

Published by Avanti Publishers

The Global Environmental

Engineers

ISSN (online): 2410-3624



An Investigation of Ag/NaTi $_2(PO_4)_3$ Seawater Battery Toward Scalable Energy Storage

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ARTICLE INFO

Article Type: Research Article Academic Editor: Erzi Tang Keywords: Silver electrode Seawater battery NaTi₂(PO₄)₃ electrode Conversion-type electrode Intercalation-type electrode *Timeline*: Received: November 03, 2024 Accepted: December 05, 2024 Published: December 27, 2024 Citation: Weng G-M. An investigation of Ag/NaTi₂(PO₄)₃ seawater battery toward scalable energy storage. Glob Environ Eng. 2024; 11: 54-62.

DOI: https://doi.org/10.15377/2410-3624.2024.11.4

ABSTRACT

The seawater battery, consisted of silver (+ve) and carbon-coated NaTi₂(PO₄)₃ (-ve), is an ecofriendly energy storage system due to the low cost and natural abundance of seawater. However, more efforts are still needed to research on the potential issues associated with the ion transport, the breakdown of voltage losses and the attempts for scaling up of such a battery system. Herein, it is found that a nonnegligible shuttle effect of Ag+ ions could pose a serious impact on the reversibility of the battery system. In addition, through the four-electrode measurement, the carbon-coated NaTi₂(PO₄)₃ negative electrode with intercalation/ deintercalation chemistry is identified as the limiting component in the current battery device. Moreover, attempts on applying semi-solid electrolytes to such a battery system are also conducted. It is found that the capacity fading is serious probably due to the hydrogen evolution side reaction at the negative side. Future technical advancements in the key materials and reactor design will make this battery technology more competitive. This work offers important insights to develop safer and scalable seawater batteries.

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1. Introduction

The generation of electrical energy from renewables has surged in recent years due to rising global demands for efficient, environmentally friendly energy production [1-3]. As a result, the development of effective energystorage systems is imperative in order to improve the ability to store and utilize the energy generated from renewables on a large scale [4-6]. In comparison to lithium-ion batteries, sodium-ion batteries have lower raw material costs (~50 times cheaper than lithium), environmental compatibility, high ionic conductivities in electrolyte, comparable ion intercalation character, and similar redox potentials (Na/Na⁺ = -2.71 V vs. SHE and Li/Li⁺ = -3.04 vs. SHE) [7-9]. Even though the specific energy capacity of sodium-ion battery [10] is less than that of conventional lithium-ion battery [11], sodium-based materials are significantly more promising due to the low cost of sodium-based precursor materials and potential to collect sodium directly from seawater [12-14]. More importantly, utilizing such systems can further reduce the R&D pressure on recycling of spent lithium-ion batteries [15-18]. There is no doubt that it is quite difficult to achieve an effective recycling of lithium-ion batteries mainly due to the recycling is most about hazardous and dangerous materials [19-21].

Herein, this work builds upon the concept of sodium-ion batteries with the "blue" matters or resources in the sea, i.e., utilizing natural seawater as the electrolyte solution. By doing so, Na⁺ and Cl⁻ ions of seawater can be served as charge carriers in such a seawater battery architecture. This seawater battery is an eco-friendly energy storage system due to the low cost and natural abundance of seawater (Fig. **1**), as ~97% of Earth's water is in the form of seawater, and thus it conveniently provides a readily available source of Na⁺ and Cl⁻ ions for redox capabilities [22, 23]. It is believed that, from a long-term and positive perspective, both seawater battery concept and taking advantage of blue energy via triboelectric nanogenerators (TENG) [24-26] are effective ways for humans to realize the "blue dream" [27], and the relationship between them should be complementary. However, as summarized in Table **1**, the commercialization of some seawater are harvested; ii) toxic chlorine gas is generated during battery charging; iii) high-risk metallic sodium is used as the negative electrode; and iv) their scale-up is challenging. Moreover, additional research on the potential issues associated with the ion transport, the breakdown of voltage losses and the attempts for scaling up should be further conducted. Accordingly, efforts have been made in this paper to unveil the full picture of such a battery system and address the above-mentioned challenges.

2. Experimental Section

The synthesis procedure of the carbon-coated $NaTi_2(PO_4)_3$ (denoted as ccNTP) can be found in ref. [35]. Silver solid material was obtained by directly oven-drying the commercial silver conductive paste. Before use, the



Figure 1: An eco-friendly concept of using seawater batteries to store and supply energy.

Ref.	Anode	Operating Voltage	Reversible Capacity	lons Utilized from Seawater		
[28]	Red phosphorous/carbon amorphous material	3.3 V (average, full cell)2.5 V (average, half-cell)		Na⁺		
[29]	Hard carbon	~3.1-3.2 V (discharge)	290 mAh/g	Na⁺		
[12]	Na metal with graphene- covered Cu current collector	~2.5 V (discharge)	Fixed total capacity: 0.075 mAh/cm ²	Na ⁺		
[30]	Na metal & hard carbon	~2.9 V (discharge, Na metal) ~4.1 V (charge, Na metal) ~2.1 V (hard carbon)	~115 mAh/g (hard carbon)	Na⁺ & evolves Cl₂ gas		
[31]	Hard carbon	Half cell: ~3.45 V (charge), ~2.98 V (discharge) Full cell: fixed current density 0.05 mA/cm ² charging, discharge cut-off 0.5 V	~191-106 mAh/g	Na⁺		
[32]	Hard carbon	~2.8 V (charge) and ~2.5 V (discharge)	296 mAh/g	Na ⁺ & evolves Cl_2 and O_2 gas		
[33]	Hard carbon	3 V (average, full cell)	120-10 mAh/g	Na^+ & evolve Cl_2 gas		
[34]	Hard carbon (starch derived)	4.0-1.7 V at 0.2 C and 4.2-1.4 V at 0.5 C	~300 mAh/g over 400 cycles	Na ⁺		
[23]	NaTi ₂ (PO ₄) ₃	Typical values: 0.86 V (charge), 0.76 V (discharge)	~40 mAh/g over 200 cycles	Na⁺ and Cl⁻		

Table 1:	An	overview	of	typical	so-called	seawater	battery	y sy	ystems
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commercial graphite felt (used as the current collector) was soaked in 95 wt% H₂SO₄ at 50 °C for 4 hours, followed by rinsing with DI water until the effluent pH is near 7 and heating treatment at 70 °C for 2 hours. Then, electrochemical anodization was performed following ref. [36]. In the tests of cyclic voltammetry (CV) and fourelectrode measurement, the negative electrodes were prepared by pasting the mixture of ccNTP, Super P and polytetrafluoroethylene binder (weight ratio of about 70:9:21) onto one side of the graphite felt; while the positive electrodes were prepared by simply pasting silver conductive paste onto one side of the graphite felt. As an aside, Super P is a high purity and structured carbon black powder commonly used in lithium-ion battery industry [37]. Before test, both electrodes were sufficiently dried to remove the residual solvent. The electrolyte solutions were prepared by dissolving corresponding analytical reagent grade chemicals (around 96-98% pure) into DI water. Also, the electrolyte solutions (including the semi-solid electrolytes) were saturated with N₂ before use, especially the ones for the ccNTP side. Semi-solid electrolytes were prepared by mixing the active material of 0.2 g (i.e., Ag or ccNTP), 0.2 g Ketjenblack and 2 mL 1 M NaCl aqueous solution via magnetic stirring for 10 mins, then tip sonication for 10 mins, and finally N₂ bubbling for 10 mins. In each electrochemical system, a piece of anionexchange membrane (Fumasep FAA-3) was used as the separator. Before use, the membrane was pretreated by immersing into aqueous salt solution overnight.

Electrochemical tests were carried out with a Biologic VMP3 electrochemical station at room temperature and the EC-Lab software (Version 11.20) was used for the analysis. The CV tests were done with a typical threeelectrode system and the CV analysis was carried out at 1 mV/s. The four-electrode measurements were conducted with a H-type glass cell, in which two additional Ag/AgCl reference electrodes were used to monitor the individual potential changes of the key components. In the demo of the scaling-up, about 1 mL as-prepared semisolid electrolyte was injected into each chamber of the semi-solid flow battery prototype. To assess the basic electrode performance, solid electrodes were also assembled into Swagelok-type cells reported in our previous work [35]. Charge and discharge measurements were carried out between 1.1 and 0.3 V at different applied currents. All experimental details are provided as above, unless otherwise stated.

3. Results and Discussion

The redox properties and potential window of each electrode material were first studied with cyclic voltammetry. Fig. (2a) shows the cyclic voltammograms (CVs) of ccNTP (-ve) and Ag (+ve) electrodes at the scan rate of 1 mV/s. At the steady state, the CV of the former one shows well defined reduction and coupled oxidation peaks at about -1.0 and -0.5 V vs. Ag/AgCl, respectively. For the latter one, oxidation and reduction peaks were observed at about +0.2 and -0.2 V vs. Ag/AgCl, respectively. Based on the following electrochemistry (Eqns. 1-2) and the CV results, such a coupling can theoretically lead to a battery system with a reversible cell voltage of about 0.84 V. Also, it is clearly seen from Fig. (2a) that the kinetics of Ag electrode is faster than that of ccNTP. This is consistent with the common sense that the conversion-type electrode material has faster kinetics than that of intercalation-type one. Some quick galvanostatic battery tests with these two electrodes were also done with different separators, e.g., porous membrane (i.e., filter papers) and cation-exchange membrane. It is found that a nonnegligible shuttle effect of Ag⁺ ions could pose a serious impact on the reversibility of the ccNTP electrode. More specifically, a light vellow or white solid film was formed onto the surface of the ccNTP electrode. Further attempt by using saturated NaCl electrolyte solution to alleviate this issue by the predominant migration flux of Na⁺ was also failed, which implies that even a trace amount of Ag⁺ ions into the ccNTP side may make a big difference. Finally, an anion-exchange membrane was used to enable a stable cell performance (as evidenced from Fig. **2b**) because it could serve as a good blockade to prevent the Ag⁺-ion crossover.

$$(-ve) \operatorname{NaTi}_2(PO_4)_3 + x\operatorname{Na}^+ + xe^- \xrightarrow{charging} \operatorname{Na}_{1+x}\operatorname{Ti}_2(PO_4)_3$$
(1)

(+ve)
$$Ag^+ + CI^- - e^- \xrightarrow{charging} AgCl$$
 (2)



Figure 2: a) Cyclic voltammograms of ccNTP and Ag electrodes at the scan rate of 1 mV/s. **b**) Cycling performance of a Swagelock-type static seawater battery cell using solid electrodes at 0.2 mA. A piece of anion-exchange membrane was used as the separator and 1 M NaCl aqueous solution was used as the electrolyte.

The analysis of voltage losses within such a seawater battery and the performance of its individual components can be assessed by examining the four voltage profiles. The overall cell voltage (Fig. **3a**) exhibits stable charge and discharge plateaus at different electrolyte concentrations, measuring approximately 0.82-0.95 V and 0.75-0.79 V, respectively. This stability is similarly observed in Fig. (**3b**) and (**3c**), demonstrating consistent positive and negative electrode reactions. Interestingly, as shown in Fig. (**3a**), the highest-concentration system shows the lowest charge voltage plateau but not the highest discharge voltage. This could be due to higher salt concentrations slow down the intercalation reaction, as observed from Fig. (**3c**). The concentration changes did not show significant effect on the Ag electrode potential and the voltage across the membrane, and thus voltage losses remain very small at the tested conditions. This also indicates that steady and reversible ionic transport

across the FAA-3 membrane could be achieved (Fig. **3d**). It is worth noting that the limiting component of the current system is the ccNTP side. Further improvement can be done by using higher-performance NTP and membrane materials.



Figure 3: Four-electrode characterization of a sweater battery using a H-type glass cell. **a**) The overall cell voltage. **b**) The potential difference between the silver positive electrode and Ag/AgCl reference electrode. **c**) The potential difference between the ccNTP negative electrode and the Ag/AgCl reference electrode. **d**) The voltage across the FAA-3 membrane measured by two Ag/AgCl reference electrodes. The applied current was 0.2 mA while the cut-off voltage range was 1.1-0 V.

In order to scale up the conceptual battery system, semi-solid electrolytes were first prepared following the commonly used recipe which is reported elsewhere [38-41]. For this preliminary trial, a home-made flow battery device was used (Fig. 4a). According to the quick tests, the estimated viscosity of the as-prepared semi-solid electrolyte was approximately 10 times higher than that of aqueous NaCl electrolyte solution (i.e., ~10 centipoise). However, the current device prototype could not lead to a continuous and smooth flow of the semi-solid electrolyte. This is because the semi-solid electrolyte flow within such a device design still relies on an externally driven mode. Thus, at this stage, the battery testing was performed in static mode. Notes: a conceptual advance of the reactor design for the semi-solid battery chemistry will be reported soon by the author's research group. As seen from Fig. (4b), the charge and discharge plateaus were similar to those of the system with solid electrodes. But the capacity fading was quite serious, probably due to the side reactions like water splitting along with the H_2 evolution at the negative side. This was further verified after dissembling the battery and the issue of water loss was found. It is believed that the imperfect conductive network (composed of active materials and conductive agents) of the semi-solid electrolyte could induce various issues like more side reactions and higher internal resistance, etc. In other words, due to the insufficient electronic conductivity of the electrode material, it is necessary to incorporate a significant quantity of conducting additives, such as carbon black, into the electrode sheet to establish a continuous conductive network that facilitates electron percolation. Therefore, it is crucial to develop new reactors that facilitate continuous and uniform semi-solid slurry replacement while maintaining uninterrupted energy storage.



Figure 4: a) A home-made flow battery prototype with semi-solid electrolytes. **b**) Cycling performance of the as-prepared semi-solid based seawater battery at 1 mA in static mode.

4. Additional Discussion

Actually, several approaches to utilize seawater have been developed [42, 43]. For example, pressure-retarded osmosis (PRO) is a process that harnesses salinity-gradient energy by employing semipermeable membranes [44-46]. This method facilitates the movement of water from a low-concentration solution, such as river water, brackish water, or wastewater, into a high-concentration draw solution. For seawater filtration facilities, the ability to identify appropriate methods and locations for the disposal of concentrated brine presents a significant environmental challenge [47-49]. Many designs, including those utilizing PRO, often depict discharge streams returning to the ocean, which could result in detrimental effects on nearby marine ecosystems [44, 50]. Furthermore, the construction of such a PRO system incurs significant costs, and there is a pressing need for the development of an efficient, high-performance, and cost-effective osmosis membrane to enhance the practicality of these technologies. In contrast, in author's opinion, harnessing the ocean's relentless movements for energy (known as blue energy) [25, 51, 52] is one of the most promising approaches toward greener future.

For the concept proposed here, a significant benefit of employing Na⁺ and Cl⁻ as the active materials is the ability to utilize an abundantly available resource that typically necessitates energy for appropriate disposal when in its concentrated state. Specifically, a significant challenge faced by seawater filtration facilities is the identification of appropriate methods for the disposal of concentrated brine, which poses a substantial environmental threat. The creation of cost-effective, safe, and efficient battery systems capable of harnessing concentrated seawater not only offers a means to store electricity but also addresses ecological issues (also can be used for desalination). The glaring advantage of such a system is that it could be located adjacent to a desalination plant and use the concentrated brine as a feed source for renewable energy storage.

To effectively harness the seawater resource, an electrochemical approach should be viable. As highlighted in their latest progress report, more efforts are needed to develop safer and scalable seawater batteries. To emphasize again, a battery system that can only utilize either Na⁺ (as listed in Table **1**) or Cl⁻ ion (e.g., chloride-ion battery [53]) is not good enough for this purpose. Additionally, coupling solar energy and seawater resource is a "green" idea and will offer a cost-effective and sustainable solution to meet the growing demand for electricity. Nonetheless, by doing so, we will have to pay attention to undesirable corrosive chemicals detract from this system causes environmental problems and waste of resources. To make it short, the minimum requirements of a seawater battery are i) no alkali metal is used; ii) no toxic gas is involved; iii) all salt ions (i.e. Na⁺ and Cl⁻) are utilized for the energy storage and conversion and iv) highly resilient in marine environments. It is foreseeable that the future technical advancements in seawater battery systems and other related aspects will make this battery technology more competitive.

5. Conclusions

In sum, a seawater battery consisted of silver (+ve) and carbon-coated NaTi₂(PO₄)₃ (-ve) was studied. Owing to the shuttle effect of Ag⁺ ions, using an anion-exchange membrane as the separator for such a battery system was particularly important to achieve a stable and reversible cell performance. By performing the four-electrode measurements, it was found that the concentration changes had little impact on the Ag electrode potential and the voltage across the membrane at the tested conditions, except for the negative electrode side with intercalation/deintercalation chemistry. To demonstrate the scaling-up of this battery technology, semi-solid electrolytes were also developed and applied to the battery system. The preliminary results showed that the charge and discharge plateaus were similar to those of the battery system with solid electrodes. However, the capacity fading was quite serious, most likely due to the side reactions like water splitting along with the H₂ evolution at the negative side. Further technical advancements in key materials and reactor design are still needed to meet the future demands. Additionally, the advancement of new reactors that enable the continuous and uniform replacement of semi-solid slurries, along with a continuous conductive network, is essential for the development of next-generation scalable energy storage solutions. It is believed that safer and scalable seawater batteries will make a big difference across the globe.

Conflict of Interest

The author declares that he has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

Funding

This work is mainly supported by Fondazione Oronzio e Niccolò De Nora Fellowship and partially supported by Shanghai Oversea Talents Award.

Acknowledgments

The author would like to thank Christopher Karpovich for the assistance in designing Fig. (1) of this work and Dr. Hang Wang for the assistance in literature searching as well as the other group members of TMD Lab for the helpful discussion. Most data were obtained with the facilities at Yale University.

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