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Preliminary Study of $NaFePO_4$ Cathode Material Synthesis through Electrochemical Sodiation for Sodium-Ion Batteries

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ABSTRACT: Since sodium is more readily available and less expensive than lithium, sodium-ion batteries are considered promising substitutes for lithium-ion batteries. $NaFePO_4$ has garnered interest among different cathode materials because of its favorable structural stability, affordability, and eco-friendliness. In this work, an aqueous Na_2SO_4 electrolyte was used in an electrochemical sodiation process to create $NaFePO_4$ cathode material. In order to facilitate sodium insertion into the $FePO_4$ structure, the synthesis was performed by immersing $FePO_4$/Al sheets in the electrolyte under electrolysis conditions. Changes in the Fe and phosphate groups during sodiation were shown by Fourier transform infrared analysis, which revealed peak shifts from 560 cm^{-1} to 555 cm^{-1} and from 1000 cm^{-1} to 997 cm^{-1}. $FePO_4$ particle diameters were found to be between 0.4 - 0.6 µm and around 0.5 µm for $NaFePO_4$ by scanning electron microscopy examination. The existence of sodium with a mass percentage of 12.26 % was also verified by energy-dispersive X-ray analysis. According to electrochemical testing, the produced sodium-ion battery has a coulombic efficiency of 17.86 % and a specific capacity of 9.23 mAh/g at 0.1 C. The relatively low electrochemical performance indicates that the electrode composition and electrochemical sodiation procedure still require improvement. However, the findings suggest that a straightforward method for $NaFePO_4$ cathode production is electrochemical sodiation.

Keywords: Electrochemical Sodiation, $NaFePO_4$, Sodium-Ion Battery, Cathode Material, Energy Storage.

1. INTRODUCTION

The development of effective and sustainable energy storage systems has grown in significance due to the steady increase in global energy consumption. Lithium-ion batteries (LIBs) have dominated the rechargeable battery industry because of their high energy density and strong electrochemical performance. However, LIB has problems such as the scarcity of lithium resources,

the comparatively high cost of production, and issue safety with thermal stability [1, 2, 3]. These limitations have encouraged the development of alternative battery systems based on more abundant and cost-effective materials.

Sodium ion batteries (SIBs) have a lot of interest lately as viable substitutes for LIBs. Sodium has electrochemical characteristics that are comparable to those of lithium and widely accessible and reasonably priced. The sodium resources are more abundant, which makes SIBs appealing for large scale energy storage applications [4, 5]. The electrochemical performance of SIBs is strongly influenced by the cathode materials.

$NaFePO_4$ has become a viable material among cathode possibilities due to its strong structural stability, affordability, environmental friendliness, and generally safe phosphate based. According to earlier research, $NaFePO_4$ based cathodes show stable cycling behavior and promising electrochemical performance for SIBs applications [6]. In addition, Fe based phosphate materials are considered more sustainable than cathode materials such as cobalt or nickel.

Numerous techniques, such as solid-state method, hydrothermal, sol-gel, and microwave assisted synthesis, have been reported for the manufacture of $NaFePO_4$ cathode materials [7]. Each approach still has several drawbacks. Hydrothermal methods require high-pressure systems and more intricate operational procedures [7], solid-state synthesis requires high temperatures and long processing durations [8]. Although microwave-assisted synthesis can shorten synthesis times, scalability and temperature consistency remain issues. Sol-gel techniques, on the other hand, offer good compositional uniformity but need comparatively difficult preparation and post-treatment procedures [3]. Table 1 summarizes a comparison of various $NaFePO_4$ production techniques in earlier research.

Table 1. Comparison of $NaFePO_4$ synthesis methods in previous research

No	Method	Advantages	Limitations	References
1.	Sol–gel	Good compositional homogeneity	Complex preparation and post-treatment	[3]
2.	Hydrothermal	Good crystallinity and morphology control	Requires high-pressure system	[7]
3.	Solid-state	Simple and widely used	High temperature and long processing time	[8]

As a method for direct sodium insertion into $FePO_4$ structures, electrochemical sodiation provides a very simple and low temperature method for $NaFePO_4$ synthesis as compared to early synthesis techniques. This technique can simplify the synthesis process and reduce excessive heat treatment. Rahmawati et al. [9] demonstrated that $NaFePO_4$ could be synthesized through electrochemical sodiation using sodium containing electrolytes. There are very few investigations on electrochemical sodiation with aqueous electrolytes, and problems including side reactions, gas development, and non-uniform sodium insertion are significant concerns that need more research.

Therefore, the preliminary production of $NaFePO_4$ cathode material using an electrochemical sodiation procedure with an aqueous Na_2SO_4

electrolyte is the primary of this study. To assess sodium incorporation into the $FePO_4$ structure, the produced materials were examined using energy-dispersive X-ray (EDX) analysis, Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). To evaluate the viability of this synthesis method for SIBs applications, the electrochemical performance of the produced SIBs was also examined.

2. MATERIALS AND METHODS

2.1 Material and Equipment

The powder of $FePO_4$, acetylene black (AB), polyvinylidene fluoride (PVDF), and N-methyl-2-pyrrolidone (NMP) were used for cathode preparation. The current collector was made of aluminum foil. The electrochemical sodiation technique employed purified water and sodium sulfate (Na_2SO_4, technical grade). Graphite was used as the anode material for sodium-ion battery fabrication.

2.2 Methodology

2.2.1 $FePO_4$/Al Electrode Preparation

Cathode of $FePO_4$/Al electrodes were prepared using $FePO_4$, AB, and PVDF with a mass ratio of 7:2:1. The mixed powders were homogenized using a mortar and pestle, followed by the NMP added incrementally to form a slurry. The slurry was coated onto aluminum foil using a doctor blade with a thickness of 0.25 mm and dried in a vacuum oven at 90 °C. The dried electrodes were subsequently cut into several sheets for the electrochemical sodiation process.

2.2.2 Electrochemical Sodiation of $FePO_4$

An aqueous electrolyte (1 M Na_2SO_4) was prepared by dissolving 71 g of Na_2SO_4 in 500 mL of distilled water under continuous stirring at 600 rpm and heating at 100 °C until a homogeneous solution was formed. Na_2SO_4 was chosen because it can provide sodium ions with good chemical stability and adequate ionic conductivity in aqueous systems. In addition, aqueous Na_2SO_4 is relatively inexpensive and safer than several organic sodium-based electrolytes commonly used in sodium-ion battery studies. The electrochemical sodiation process was performed by immersing two $FePO_4$/Al electrodes into the Na_2SO_4 electrolyte and and connecting them to a direct-current (DC) power supply operating at 9 V. Electrolysis was carried out for 5 min to promote sodium insertion into the $FePO_4$ structure. During the electrolysis process, gas bubbles were observed around the electrodes. These bubbles were likely related to hydrogen and oxygen evolution caused by water electrolysis reactions in the aqueous electrolyte.

The sodium insertion process during electrochemical sodiation is proposed to occur through the following reaction:

$$FePO_4(s) + xNa^+(aq) + xe^- \rightarrow Na_xFePO_4(s) \quad (1)$$

In addition, possible side reactions during electrolysis may occur through water decomposition reactions:

$$2H_2O(l) + 2e^- \rightarrow H_2(g) + 2OH^-(aq) \quad (2)$$

$$2H_2O(l) \rightarrow O_2(g) + 4H^+(aq) + 4e^- \quad (3)$$

After electrolysis, the electrodes were dried in a vacuum oven to obtain $NaFePO_4$ cathodes. The electrochemical sodiation process is expected to facilitate sodium-ion insertion into the $FePO_4$ framework through a reduction process under an applied electric field.

2.2.3 Material Characterization and Battery Testing

The synthesized materials were characterized using Fourier transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), and energy-dispersive X-ray spectroscopy (EDX) to observe structural changes, particle morphology, and elemental composition after the electrochemical sodiation process. Graphite was used as the anode material and $NaFePO_4$ as the cathode in

the manufactured sodium-ion battery. To investigate the battery's specific capacity, coulombic efficiency, and charge-discharge experiments were performed at 0.1 C.

3. RESULTS AND DISCUSSION

The FTIR spectra of the $FePO_4$ and $NaFePO_4$ materials at the electrochemical sodiation procedure are displayed in Figure 1. Similar absorption bands linked to phosphate and Fe–O groups can be seen in both samples. The absorption peak in $FePO_4$ at about 560 cm^{-1} moved to about 555 cm^{-1} after electrochemical sodiation, which would suggest that sodium insertion into the $FePO_4$ structure changed the local Fe–O bonding environment. Moreover, following sodiation, the phosphate-related peak moved from about 1000 cm^{-1} to 997 cm^{-1}. Previous research has reported similar peak shifts, which are frequently linked to structural alterations brought on by ion incorporation into phosphate-based cathode materials [10, 11]. The comparatively minor peak changes imply that the $FePO_4$ may be somewhat modified by the electrochemical sodiation process without significant structural changes. Nurohmah et al. have also reported similar findings about structural alteration and ion insertion behavior in sodium-based battery materials [12].

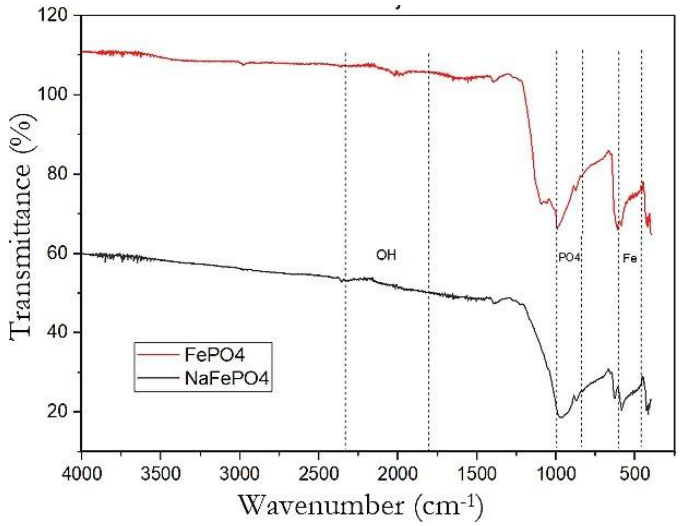

Figure 1. FTIR spectra of $FePO_4$ and $NaFePO_4$ materials before and after the electrochemical sodiation process

Material of $FePO_4$ and $NaFePO_4$ were characterized using a SEM to evaluate their shape, particle size, and agglomeration. Figure 2 displays the pictures obtained from SEM at magnifications of 1000×, 2500×, and 5000×. The $FePO_4$ sample (Fig. 2a—c) showed a distinct granular morphology with a small amount of aggregation. The $NaFePO_4$ (Fig. 3a—c) cathode sheets, in contrast, were less distinct, perhaps because of the PVDF and AB utilized during electrode production. These substances may mask the active material's actual morphology, making it more challenging to see particle properties in the finished cathode sheets. As a result, SEM analysis works better on $FePO_4$ powder samples prior to cathode sheet production. While the $NaFePO_4$ sample showed a comparable average particle size of roughly 0.5 microns, the average particle size of $FePO_4$ was found to range between 0.4—0.6 microns. In comparison to the anticipated nanoscale particle sizes commonly found in high-performance cathode materials, these sizes are very big.

Galceran et al. and Stulasti et al. reported that $NaFePO_4$ can have particle sizes ranging from 50 to 300 nm, suggesting that the greater particle size in this work could lead to slower sodium-ion diffusion, and as a result, poorer electrochemical performance [6, 11].

To determine the elemental composition of $FePO_4$ and $NaFePO_4$ samples and validate sodium incorporation into the structure, EDX analysis was performed. The EDX composition (Table 2) and spectra (Figure 4) showed that the $NaFePO_4$ material had a sodium concentration of 12.26 %wt. This shows that the electrochemical sodiation process was successful in inserting sodium into the $FePO_4$. The EDX spectrum of the $FePO_4$ sample revealed the anticipated presence of iron (Fe), phosphorus (P), and oxygen (O), but no detectable sodium. The FTIR results, which revealed distinctive peak shifts following sodium inclusion, provide more evidence for this. The material $NaFePO_4$ achieved sodium content 12.26 wt.% indicates that sodium insertion occurred significantly. Several identified performance issue, such as the comparatively low specific capacity and coulombic efficiency, may be caused by this non uniform salt distribution. The electrochemical characteristics of the cathode material, such as its charge-discharge behavior and cycling stability, may also be affected by the addition of sodium to the $FePO_4$ structure. Nurohmah et al. have noted similar difficulties with sodium inclusion in phosphate-based cathode materials [4].

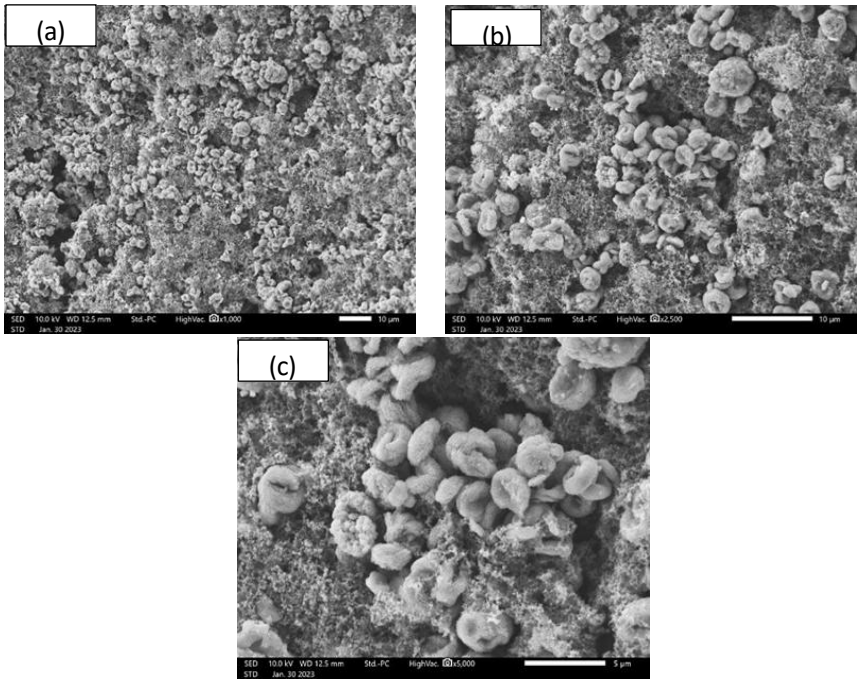

Figure 2. SEM images of $FePO_4$ materials at magnifications of 1000×, 2500×, and 5000×.

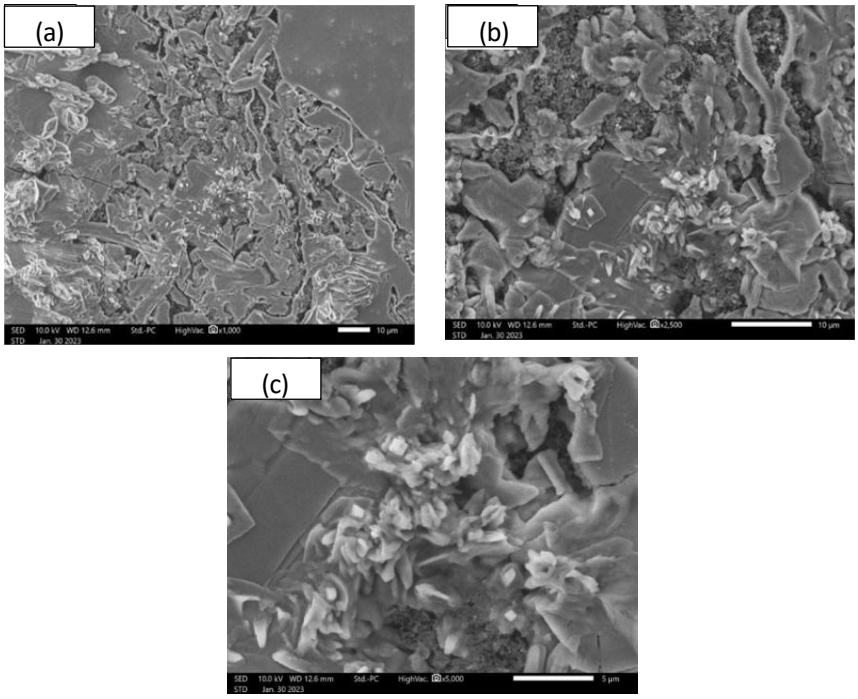

Figure 3. SEM images of $NaFePO_4$ materials at magnifications of 1000×, 2500×, and 5000×.

Table 2. EDX analysis of $FePO_4$ and $NaFePO_4$

Element	$FePO_4$		$NaFePO_4$	
	(wt.%)	(at.%)	(wt.%)	(at.%)
O	9.20	24.67	35.89	48.79
Na	–	–	12.26	15.81
P	9.06	12.55	18.48	17.68
Fe	81.73	62.78	33.37	17.72

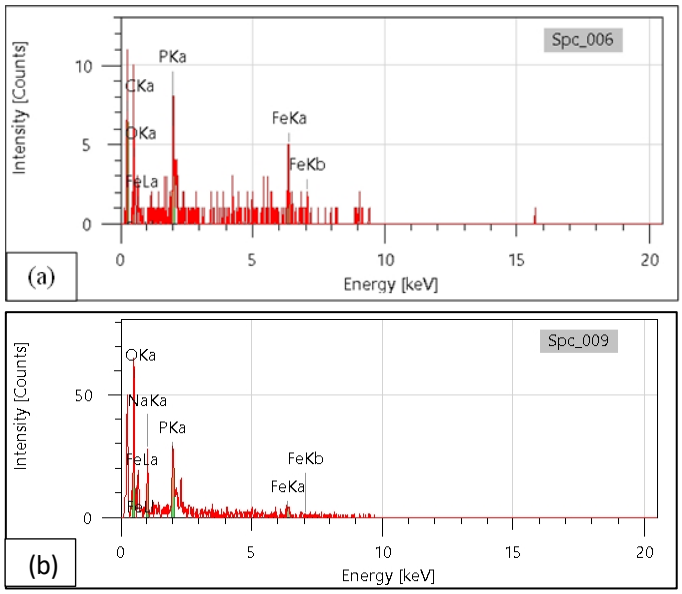

Figure 4. EDX spectra of (a) $FePO_4$ and (b) $NaFePO_4$ materials

Figure 5 illustrates how charge-discharge testing at 0.1 C was used to assess the electrochemical performance of the manufactured $NaFePO_4$/graphite sodium-ion battery. The fabricated cell operated at a nominal voltage of 3.7 V using a cathode with dimensions of 19.5 cm × 5.6 cm, a total cathode mass of 1.35 g, and an active material content of 70 wt.%. The specific capacity of the fabricated battery was calculated using Equation (4):

$$C_{sp} = \frac{C_d}{m} \quad (4)$$

where:

C_{sp} = Specific Capacity ($mAh\ g^{-1}$)

C_d = discharge capacity (mAh)

m = mass of active material (g)

Based on the calculation, the fabricated sodium-ion battery exhibited a specific capacity of 9.23 mAh/g at 0.1 C. The relatively low electrochemical performance suggests that sodium-ion intercalation during cycling was still limited. This behavior may be associated with incomplete sodium insertion during the electrochemical sodiation process, side reactions occurring in the aqueous electrolyte system, and limitations in the cathode formulation.

In addition, the relatively large particle size and limited electronic conductivity of $FePO_4$-based materials may have contributed to sluggish sodium-ion transport within the electrode. Similar low coulombic efficiency behavior in $NaFePO_4$ systems has also been reported by Sun et al., who observed poor cycling performance during the initial cycles [8].

The coulombic efficiency of the fabricated battery was calculated using Equation (5):

$$\eta = \frac{C_d}{C_c}\ x\ 100\% \quad (5)$$

where:

η = coulombic efficiency (%)

C_d = discharge capacity (mAh)

C_c = charge capacity (mAh)

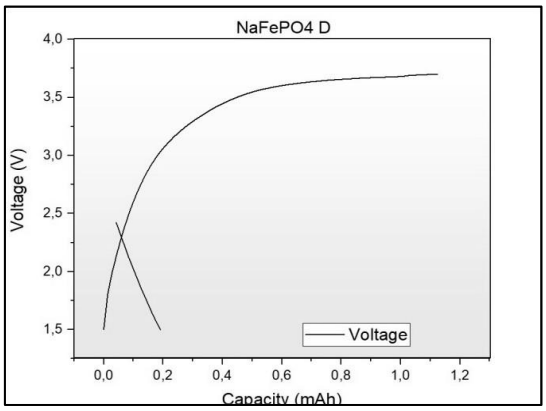

Figure 5. Charge–discharge profile of the $NaFePO_4$/graphite SIBs at 0.1 C.

The battery exhibited a coulombic efficiency of 17.86 %, indicating relatively low charge-transfer efficiency during the electrochemical process. The low coulombic efficiency may be related to irreversible side reactions, incomplete sodium-ion intercalation, and poor contact between the active material and the aluminium current collector. Similar findings have been reported by Sun et al. and Nurohmah et al., where challenges in electrochemical performance of $NaFePO_4$ were attributed to poor sodium insertion and side reactions [8, 12]

To further investigate the low electrochemical performance, the battery was disassembled after testing. Partial delamination of the cathode material from the Al foil current collector was visible, suggesting that there was not enough adhesion between the active material and the Al foil. During charge-discharge, poor adhesion can increase internal resistance (IR) and limit the flow of electrons and ions. Borah et al. have also examined similar findings about how electrode integrity affects battery performance [13].

4. CONCLUSION

The potential of the electrochemical sodiation process for SIBs applications has been indicated by the effective synthesis of $NaFePO_4$ cathodes. Fe (from 560 cm^{-1} to 555 cm^{-1}) and PO_4^{3-} (from 1000 cm^{-1}

to 997 cm^{-1}) showed characteristic peak changes in FTIR spectroscopy, suggesting that sodium was successfully inserted into the $FePO_4$ structure. The addition of sodium ions was confirmed by EDX analysis, which showed a mass percentage of 12.26 %. According to SEM analysis, the $NaFePO_4$ particles were roughly 0.5 μm in size, whereas the $FePO_4$ particles were between 0.4—0.6 μm. The $NaFePO_4$/graphite SIBs showed a specific capacity of 9.23 mAh/g at 0.1 C with a coulombic efficiency of 17.86 %, although these findings suggest that more tuning is required. Poor adhesion between the cathode material and the aluminum current collector, side reactions, and inadequate sodium-ion insertion are probably some of the factors causing the low electrochemical performance. Future work will focus on improving the electrochemical sodiation process, optimizing electrode formulations, and enhancing material adhesion to improve the battery's overall performance

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AUTHOR CONTRIBUTION

The research experiment and writing article was carried out by Amirza Fahma Addiniyyah and Khikmah Nur Rikhy Stulasti. Supervision and review article by Fahru Nurosyid. Conceptualization and wrote final article by Windhu Griyasti Suci. The final report was committed by all contributors.

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