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Journal homepage: https://talenta.usu.ac.id/JoTP

Cycle Voltametry Performance of Nitrogen-Doped Reduced Graphene Oxide Derived from Oil Palm Empty Fruit Bunch for Sodium-Ion Batteries

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ARTICLE INFO

Article history:
Received 29 September 2024
Revised 20 Maret 2025
Accepted 04 April 2025
Available online 21 April 2025

E-ISSN: 2656-0755
P-ISSN: 2656-0747

How to cite:
G. Sihombing, O. M. Yamin, Z. Noer, H. Lubis, M. A. A. Agus, D. Idamayanti " Cycle Voltametry Performance of Nitrogen-Doped Reduced Graphene Oxide Derived from Oil Palm Empty Fruit Bunch for Sodium-Ion Batteries," Journal of Technomaterial Physics, vol. 07, no. 01, pp. 45-50, Feb. 2025, doi: 10.32734/jotp.v7i1.18305.

ABSTRACT

This study investigates the electrochemical performance of nitrogen-doped reduced graphene oxide (NRGO) derived from oil palm empty fruit bunches as an anode material for sodium-ion batteries (SIB). The aim is to evaluate the potential of NRGO to enhance sodium-ion storage through cyclic voltammetry (CV) analysis. The NRGO was synthesized using a modified Hummers method followed by nitrogen doping through thermal treatment under an ammonia atmosphere. Cyclic voltammetry measurements were conducted at scan rates of 0.2 mV/s, 1 mV/s, and 10 mV/s to analyze the redox behavior and charge storage capacity. At a low scan rate of 0.2 mV/s, the current response was minimal, indicating limited sodium-ion intercalation. At 1 mV/s, the current increased, suggesting enhanced ionic mobility, though no distinct redox peaks were observed, implying a primarily capacitive mechanism. At the highest scan rate of 10 mV/s, the current response increased further, but the absence of clear redox peaks persisted, indicating limited faradaic reactions. The initial CV cycles showed a higher current due to the formation of a solid electrolyte interphase (SEI) layer and structural rearrangements, which stabilized in subsequent cycles. The overall charge storage mechanism appears to be dominated by double-layer capacitance rather than faradaic processes. These findings suggest that NRGO derived from oil palm empty fruit bunches exhibits moderate electrochemical performance as a SIB anode material. While the material demonstrates promising charge storage capabilities, further optimization is required to enhance redox activity. Future research should focus on improving synthesis conditions, such as increasing nitrogen doping levels and enhancing surface area, to achieve better electrochemical performance and make NRGO a viable candidate for sodium-ion battery applications.

Keywords: Oil Palm Empty Fruit Bunch, Electrochemical Performance, Sodium Ion Batteries

ABSTRAK

Studi ini menyelidiki kinerja elektrokimia dari nitrogen-doped reduced graphene oxide (NRGO) yang berasal dari tandan kosong kelapa sawit (TKKS) sebagai material anoda untuk baterai ion natrium (SIB). Tujuan dari penelitian ini adalah untuk mengevaluasi potensi NRGO dalam meningkatkan penyimpanan ion natrium melalui analisis voltametri siklik (CV). NRGO disintesis menggunakan metode Hummers yang dimodifikasi, diikuti dengan proses doping nitrogen melalui perlakuan termal dalam atmosfer amonia. Pengukuran voltametri siklik dilakukan pada laju pindai 0,2 mV/s, 1 mV/s, dan 10 mV/s untuk menganalisis perilaku redoks dan kapasitas penyimpanan muatan. Pada laju pindai rendah 0,2 mV/s, respons arus yang dihasilkan minimal, menunjukkan interkalasi ion natrium yang terbatas. Pada 1 mV/s, arus meningkat, mengindikasikan peningkatan mobilitas ionik, meskipun tidak teramati puncak redoks yang jelas, yang mengisyaratkan bahwa mekanisme penyimpanan muatan bersifat kapasitif. Pada laju pindai tertinggi 10 mV/s, respons arus meningkat lebih lanjut, namun tetap tidak muncul puncak redoks yang signifikan, menunjukkan keterbatasan reaksi faradik. Siklus awal CV menunjukkan arus yang lebih tinggi akibat pembentukan lapisan antarmuka elektrolit padat (SEI) dan penataan ulang struktur, yang kemudian stabil pada siklus berikutnya. Secara keseluruhan, mekanisme penyimpanan muatan tampaknya didominasi oleh kapasitansi lapisan ganda daripada proses faradik. Temuan ini menunjukkan bahwa NRGO yang berasal dari TKKS menunjukkan kinerja elektrokimia yang sedang sebagai material anoda untuk baterai ion natrium. Meskipun material ini menunjukkan kemampuan penyimpanan muatan yang menjanjikan, diperlukan optimalisasi lebih lanjut untuk meningkatkan aktivitas redoks. Penelitian mendatang sebaiknya difokuskan pada perbaikan kondisi sintesis, seperti peningkatan kadar doping nitrogen dan perluasan luas permukaan, guna mencapai kinerja elektrokimia yang lebih baik dan menjadikan NRGO kandidat yang layak untuk aplikasi baterai ion natrium.

Kata Kunci: Tandan kosong kelapa sawit, kinerja elektrokimia, baterai ion natrium

http://doi.org/10.32734/jotp.v7i1.18305

1. Introduction

Fertilization techniques are crucial in organic crop cultivation. Uneven, erratic, and unmeasured fertilization processes can negatively affect crop yields [1]. To improve crop production prospects and make fertilization more effective and efficient, an independent liquid fertilizer application system for organic crop cultivation must be developed [2], [3]. This tool is used in organic crop cultivation to achieve better results due to faster spraying time, more comprehensive range, ease of management, and energy saving [4]. This can be used as a guide to create an autonomous liquid fertilizer application device for growing organic crops based on an energy-saving Arduino Uno microcontroller [5], [6].

So far, various types of fertilizers have been used in organic farming, including liquid organic fertilizers such as fermented manure, green manure, and biofertilizers such as Effective Microorganism (EM4). EM4 fertilizer is often used because it contains microorganisms that can improve soil fertility and accelerate the decomposition of organic matter. However, in its application, EM4 fertilizer and other liquid fertilizers still face various challenges, such as uneven mixing due to manual stirring, suboptimal nutrient distribution, and dependence on labor-intensive manual labor. Water and nutrients act as external influences on plant growth, development, and production. However, fertilization will reduce crop yields if not done correctly and according to the plant's needs [7]. To overcome these weaknesses, a method to regulate the application of liquid fertilizer solutions, including water, called fertigation, is needed to help farmers fertilize effectively while reducing the need for labor-intensive manual labor [8], [9]. An automated fertigation method can be used to fertilize liquid effectively and efficiently [10].

The advancement of science has a beneficial effect on the advancement of electronics, particularly in agriculture [11]. One of these is attaching a DC motor to a fertilizer mixer [12], [13]. Many traditional fertilizer mixers are still manually operated, which can result in inconsistent mixing quality. The advancement of science has beneficial effects, especially on the advancement of electronics. Although there are undoubtedly many disadvantages, the novelty of creating a gadget whose rotation can be regulated quickly, precisely, and precisely [14], [15].

Fertilization uses an EM4 solution to supply these nutrients to the soil. Fertigation using drip fertigation automatically feeds nutrients that have been dissolved into the tank to the plants while irrigating and fertilizing them [16]. Since it will not damage the seedling roots of the plants, the drip fertilization technique has several advantages in this test. The difference in density test results between manual stirring as a benchmark for perfect stirring and automatic stirring for EM4 fertilizer gives a success rate of 98.88%. In comparison, green fertilizer has a success rate of 89.82%. So, it can be said that the liquid fertilizer solution stirrer can work well.

The purpose of this research is to develop an independent and energy-saving liquid fertilizer application system based on Arduino Uno microcontroller to improve the efficiency and effectiveness of fertilization in organic crop cultivation. This research also aims to evaluate the application of automatic fertigation methods in optimizing nutrient distribution and reducing the need for labor-intensive manual labor. In addition, this research will analyze the performance of an automatic liquid fertilizer mixer using a DC motor in ensuring more optimal fertilizer mixing compared to the manual method. By comparing manual and automatic stirring techniques in mixing EM4 fertilizer solution, this research is expected to identify the success rate of each method and its impact on the growth and production of organic crops.

2. Method

This research method involves cleaning of the palm empty fruit bunch using running water and drying it until the moisture content decreases. The dried of the palm empty fruit bunch is then ground with a grinding machine and sieved to obtain a powder that passes through a 60 mesh sieve. The preparation of active carbon from the palm empty fruit bunch ncludes sintering at 300°C for 1 hour. The material is then ground and sieved again to a size of 100 mesh. The Graphite Oxide process is synthesized using the Hummers method. One gram of graphite powder is oxidized using 30 ml of H_2SO_4, 0.5 grams of $NaNO_3$, and 0.5 grams of $KMnO_4$. The mixing of materials is done in an ice bath, maintaining the temperature below 20°C. The solution is then stirred at 40°C in a heat bath. After 14 hours of stirring, the solution is transferred to an ice bath, and 60 ml of H_2O is gradually added while keeping the temperature below 100°C. The solution is heated to 50°C in the heat bath and stirred for 60 minutes. The solution is then removed from the heat bath, and 150 ml of H_2O is added. At this stage, the solution turns brown. Four ml of H_2O_2 is added to the solution and stirred for 30 minutes, resulting in a color change to yellow. At this point, graphite oxide dispersed in the solution is obtained. Nitrogen doping into the GO (N-GO) was achieved through thermal treatment under an ammonia (NH_3) atmosphere. The dried GO was mixed with urea at a mass ratio of 1:10 and heated at 600°C for 2 hours in a tubular furnace under an NH_3 flow. This process enables nitrogen atoms to be incorporated into the carbon framework, enhancing its electronic conductivity and electrochemical performance. The cyclic voltammetry (CV) tests of the nitrogen-doped reduced graphene oxide (NRGO) material derived from oil palm empty fruit bunches were conducted to evaluate its potential as an electrode material for sodium-ion batteries. The electrochemical performance was assessed at various scan rates of 0.2 mV/s, 1 mV/s, and 10 mV/s.

3. Results and Discussion

3.1. Low Scan Rate (0.2 mV/s and 1 mV/s)

At the lowest scan rate (Figure 1), the current is minimal across the entire potential window, indicating that no significant redox reactions occur at 0.2 mV/s. The absence of distinctive peaks typically observed in Na-ion systems suggests poor electrochemical activity at this low rate. It can be hypothesized that at such a low scan rate, the kinetics of sodium-ion intercalation and deintercalation are insufficient to trigger substantial redox reactions [14]. The limited electrochemical activity could be attributed to high charge transfer resistance or a poorly accessible active surface area at this stage[15].

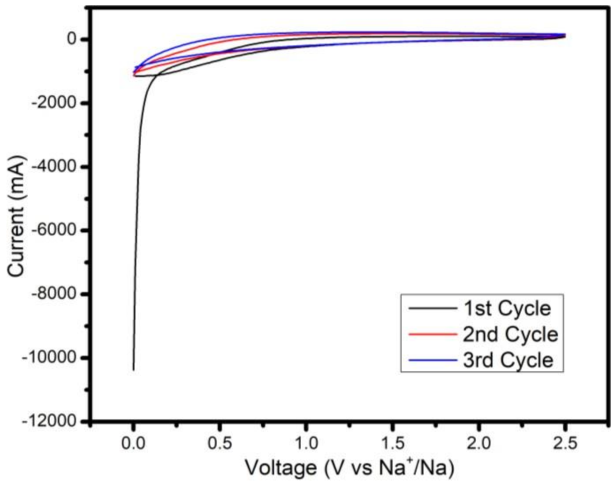

Figure 1. Cyclic voltammograms of NRGO material from oil palm empty fruit bunches at a scan rate of 0.2 mV/s

The findings are consistent with previous studies [16], where a low scan rate often leads to sluggish ionic diffusion within electrode materials. This behavior is especially pronounced in systems where the active material has not fully undergone electrolyte penetration and surface activation.

At a higher scan rate of 1 mV/s (Figure 2), there is a noticeable increase in current, although no redox peaks are yet visible. The enhanced current suggests improved ionic mobility and slight activation of the NRGO material. The absence of defined redox peaks at this stage could imply that while the material has begun to engage in reversible sodium-ion interactions, the mechanisms are primarily capacitive rather than faradaic[17].

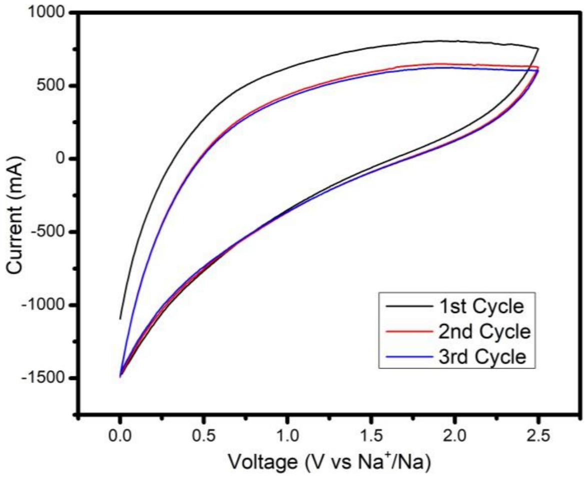

Figure 2. Cyclic voltammograms of NRGO material from oil palm empty fruit bunches at a scan rate of 1 mV/s

This is supported by the broader and more capacitive shape of the CV curves in subsequent cycles, implying that the NRGO material exhibits significant double-layer capacitance but limited redox activity. Similar findings have been observed in graphene-based materials for sodium-ion battery applications, where faradaic reactions are less pronounced due to the material's structure and surface properties [18].

3.2. High Scan Rate (10 mV/s)

At the highest scan rate of 10 mV/s (Figure 3), the CV curves display a marked increase in current. This can be attributed to enhanced ionic mobility and improved interaction between sodium ions and the NRGO material. Although the current increases with the scan rate, the expected redox peaks for sodium-ion intercalation are still not clearly defined, suggesting that the NRGO from oil palm empty fruit brunches may not fully support efficient faradaic reactions even at higher rates [16].

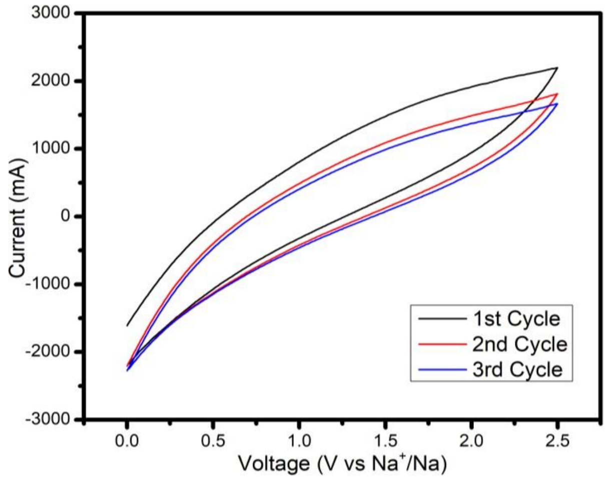

Figure 3. Cyclic voltammograms of NRGO material from oil palm empty fruit bunches at a scan rate of 10 mV/s

The anomaly observed in the first CV cycle in each figure can be attributed to the formation of a solid electrolyte interphase (SEI) layer, structural rearrangements in the electrode material, and the initial wetting of the electrolyte. During the first cycle, irreversible reactions occur as electrolyte components decompose, forming an SEI layer that stabilizes in subsequent cycles. Additionally, sodium-ion insertion induces structural modifications, activating previously inaccessible sites and altering charge transfer dynamics. The gradual infiltration of the electrolyte into the porous NRGO structure further contributes to the initial variation in electrochemical response. These factors collectively result in a higher current response in the first cycle, which stabilizes in later cycles as the electrode reaches electrochemical equilibrium.

The slight increase in current with successive cycles can be attributed to improved electrode wetting and reduction in internal resistance, leading to better electrolyte penetration and surface interaction. However, the absence of distinct redox peaks points to a primarily non-faradaic mechanism, dominated by double-layer capacitance. This behavior is typical of materials with limited redox-active sites or slow ion diffusion dynamics, as previously noted in similar carbon-based electrodes [14], [15], [17], [18], [19].

4. Conclusion.

The CV results indicate that the NRGO material derived from oil palm empty fruit bunches shows potential as an electrode for sodium-ion batteries, although further optimization is needed to enhance its redox activity. The absence of distinct redox peaks at all scan rates suggests limitations in supporting faradaic reactions, possibly due to structural defects or insufficient activation of nitrogen-doped sites. However, the observed charge storage capability suggests that NRGO could be further developed to improve sodium-ion storage efficiency through faradaic mechanisms. Therefore, further research is required to modify the synthesis conditions, such as increasing nitrogen doping levels or enhancing surface area, to improve the material's electrochemical performance. With these improvements, NRGO has the potential to become a more competitive candidate for sodium-ion battery applications.

Acknowledgments

The authors are grateful to Direktorat Riset, Teknologi, dan Pengabdian Kepada Masyarakat, Direktorat Jenderal Pendidikan Tinggi, Riset, dan Teknologi, Kementerian Pendidikan, Kebudayaan, Riset, dan Teknologi Republik Indonesia for Fiscal 2024 for providing funding in the future entire completion of this research with contract number 103/E5/PG.02.00.PL/2024, dated 11 June 2024, 022/LL1/AL.04.03/2024, Number 011/LPPM-Unham/VI/2024 dated June 20, 2024; June 21,2024.

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