Article

Incorporating graphite from pencils as a component of lithium-ion batteries

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SUMMARY

Rechargeable lithium-ion batteries operate on reversible insertion of lithium ions into host anode and cathode active materials to realize energy storage, and graphite-based anode is one of the many components of a lithium-ion battery where oxidation, or transfer of electrons occurs. With global energy consumption rising by 5% in 2021 alone, and the United States throwing away 1.6 billion pencils annually, sustainable power options are essential for the future. Here we hypothesized that graphite derived from pencils could be directly used for lithium-ion battery anode applications, especially from pencil stubs too small for writing. To test this hypothesis, we constructed and tested 20 lithium battery coin cells (10 control, 10 pencil graphite-based cells). We focused on evaluating cell charge and discharge properties through a battery tester. Results of the testing showed that pencil graphite cells had a first charge capacity of 397.3 mAh/g on average, a poor coulombic efficiency of around 52%, and low-rate capability compared to the control group. Past research reveals that up to 25% of a pencil is composed of non-graphite elements, likely a major source for poor efficiency rate capability. To overcome this obstacle, we applied prelithiation to pencil graphite anode via lithium metal in a Swagelok half cell, resulting in improved efficiency and better cycle life than control in a full cell built with $LiFePO_4$ cathode. These results implied that pencil graphite could not be used as a drop-in replacement for battery-grade graphite, but prelithiation treatment could make it a viable material for lithium-ion batteries.

INTRODUCTION

Much of today's population is very dependent on energy for their daily lives, whether this is in the form of fossil fuels to power their cars or batteries found in cell phones and other electronics (1). It is very important to find alternative energy sources and more effective ways to produce power due to the rising energy demands in the world. Lithium-ion batteries are very good at storing energy because they charge faster, last longer (three times more than nickel-based batteries), and have a higher energy density (100-265 Wh/Kg) for more battery life in a lighter package (2). In commercially produced lithium-ion batteries, a key component of the battery is graphite, whether it be synthetic or natural graphite (3). Due to rising battery demands, more raw materials will be needed to make the

batteries (4). Many alternatives are being researched, such as recycling used batteries (5). Pencils are also widely used and commonly found in the modern world (6). When pencils are used, they gradually become smaller and are eventually thrown away when the pencils are too small to write with. Graphite from pencils may be a practical alternative to the graphite currently used in commercial lithium-ion batteries, which may help solve the issues regarding material shortages in batteries.

In a lithium-ion battery, there are four major parts: cathode (or positive electrode), anode (or negative electrode), separator and electrolyte. The cathode and anode enable the flow of electrical current, the porous polymer separator is placed between the two electrodes to prevent electrical shorting but allows lithium ions to pass through, and the liquid electrolyte promotes smooth transport of lithium ions between the electrodes (7). The most crucial part for this project is the anode. The anode in the battery is one of the two electrodes, which store the lithium ions (7). The composition of the anode is crucial to the battery's energy density and power density (3). Power density is how much power the battery can output compared to its size, while energy density is how much energy the battery can store based on its mass (7). The way a lithium-ion battery works is that the lithium ions travel between the positively charged cathode and the anode through the electrolyte of cell. The electrolyte acts as a catalyst by promoting the movement of ions between the two electrodes. Depending on the type of battery, the electrolyte can be a liquid or a paste-like material (8). This movement of ions stores and releases energy.

For nearly 30 years, graphite has been the best material for the negative electrode in lithium-ion batteries (3). This is because graphite is highly conductive with a conductivity of 2-3 $\times 10^5$ siemens per meter (S/m) in basal plane and can store 372 milliampere per hour/gram (mAh/g) of lithium energy while being reliable and having a good lifespan depending on factors such as temperature (3, 7, 9, 10). Graphite can vary in purity and particle size which will affect its cost. Graphite for batteries is sourced in two ways: it can be naturally found, or it can be synthetically made. Natural graphite is mined while synthetic graphite is produced from petroleum feedstocks; however, both are processed heavily to make them usable (3, 11). Artificial graphite tends to fare better as an anode material due to the more uniform hexagonal structures that are achieved in its manufacturing, allowing it to better store and release lithium ions (11, 12).

Another material also used as an anode material in commercial lithium-ion batteries is silicon. A more modern alternative, it can handle around 10 times as much lithium energy as graphite (13). The downside of using silicon as

opposed to graphite as an anode material is that since it can process more lithium energy, it tends to degrade and fade very quickly and thus is very limited in its lifetime of usage (13). A few previous studies were done to investigate the effectiveness of pencil graphite as a cost-effective alternative for anode materials, their findings suggest that pencil graphite is both more efficient and more cost-effective than industrially used graphite (14, 15). We found the results of previous study surprising because it was conducted with a specific focus on 4B pencils, out of a wide variety of pencils with hardness rated by a combination of letters and numbers (14). Based on the classification of pencil types, 4B pencils differ in composition when compared to 2B pencils (6). 4B pencils are softer which means more graphite than 2B pencils, according to the Faber-Castell scale for pencil hardness (6). The difference in hardness may play a part in how effective the graphite source will be as an anode material.

The average retail price for a single pencil is around $0.10, and it is widely used as a writing or drawing tool in daily life. Given the low cost and abundance of pencils, it is quite apparent that pencil graphite is likely not high quality (16). 4B pencil graphite has been tested for lithium-ion battery anode and showed comparable results to control groups (14). Other types of pencils, such as 2B, as a possible material needs further study. Therefore, we hypothesized that graphite recovered from pencils could be directly employed as an anode for lithium-ion batteries.

We utilized a standard industrial graphite provided by BTR New Energy Materials Co. Ltd., a leading global graphite anode supplier, as the control group (7). Our hypothesis in this study was that there was no significant difference between the control and pencil graphite groups when used as a component for lithium-ion batteries. Initial comparison of pencil graphite and control group via CR2032 coin cell revealed major disadvantages for pencil graphite, namely poor first cycle coulombic efficiency (which led to low capacity) and low-rate capacities, (which meant low power) which prevented pencil graphite from directly being used for lithium-ion batteries. Those disadvantages were traced back to non-graphite ingredients presented in the pencil graphite, in the form of clay and wax added during the pencil manufacturing process (17). To eliminate the irreversible reactions associated with

those ingredients in lithium-ion batteries and thereby improve coulombic efficiency, we first treated pencil graphite with lithium metal prelithiation via a Swagelok cell, followed by a full cell construction and testing with lithium iron phosphate ($LiFePO_4$) cathode (7, 18). As expected, prelithiated pencil graphite cells demonstrated comparable coulombic efficiency to the control group, but with better cycle life and discharge capacity retention. Those results suggest that even though pencil graphite may not be directly recycled for lithium-ion battery anode feedstock, it can still be engineered for battery materials by leveraging existing technologies such as prelithiation. This research further implies that material with hidden value can be explored and leveraged from new applications through rigorous scientific investigation.

RESULTS

Our analysis focused on the characteristics of the graphite, first charge capacity, first discharge capacity, first coulombic efficiency (FCE, a ratio of first discharge capacity to first charge capacity), rate capability, and cycle life. This was an assessment of the viability of pencil graphite as an anode material for lithium-ion batteries based on the data below. We carried out the testing with 10 batteries in each group for this study.

First, we tested the first charge capacity of the pencil and control graphite. We found that the pencil graphite had a significantly higher first charge capacity than the control graphite, 397.3 ± 32.9 mAh/g vs 208.5 ± 7.4 mAh/g, respectively ($p < 0.0001$, **Figure 1**). The control group first charge capacity (208.5 mAh/g) was much lower than the theoretical (ideal) value of 372 mAh/g.

Second, we measured the first discharge capacity of the pencil and control graphite. We noticed that the pencil graphite showed a higher first discharge capacity than the control graphite, 217.5 ± 24.5 mAh/g vs. 187.9 ± 6.8 mAh/g, respectively ($p=0.004$, **Figure 2**). We considered the first charge and first discharge capacity, respectively as key metrics to determine energy storage capability of batteries, and preferred higher values for higher capacity and better functioning battery devices. Both groups were much closer in value than the first charge capacity, though the mean difference between the two groups was significant. With 372
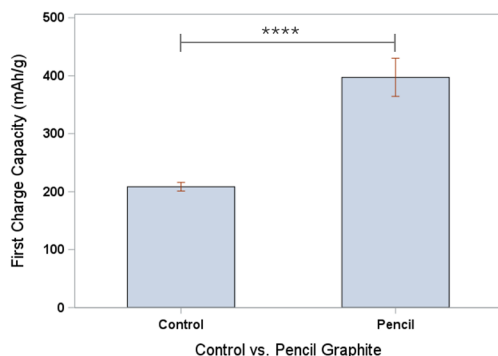

Figure 1. First Charge Capacity. Average first charge capacity for control and pencil graphite-based cell formation test conducted at WPI research lab. Error bars represent one standard deviation, ****p<0.0001. Pencil graphite is significant compared to the control graphite, two tailed t-test, 10 replicates for each group.

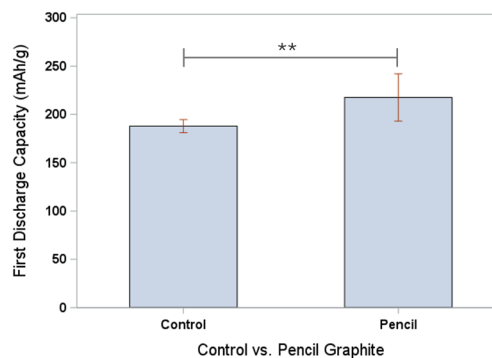**Figure 2. First Discharge Capacity.** Average first discharge capacity for control and pencil graphite-based cell formation test conducted at WPI research lab. Error bars represent one standard deviation, **p=0.004. Pencil graphite is significant compared to the control graphite, two tailed t-test, 10 replicates for each group.

mAh/g being the theoretical capacity of graphite, both groups failed to reach this number again.

We then compared the mean and standard deviation of the FCE for the pencil and control graphite groups and uncovered clear differences, 54.6 ± 1.8% vs. 89.8 ± 1.2%, respectively ($p<0.0001$, **Figure 3**). The control graphite did have a higher coulombic efficiency by a significant margin than pencil graphite. Since FCE was the ratio of first discharge capacity to first charge capacity, a much higher first charge capacity seen in pencil graphite led to lower FCE when its first discharge capacity was comparable to that of control graphite.

We also tested discharge rate capability to assess available battery capacity under various discharge rates. We found that the pencil group displayed similar discharge rate capability to the control group at 0.5C rate or less (**Figure 4**). The C rate is a measure of battery charge or discharge time, 1C means charging or discharging the battery for 1 hour, 2C means 0.5 hours and 0.5 C equals 2 hours, and so forth. This metric indicates accessible battery capacity depending on how fast the battery needs to be charged or discharged. We presented averaged values of the 10 cells from each group for the chart to provide clarity. When the C rate was equal to 0.1C, 0.2C, and 0.5C, the pencil graphite and control graphite performed about the same, average rate capability of 234.6 ± 17.2 mAh/g vs. 224.8 ± 11.5 mAh/g at 0.1C ($p=0.093$), 239.6 ± 17.1 mAh/g vs. 235.1 ± 14.0 mAh/g at 0.2C ($p=0.530$) and 241.3 ± 15.5 mAh/g vs. 254.4 ± 15.8 mAh/g at 0.5C ($p=0.078$), very comparable between the two groups. When the C rate was equal to 1C, the mean of the control graphite was significantly higher than the pencil graphite, 283.5 ± 15.1 mAh/g vs. 242.7 ± 16.0 mAh/g ($p<0.0001$). Also, with the C rate increasing, the pencil graphite increased only slightly. However, the control graphite increased correspondingly.

We further selected two cells from each group to conduct cycle life test, and we found that the pencil graphite demonstrated higher capacity retention than the control graphite (**Figure 5**). Due to the channel limits in the battery cycler, it was not possible to run cycle life test for all cells simultaneously. Therefore, two representative cells were chosen from each group to test and compare cell cycle life performance. In the first 50 cycles, there was an increase in capacity retention percentage for both groups which was an indication of electrolyte wetting, which is not uncommon in batteries (19). Electrolyte wetting was when the liquid electrolyte wetted the graphite and allowed for more graphite to participate in the lithium ion reactions which increased the capacity. However, this eventually peaked and began to plateau when all of the graphite became saturated. The capacity started to decrease around 50-100 cycles after plateauing because the batteries did not have 100% coulombic efficiency, and this meant a decrease in capacity over time. Looking at the capacity retention, at 100 cycles, the pencil graphite appeared to have around 85% retention compared to that of the control group around 43%. In the real world, this implied the pencil graphite batteries could last for longer because they had more capacity retention than the control group's overall cycles. The true mechanism behind this is yet to be determined. We believe this is important, because understanding what is influencing battery performance could be vital to the pencil graphite for battery applications.

We tested the mean difference in cell cycle life capacity retentions between the pencil graphite and control graphite. The mean of the pencil graphite (96.5%) was significantly higher than the mean of the control graphite (53.7%) ($p<0.0001$).

Next, we aimed to analyze the microstructure of both pencil and control graphite powders. We performed Scanning Electron Microscopy (SEM) and discovered that pencil and control graphite powder showed quite different features (**Figure 6**). Control graphite had defined flake-like shape and narrow particle size distribution with average particle size around 15 μm; by contrast, pencil graphite showed much broader particle size and irregular shape (**Figure 6**). We also performed a chemical composition analytical technique, energy dispersive X-ray spectroscopy (EDS) during the same SEM analytical experiment and confirmed elemental composition for pencil graphite was 69.7 wt% (weight percentage) in the form of carbon, whereas control graphite was 95.0 wt%. This data further highlighted the impurity difference between the control and pencil graphite.

In an experiment to improve the first coulombic efficiency, we treated pencil graphite with lithium metal, a process called prelithiation (18). We found that prelithiated pencil
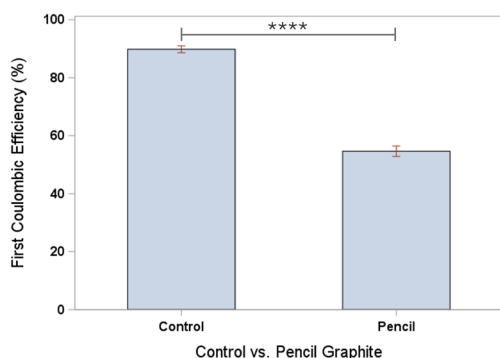
Figure 3. First Coulombic Efficiency. Average first coulombic efficiency for control and pencil graphite-based cell formation test conducted at WPI research lab. Error bars represent one standard deviation, ****$p<0.0001$. Pencil graphite is significant compared to the control graphite, two tailed t-test, 10 replicates for each group.
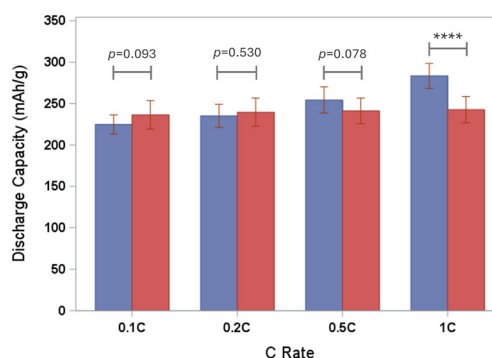
Figure 4. Discharge Rate Capability. Average discharge rate capability for control (blue) and pencil (red) graphite-based cell formation test conducted at WPI research lab. Error bars represent one standard deviation, ****$p<0.0001$, two tailed t-test, 10 replicates for each group.
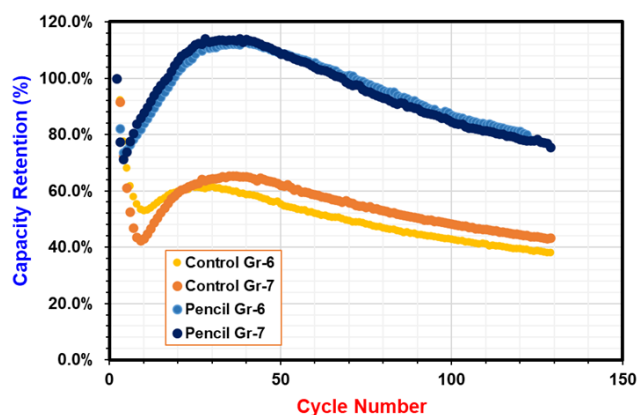
Figure 5. Cycle life test for control and pencil graphite-based cells. Cycle life comparison test conducted at WPI research lab. Cells 6 and 7 from each group were selected for the cycle life test, capacity retention was calculated based on the ratio of a given cycle to the first cycle capacity. The blue colors represented the pencil groups, while the yellow and orange indicated values for the two control groups.

graphite not only exhibited much improved coulombic efficiency (>99.6%), but long-term cycle life was also better than control graphite (**Figure 7**). In a voltage range of 3.6 to 2.0V, control graphite cell displayed relatively faster capacity fading than the prelithiated pencil graphite cell at C/2 charge and discharge rate. In the first 30 cycles, the control cell was better, but beyond that capacity retention was worse than prelithiated pencil graphite with an increasing gap up to 100 cycles. This preliminary result demonstrated that pencil graphite with proper treatment such as prelithiation could be a possible alternative source for lithium-ion batteries.

DISCUSSION

Pencil graphite exhibits a much higher first charge capacity than control graphite, but the first discharge capacity of both groups is similar resulting in quite different first coulombic efficiency for both groups, which could be due to excessive side reactions with lithium (7, 12). These side reactions would be irreversible and related to other non-graphite impurities, the effects of which could be seen in the first discharge capacity. From the rate performance perspective, pencil graphite and control graphite demonstrate similar rate capability, a measure of battery charge release/storage rate (or power performance). In the long term cyclability category, pencil graphite appears to cycle better than control graphite in the room temperature cycle life test. At the material microstructural level, pencil graphite and control graphite display very different particle sizes and shapes, resulting in different electrochemical performances. We can also relate material composition difference to aforementioned significant discrepancy in FCE. According to previous literature, 2B pencil graphite was 74% graphite, 20% clay, and 5% wax (17). This composition is reasonably close to the 69.7 wt% graphite observed in this work. Only graphite could react with the lithium ion reversibly through the intercalation mechanism whereas clay and wax contributed to parasitic reasons, resulting in low first coulombic efficiency (12). As such, pencil graphite (2B grade) used in this study cannot be directly used for battery anode due to other components, clay, and wax.
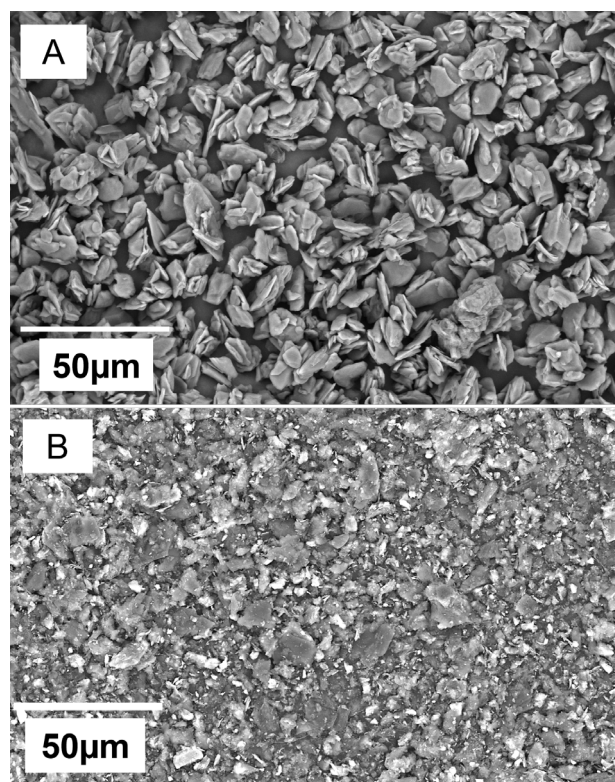

Figure 6. SEM micrographs for control and pencil graphite powders. Microstructure analysis conducted by SEM. A) Control and B) pencil graphite. Both images were taken at a magnification of 2500x, microbar represents 50μm.

However, pencil graphite can perform better than industrial control with prelithiation treatment, making it a viable source for lithium-ion battery anode material.

A few conclusions can be drawn from the data collected and analyzed with potential limitations due to small sample size (10 cells each). This shows that pencil graphite discharges a similar amount of energy even when given 1 or 10 hours. Comparing this to control, we see that control is steadier and still increases in DRC (discharge rate capacity) over time when the pencil group remains stagnant. Simply put, this means that the control group is more efficient and that it can be more efficient with energy. As is, when graphite is collected from pencils without purification or proper treatment such as prelithiation, it will perform worse in terms of coulombic efficiency, as it is less capable of being effective long-term than the control graphite as shown in this study.

Prelithiation of pencil graphite was tested in order to improve the coulombic efficiency. The principle behind prelitation is that parasitic reactions stem from clay and wax in the pencil graphite is largely responsible for poor FCE, and those irreversible reactions could be remedied through electrochemical treatment with Lithium, also known as prelithiation, a commercially viable technique being widely studied to improve anode materials with low FCE, such as silicon and silicon oxide based anode materials (20). The prelithiated pencil graphite reported in this work exhibited not only much improved coulombic efficiency, but also superior cycle life to control graphite, supporting the hypothesis set forth in this research.
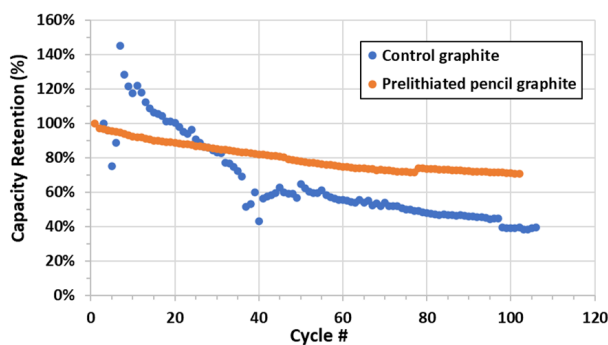
Figure 7. Cycle life test of control and prelithiated pencil graphite. Cycle life comparison test with Swagelok cells conducted at Shanghai Jiaotong University research lab (LiFePO$_4$ cathode, 3.6-2.0V, +C/2, -C/2, 23°C). Capacity retention was calculated based on the ratio of a given cycle to the first cycle capacity.

Another approach could be to purify the pencil graphite and remove the other materials (clay and wax) from it. This would likely make pencil graphite comparable to the control graphite, as shown in the first discharge capacity and rate capability results. Both groups are comparable in these categories, which means that the graphite portions of both groups are very similar to each other. This is because the lithium-ion chemical interaction with graphite is reversible and highly efficient (3, 7, 11, 12).

Overall, our results show that pencil graphite may not be directly incorporated as a component for lithium-ion batteries. However, we demonstrate that it can become a viable alternative through the prelithiation treatment, thereby providing additional solution to support future growth of the lithium-ion battery industry.

MATERIALS AND METHODS
Anode materials
A significant amount of pencil graphite was prepared by cracking wooden shell of type 2B pencil stubs to recover graphite cores, which were further hand crushed with a mortar and pestle to obtain fine powder, as a graphite containing water-based ink will be needed at future steps. After sieving with a 200-mesh screen (Stainless steel, 75 µm, Fisher Scientific, catalog# No.200), coarse particles retained were further crushed until they went through the screen. Control graphite under the trade name A1 graphite, along with LiFePO$_4$ cathode (trade name S20) were obtained from BTR new energy materials Co. Ltd in Shenzhen, China and used as received.

Anode electrode preparation
Water-based anode slurries containing 90 wt% graphite powder were prepared via a THINKY mixer (Model ARE-310, THINKY Co, Japan); other ingredients in the slurry included 5 wt% binder (2.5 wt% carboxymethyl cellulose (CMC), MTI Co, catalog#Lib-CMC, 2.5% styrene-butadiene rubber (SBR), MTI, catalog #Lib-SBR,) which was used to hold the active materials graphite and the current collector copper foil together, and 5 wt% carbon black (Imerys S.A. trade name Super P® Li), which was a conductive additive used to make the anode more conductive so there was less resistance (21, 22). This was mixed with deionized water and turned into a paste with 45 wt% solids loading. The paste was

put onto a 9 µm thick copper foil (10 cm long, 6 cm wide, MTI Co.) using a thin film applicator (MTI Co. catalog # MSK-AFA-III 110), followed by drying to remove the liquid solvent and calendering to achieve target electrode thickness and porosity. This was where the desired circular shape could be punched out for future coin and Swagelok cell assembly when combined with the other cell components.

Cathode electrode preparation
N-Methyl-2-pyrrolidone (NMP, MTI Co, catalog# Lib-CHNO) solvent based cathode slurries containing 92 wt% LiFePO$_4$ powder, 5 wt% PVdF binder (polyvinylidene fluoride, MTI Co, catalog# Lib-PVDF) and 3 wt% carbon black super P® Li were also prepared via the THINKY mixer and coated on a 15 µm thick aluminum foil (10 cm long, 6 cm wide, MTI Co.) to obtain cathode electrode, a process similar to the anode slurries (21, 22).

Cell assembly
Coin cells were fabricated using CR2032 parts (MTI Co, catalog# EQ-CR2032-CASE), 16 µm polypropylene separator (Celgard Co, catalog# H1609), 0.6 mm thick Li metal counter electrode (MTI Co, catalog# EQ-Lib-LiC) and above-mentioned anode electrodes. For statistical comparison, 10 cells with pencil graphite anode and 10 cells with control graphite were prepared and tested. A standard Lithium-ion battery electrolyte with 1.0 M LiPF$_6$, EC/EMC (70:30) plus 1%VC was used for all cells (MTI Co, catalog# LBC3015B-LD)

Cell testing
A 48 channel battery tester (Wuhan LAND Electronic Co. Ltd, Wuhan, China) was used to form the cells, the formation procedure was as follows: charge and discharge the cell at a current rate of 0.1C (C rate is a measure of battery charge or discharge time, 1C means charging or discharging the battery for 1 hour, 2C means 0.5 hours and 0.5 C equals 2 hours, and so forth), at a voltage range of 0.05V to 1.5V, rest for 5 minutes after each step and repeat the charge/discharge cycle 3 time to complete cell formation. Rate capability test was conducted by charging the cell at 0.2C rate, rest for 5 minutes, and discharging are at 0.2C, 0.5C, 1C, and 2C, respectively. Post rate testing cells were also cycle life tested at 0.5C rate until capacity retention was below 80% (with respect to initial cell capacity). The voltage range for rate and cycle life tests was set between 0.05V and 1.5V.

Cell prelithiation
Swagelok cells with pencil graphite anode were assembled following previous literature with Lithium metal counter electrode and 16 µm polypropylene separator, and activated with the same electrolyte from MTI Co (23). After completing the first formation cycle at 0.1C rate, cells were disassembled to recover the pencil graphite anode and paired with LiFePO$_4$ cathode to construct new Swagelok cells using the same assembly process. A control group was also built using graphite and LiFePO$_4$ but without anode prelithiation. Both groups were formed at 0.1C charge and discharge rate for three cycles in a voltage range of 3.6V to 2.0V. Cycle life test for both control group and prelithiated group was conducted at 0.5C rate, from 3.6V to 2.0V at room temperature (23°C).

Materials characterization

A scanning electron microscope (Phenom Pro desktop SEM, Nanoscience Instruments, Inc.) was employed to perform microstructure analyses for graphite powders; elemental mapping of those samples was carried out with energy dispersive X-ray spectroscopy associated with the Phenom Pro desktop SEM. Each SEM specimen was prepared by carefully placing a thin layer of graphite powder on a standard SEM stub (Ted Pella, Inc., product# 16111). To eliminate charging on non-conductive sample, each specimen was vacuum deposited with gold before measurement (24).

Data analysis

A two tailed t-test was applied to compare the means of first charge capacity, first discharge capacity, first coulombic efficiency, discharge rate capability and cycle life of the two groups (the BTR control group against the pencil graphite group). A p-value of 0.05 or less was used as the predetermined criterion for statistical significance. The Equality of Variances test was used to check the variance of the two groups. The p-value of the Equality of Variances test determined if the two-sample t-test with equal variance or the two-sample t-test with unequal variance would be used for the analysis.

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