusion rate in the membrane, contamination time, and contamination concentration under salt spray environment was proposed, constructing a prediction model for contaminated ion diffusion rate applicable to actual working conditions under cell operation time and salt spray salt content.

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Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this article.

Research Data

All data can be requested from the corresponding author.

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Note: Figure translations are in progress. See original paper for figures.