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The Effects of Graphene and Acetylene Black as Conductive Materials for Lithium-Ion Batteries

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Abstract: The quality of lithium-ion batteries is significantly impacted by the cathode. Besides their advantages in capacity and cycle life, NMC cathode has low electronic conductivity, which can affect electron transfer. To improve conductivity, the cathode material can be added with conductive additives. Commonly utilized as a conductive additive in the cathode of lithium-ion batteries is acetylene black. On the other hand, graphene offers superior properties, such as its large active surface area and conductivity. This research was conducted to compare AB, graphene, and their combinations as conductive additives for NMC cathode. The test results show that the combination of AB and graphene with a ratio of 1:1 produces the highest specific capacity, which is 161.32 mAh/g. The rate performance result produced by this combination is very good, with 91.38% and 80.07% capacity retention for discharging and charging rate performance, respectively, at 3C current. For the life cycle test after 50 cycles, the combination of AB and graphene with a ratio of 1:1 resulted in a capacity retention of 93.26%, which was higher than a battery that only uses AB or graphene as a conductive material at the cathode. Therefore, the combination of graphene and AB as conductive materials in lithium-ion batteries can produce batteries with good performance.

Keywords: acetylene black; cathode; conductive; graphene; lithium-ion battery

1. Introduction

Energy sources from new and renewable energy are needed by the world today^{1,2}. The most potential power source for portable devices is lithium-ion batteries because of their outstanding energy and power density^{3,4}. When compared to secondary batteries of a previous generation, this battery offers several advantages, including light weight, low price, high level of energy, no memory effect, environmental friendliness, and very safe^{5,6}. Cathode, anode, electrolyte, and separator are important parts of lithium-ion batteries⁷.

One way to increase the capacity of a battery is to modify the material that supports the cathode, or the positive electrode of the battery^{8,9,10}. The LIB cathode is composed of an active material, a conductive agent, and a polymeric binder. Lithium Nickel Manganese Cobalt Oxide (NMC) is the active material that is most frequently utilized today¹¹. Lithium batteries' cathode material, NMC, provides great benefits like a large capacity, a high

current rate, a long cycle life, and a relatively high volume capacity¹². Unfortunately, this material's electronic conductivity is lower and becomes worse as the lithium level rises^{13,14}.

During the manufacture of the electrode, a conductive material and a binder are needed. The conductive material and the active material are bonded together by the binder to adhere to the Al foil. This binder can improve the coating's mechanical stability^{15,16}, and mechanical link between the adjacent active¹⁷. The binder that is often used is polyvinylidene difluoride (PVDF). Liu reported that increasing the PVDF content in the cathode electrode can lead to a decrease in the cell impedance and electron conductivity of the cathode¹⁸.

Therefore, additional conductive material is needed. The conductivity of the electrode is significantly increased by the conductive substance, which is primarily carbon-based, and creates an electron transport percolation network in the electrode layer¹⁹. Thus, electron transfer will be easier²⁰. For lithium ion batteries, a variety of

conductive materials have been created, including carbon fiber, activated carbon, carbon black (CB), AB, KS6, and graphene^{21),22),23),24)}.

Research on conductive carbon material addition to battery cathodes was started in 2001. Huang made LFP cathode nanocomposites with the addition of conductive carbon which was proven to increase battery capacity up to 90% of its theoretical capacity. In addition, the cathode's insertion of conductive carbon also results in a battery with good rate ability and cycle life²⁵⁾. Conductive materials like carbon black are commonly employed in the cathode to provide electrical conductivity at the electrodes²⁶⁾. A unique variety of conductive carbon black, acetylene black (AB), is produced when acetylene undergoes exothermic decomposition at medium or high temperatures²⁷⁾. In lithium-ion battery cathodes, acetylene black (AB) has long been utilized as a conductive additive. AB has been widely used as an electrode additive because of its electronic conductivity compared to other carbon compounds, including activated carbon (AC)²⁸⁾. Several previous researchers tested the cathode material by using AB as a conductive additive^{29),10)}. In 2018, Haris conducted battery testing with nano-sized V₂O₅ and acetylene black. The conductive character of acetylene black, which may enable diffusion and can carry charge effectively so that the cathode's ionic/electronic conductivity improves and its resistive value is reduced, also contributes to improved battery performance. So that the use of conductive materials such as acetylene black can be used as a promising material for battery cathodes³⁰⁾.

However, conductive materials such as graphene offer superior features include their conductivity and great active surface area^{31),32)}. Graphene is a material that has been widely used in various applications, especially in conductive coatings and supercapacitors³³⁾ because graphene has high thermal, mechanical, and electrical characteristics³⁴⁾. Graphene has also been used as an additional conductive material in the LFP cathode and provides excellent performance³⁵⁾. Wang has tested LFP, LCO, and NMC cathode materials with and without graphene in 2019. When compared to commercially available materials with no addition of graphene, it has been demonstrated that using graphene at the cathode of lithium-ion batteries increases their specific capacity. The conductive network on the graphene material reduces the internal resistance of the cathode, so the use of graphene can make LIB cathodes have high performance³⁶⁾. This opens up opportunities for using graphene as an additional component that enhances conductivity to the lithium-ion battery cathode.

In 2020, the concurrent use of AB and graphene was evaluated at the lithium-sulfur battery cathode, resulting in increased capacity with high coulomb efficiency³⁷⁾. This shows that the conductive material greatly determines the performance of a cathode. The higher conductivity that can be produced, the easier the electron transfer occurs, so that an increase in battery performance

can be obtained²⁰⁾. So, the selection of conductive material at the NMC cathode is very necessary to determine the most suitable material for the cathode used. In addition, the right composition of conductive material will produce the best battery performance. This research was done to evaluate the effectiveness of AB and graphene and their combination as conductive materials at the cathode of NMC batteries using a simple solid state method.

2. Materials and Methods

2.1 Materials

The main materials needed in this research are active cathode material in the form of NMC 811 and conductive materials such as acetylene black or AB and graphene powder. Other additional materials are PVDF, NMP, graphite, CMC, SBR, deionized water, LiPF₆ electrolyte with a concentration of 1M in a 3:7 volume ratio of EC+EMC, and PP separator. All of the materials were used without further purification.

2.2 Methodology

Several formulations for NMC cathode compositions were made for this investigation. The formulation used was to determine the optimum conductive material composition for the NMC cathode. The NMC cathode was made up of 5%wt of PVDF, 0-8%wt of graphene, 0-8%wt of AB, and 87%wt of NMC active material. The first formulation labeled NMC/AB, contained 0% graphene with a ratio of graphene:AB = 0:2. The second formulation, labeled NMC/AB/G, contained 4% graphene with 4% AB with a ratio of graphene:AB = 1:1. While the third formulation, labeled NMC/G, contained 8% graphene with a ratio of graphene:AB = 2:0. To create a uniform powder mixture, all of the formulation materials were combined using the solid state method.

2.2.1. Preparation of Cathode

Cathode preparation was started with sifting NMC powder on a 200 mesh sieve, then mixing it with graphene and AB according to the formulation determined by the solid state method using a mortar for approximately 1 hour. The mixture of NMC, graphene, and AB powders was mixed in a PVDF solution in NMP solvent and then stirred for about 3 hours at 1000 rpm to make a homogeneous paste. The slurries were then cast onto aluminum foil with a thickness setting of 200 microns. The slurry was then dried for an entire night at 90°C in a vacuum oven.

2.2.2. Materials Characterization

Material characterization was performed using XRD and SEM. Electrochemical tests on 18650 cylindrical cells were performed using a graphite anode, a PP separator, and 1M LiPF₆ in EC+EMC with a 3:7 (volume ratio) electrolyte. The battery performance of cylindrical cell batteries was then analyzed for electrochemical

performance using a NEWARE Battery Analyzer and BTS program over a voltage window of 2.6–4.3 V.

3. Results and Discussion

By using an X-Ray Diffractometer (XRD), starting material characteristics were tested. XRD testing was conducted to observe the crystal structure of the material formed. Figure 1 displays the results of the XRD tests performed on the cathode materials.

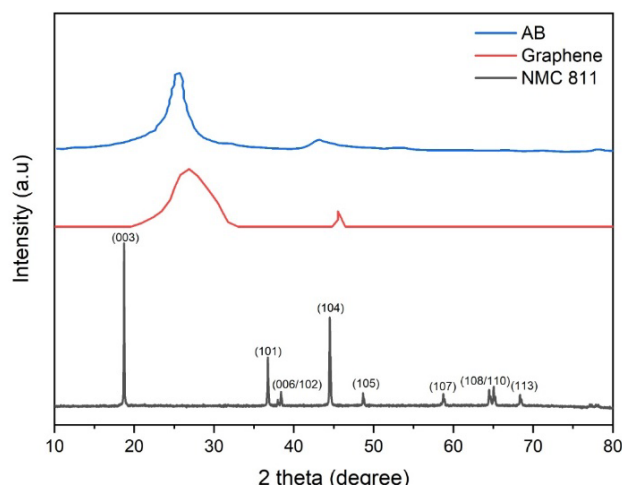


Fig. 1: Test XRD patterns of pristine NMC, Graphene, and AB materials used

In pure NMC, all of the diffraction peaks are distinct and sharp, as shown in Fig. 1, showing that the starting material has good crystallization. With the exception of one impurity phase, all materials may be classified as layered oxide structures based on the hexagonal α - NaFeO_2 structure with space group $R3m^{38}$. The material has a highly organized layered structure, as shown by the peak separations of (006)/(102) and (108)/(110) at 38° and 65° , respectively³⁹. Graphene's XRD pattern is depicted in Fig. 1 has a sharp peak at 25° , which can be attributed to the complete oxidation of graphite to graphite oxide³⁹. The raw material for acetylene black (AB) is compatible with the crystal structure of graphite²¹, but exhibits poor crystallinity properties. No impurities were found in any of the raw materials, proving their high purity and allowing for full material-only reference in subsequent measurements and evaluations of cell performance.

The material's morphological structure was examined with a Scanning Electron Microscope (SEM). In Figure 2(a), the pure NMC particles have a uniform particle size between 1–20 μm . Figure 2 (b) is an NMC/AB particle which is indicated by the presence of AB particles around the NMC particle. AB material is visible on the surface of the NMC material that connects one NMC particle to the

others. Figure 2 (c) represents the NMC/G particles, where flat solids shaped like corn flakes are seen between the uniform NMC particles, indicating the presence of graphene around the NMC particles⁴⁰. While Figure 2(d) shows NMC/AB/G particles, showing NMC particles surrounded by graphene flakes, with AB particles filling the empty area around the NMC and graphene particles. So that the conductive particles are shown to wrap the NMC active material properly. The distribution of NMC material with AB and graphene was further tested with element mapping, which can be seen in Fig. 3. Figure 3 (a) shows the distribution of elements in NMC active material. Figure 3 (b–d) shows the distribution of elements in NMC/AB, NMC/G, and NMC/AB/G, respectively. The blue color in Fig. 3(b) indicating the presence of carbon element in AB. The carbon element is seen to be distributed on the surface of NMC material quite well, but the mapping of carbon atoms is not uniform all over the part. While in NMC/G, the intensity of the carbon element in the graphene particles shown in Fig. 3 (c) is more clearly observable. While in Figure 3 (d), the NMC/AB/G particles show that the AB and graphene materials appear more evenly distributed when used together. The combination of graphene around the NMC material and AB filling in the surrounding area makes the conductive material better distributed. Based on this material test, mixing materials with the solid state method is able to mix these materials evenly. The addition of a conductive material with a uniform distribution can enhance the material's electrochemical characteristics, so that it can improve battery performance⁴¹. Thus, the presence of AB and graphene surrounding the NMC particles can increase the conductivity of the particles, increasing electron transfer and resulting in better cathode performance.

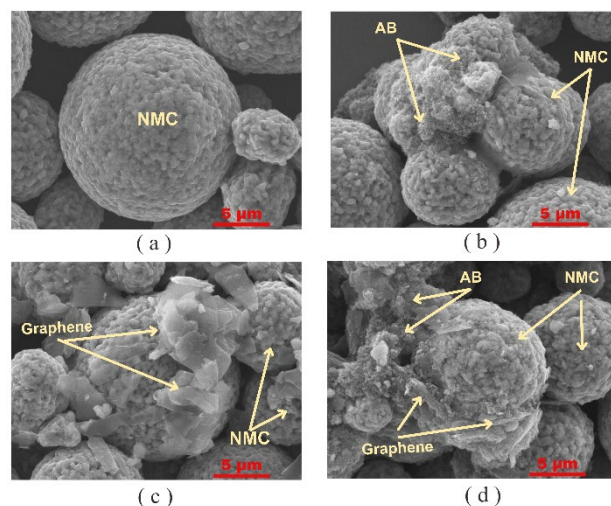


Fig. 2: Morphological structure test results of (a) NMC, (b) NMC/AB, (c) NMC/G, and (d) NMC/AB/G

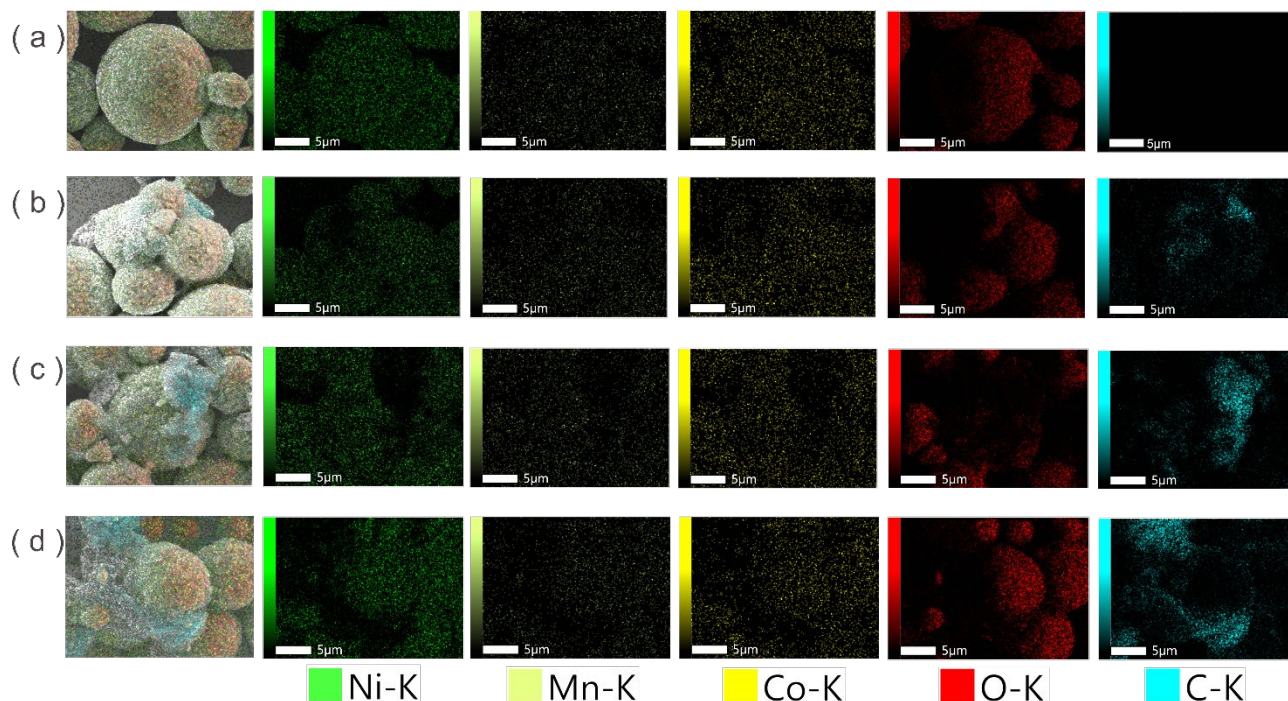


Fig. 3: Element Mapping test results of solid-state result powder. The element distribution of (a) NMC, (b) NMC/AB, (c) NMC/G, and (d) NMC/AB/G

Table 1. The Cathode's Material Composition

No	Sample Code	Material Composition				Graphene to AB mass ratio
		PVDF	NMC	Graphene	AB	
1.	NMC/AB	0.05	0.87	0	0.08	0 : 2
2.	NMC/AB/G	0.05	0.87	0.04	0.04	1 : 1
3.	NMC/G	0.05	0.87	0.08	0	2 : 0

Battery electrochemical analysis was performed on 18650 cylindrical cells by testing the battery's specific capacity, rate capability, and cycle capability. Full-cell batteries were tested using graphite-based anodes with celgard separators, and filled with 1M LiPF₆ electrolyte in EC+EMC at a volume ratio of 3:7. The cathode film was prepared based on the mass composition of each material as shown in Table 1. The charging and discharging test results for the first cycle are shown in Fig. 4.

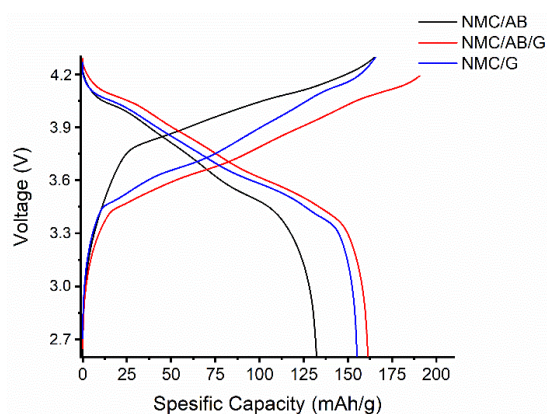


Fig. 4: Charge-discharge Test Results

With graphene addition, there is a significant increase in specific capacity. Based on the test results, the batteries showed good performance. The specific capacity of the batteries produced was 132 mAh/g and 155.11 mAh/g for NMC/AB and NMC/G batteries, respectively. However, batteries with conductive additives in the form of a mixture of AB and graphene, or NMC/AB/G batteries, produced a better capacity than NMC/AB and NMC/G batteries, which was 161.32 mAh/g. This shows that the type of conductive material used affects the battery capacity. The presence of graphene at the right composition can improve electrochemical performance³⁹⁾. The volume fraction of the conductive filler material affects the electrical conductivity value. As the concentration of the conductive material increases, a continuous network is formed in the matrix which facilitates conduction for the transport of the conduction electrons⁴²⁾. However, a decrease in specific capacity also occurs if the amount of graphene added is too much. This is in accordance with the results of SEM tests on the material, which show that the right composition between AB and graphene can connect the NMC active material well, so that electron transfer can be more effective.

Battery life testing is carried out to determine how long the battery can be used. Cycle life testing was done by

testing the remaining battery capacity after going through 50 cycles at a discharge current of 1C and a charge current of 0.5C. The test results of the battery cycle capability are shown in Fig. 5. Based on the test results, it was found that the decrease in the capacity of the NMC/AB battery after 50 cycles was the largest, which was around 10.05% with a remaining capacity of 89.95%. While on the NMC/G battery, the remaining capacity after 50 cycles was 91.40%. The NMC/AB/G battery had the highest remaining capacity among the others after 50 cycles, which is 93.26%. This shows that NMC/AB/G batteries can maintain better capacity under repeated use. This ability shows that NMC/AB/G batteries are capable of long-term use.

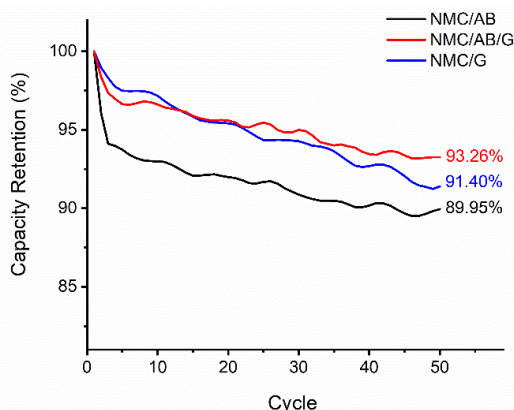


Fig. 5: Cycle Life Test Result

The addition of graphene as a conductive material in the battery was then tested to determine its discharging and charging capabilities at high currents. The battery tested was NMC/AB/G battery, which produced the best performance in terms of capacity and lifetime. The results of the rate ability test are shown in Fig. 6. The test was carried out at a current of 0.5-3C to test the ability of the battery with various discharging and charging currents. The specific capacity decreases as the current applied to the battery increases. In rate discharging ability test, there was a decrease in the specific capacity of the NMC/AB/G battery by 2.51%; 4.10%; 6.62%, and 8.62% for each variation of the discharging current of 0.5C until 3C. Furthermore, the rate ability test for charging rate was carried out which obtained results in line with the discharge rate ability test. In the charging rate capability, the specific capacity of the NMC/AB/G battery decreased by 5.85%; 9.05%; 14.81%, and 19.93% for each variation of the charging current 0.5C until 3C.

After the discharging and charging rate tests were carried out, the batteries were tested for their ability to maintain capacity after being applied to high currents. This was done by applying a current of 0.1C to each battery. The capacity that can be maintained was quite good, at 95.21% of the first discharge capacity for NMC/AB/G battery. The high electron conductivity of graphene helps to provide a conduction path between the

cathode NMC particles. This causes the electrical contact between the NMC particles to be increased, so that the rate capability is increasing. It means that the presence of graphene can improve battery properties of high currents⁴³).

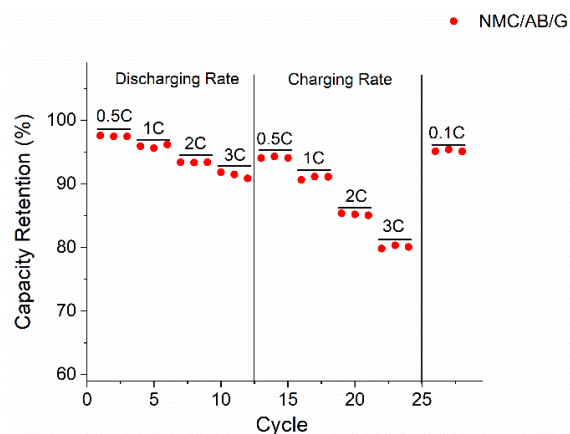


Fig. 6: Rate Ability Test Result

These findings lead to the conclusion that the partial substitution of AB with graphene can increase the stability of the battery cycle. This is due to the graphene structure that forms layers so that it can reduce the impedance of Li^+ migration across graphene's carbon layer⁴⁴). The results of the three compositions are comparable in terms of life cycle, specific capacity, and rate ability. Graphene is superior to other carbon-conductive materials like AB in terms of surface area, conductivity, and mechanical properties⁴⁵). However, graphene can agglomerate and reform graphite thus limiting its advantages in its application. AB can act as a spacer that inhibits graphene agglomeration to maintain its layer structure⁴⁶). This can cause the penetration of the electrolyte to be optimized and electron transfer will be easier, so that the electrode performance will increase⁴⁷). Thus, the combination of graphene and AB as a conductive agent in lithium-ion batteries can produce batteries with good performance. Further research related to determining the optimum composition ratio between graphene and AB is still needed to obtain the best results.

4. Conclusion

Today's electrical appliances are increasingly using li-ion batteries. The cathode produces a significant effect on the performance of li-ion batteries. Due to their benefits in terms of high current rate, high capacity, relatively high volume capacity, and longer lifetime, NMC cathodes are being used more frequently. However, the low electronic conductivity of NMC may impact electron transfer. To increase the conductivity and improve the electron transfer process, there must be conductive components added in the cathode material. Acetylene black is often used in li-ion battery cathodes as a conductive additive. On the other hand, graphene has superior qualities such as

a very large active surface area and conductivity. The purpose of this study is to compare the performance of batteries with a variation of conductive materials using AB, graphene, and a mixture of both on the NMC cathode. According to the test results, AB and graphene combined in a 1:1 ratio produce the maximum specific capacity, which is 161.32 mAh/g. The rate performance is relatively good, with 91.38% and 80.07% for discharge and charge rate performance at 3C current in this combination. The capacity retention of the battery using only AB or graphene as the cathode conductor material was lower after 50 cycles in the life cycle test than the battery using AB and graphene in a 1:1 ratio, which retained 93.26% capacity. Therefore, the combination of graphene and AB as conductive materials with the right composition in lithium batteries can generate batteries with great performance.

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