# Improving the electrical performance of Lithium-ion battery using SilicaCarbon anode through technique

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#### Abstract

Nowadays, a hybrid composite SiO2/C has been paid attention to improving battery performance in Li-ion batteries (LIBs) as the anode. However, this material unexpectedly suffers from initial active lithium loss caused by the solid electrolyte interface (SEI) formation leading to low initial Coulombic efficiency and significantly reducing the initial capacity. In order to solve these issues, pre-lithiation has been considered an effective approach to limit active lithium loss and increase cycling performance. This work focuses on the two most common techniques, including the direct contact method (CM) and the electrochemical method in half-cell (EM). After the pre-lithiation process, the anodes would be evaluated in full-cell with LiNi0.6Mn0.2Co0.2O2 (NMC622) cathode. According to electrochemical properties evaluations, pre-lithiation could enhance discharged capacity and initial coulombic efficiency. Without the pre-lithiation method, the discharged capacity in full-cell only witnessed 66.9 mAh.g-1, while CM and EM methods illustrated a better battery performance. In detail, EM exhibited a higher discharged capacity and initial coulombic efficiency (137.06 mAh.g-1 and 99.08%, respectively) compared to CM (99.08 mAh.g-1 and 93.23%) method. Besides, the capacity retention using EM achieved 71.4% and the discharged capacity illustrated 97.87 mAh.g-1 after 100 cycles, which is better than using CM, which only showed 71.40 mAh.g-1.

#### 1. Introduction

Nowadays, the depletion shortage of non-renewable natural fossil resources and environmental damages has led to the development of battery storage energy. Therein, energy storage through electrochemical cells has been considered the most satisfactory solution due to its high energy density, high power density, flexibility, and simplicity of maintenance. Moreover, as shown as the state-of-the-art technology for various applications, LIBs are required to show lightweight, long lifespans, high specific capacity, and good retention capacity. [1-4]

In recent years, the silicon-based anode has attracted more attention because of its superior theoretical capacity of 3579 mAh.g<sup>-1</sup>. [5] However, the formation of  $\text{Li}_x \text{Si}_y$  alloy makes the silicon anode suffers a high-volume expansion (over 280%), which leads to concerns related to the safety and disconnection of electric contact, and then a severe decrease of capacity after the first cycles. Therefore, silica materials have been alternatives to develop silicon-based anodes owing to less volume expansion and high capacity of 635.7 mAh.g<sup>-1</sup> for LIBs. [6-7]

Although those materials overcome the weakness of silicon anode, their low conductivity is one of the main reasons to prevent commercialization. As a solution, a hybrid composite  $SiO_2/C$  was used to enhance battery performance. In detail, the hard carbon in the  $SiO_2$  structure enhances the ionic and electronic conduction and the kinetic migration of Li<sup>+</sup> inside the materials by creating a linking network to boost electrons movements to convert  $SiO_2$  into Si and co-products faster. [8-10] Nevertheless,  $SiO_2/C$  based as an anode suffers from significant active lithium loss, which leads to low initial Coulombic efficiency in Liion batteries. This phenomenon is caused by some  $Li^+$  ions trapped in an anode structure. In detail, the irreversible reaction and volume expansion create cracks inside the electrode, which need a large amount of the electrolyte. As a result, the thickness of the SEI layer increases after the first cycle, causing a significant amount of  $Li^+$  ions to be trapped inside the electrode by the SEI layer. [11-12]

To deal with this challenge, the pre-lithiation process was used to compensate for the active lithium loss of the anode to enhance the practical battery performance. [13-17] In this research, the two common prelithiation methods were used, which include the direct contact method (CM) using lithium foil and the halfcell electrochemical method (EM). [18-22] These techniques were applied for the composite anode  $SiO_2/C$ , before matching in the full-cell with NMC622, cathode. In CM, the time for anode directly contacted with lithium was investigated. Meanwhile, regarding the electrochemical method, the anode was assembled in half-cell and pre-lithiated at various cycles to activate prior to the full-cell assembly. This study hopefully brings new insights into selecting an appropriate approach to successfully construct a high-performance lithium full-cell with silica/carbon-based anode.

## 2. Experiment

#### 2.1. Experimental samples

Table 1. List of full cells, detailed anodes component and techniques used for pre-lithiation.

Full cell	Anode	Cathode	Method
100GR	100 wt.% graphite: 1.5 wt.% CMC: 2.5 wt.% SBR	NMC622	Without pre-lithiation method
Baseline	80 wt.% graphite: 20 wt.% $SiO_2/C$ : 1.5 wt.% CMC: 2.5 wt.% SBR	NMC622	Without pre-lithiation method
CM10		NMC622	CM method within 10 min
CM20		NMC622	CM method within 20 min
CM30		NMC622	CM method within 30 min
CM40		NMC622	CM method within 40 min
EM1C		NMC622	EM method with one cycle
EM2C		NMC622	EM method with two cycles
EM3C		NMC622	EM method with three cycles
EM4C		NMC622	EM method with four cycles

In this work, ten different samples were investigated for pre-lithiation techniques, namely 100GR, baseline, CM10, CM20, CM30, CM40, EM1C, EM2C, EM3C, and EM4C, respectively (**Table 1**). Among them, 100GR was represented for full-cell consisting of 100 wt.% graphite: 1.5 wt.% CMC: 2.5 wt.% SBR as anode and NMC622 as cathode. The other samples included 80 wt.% graphite: 20 wt.% SiO<sub>2</sub>/C: 1 wt.% CG5: 1.5 wt.% CMC: 2.5 wt.% SBR as anode and NMC622 as cathode. In detail, the 100GR and baseline were named full-cells without prelithiated anode. In contrast, the CM samples mean the anode using the direct contact method. For instance, "CM10" indicates that full-cell was assembled using the electrochemical method, whereas the anode was assembled in half-cell and cycled in several cycles. For example, "EM1C" indicates that the full-cell was assembled by the anode using an electrochemical technique and activated by one cycle.

## 2.2. Electrode preparation

The anode was prepared by mixing conductive carbon (C65, Imerys, Belgium), synthesized silica-carbon composite  $(SiO_2/C)$  [23,24], and carboxymethyl cellulose binder (CMC, MTI, USA). After being mixed in a planetary mixer (Thinky, Japan) at 2000 rpm for 20 minutes, an amount of graphite was added into a mixture and continuously mixed for the next 20 minutes. Then, SBR binder was incorporated and mixed for the last 5 minutes. The homogeneous slurry with the aforementioned ratio was cast on copper foil and then

dried in a vacuum oven at 80 °C for 12 h. The electrode film was finally punched into a 15 mm diameter round plate. The mass loading of the anode was around 12 mg.cm<sup>-2</sup>.

The positive electrode was fabricated by mixing an active material of  $LiNi_{0.6}Mn_{0.2}Co_{0.2}O_2(NMC622, MTI, USA)$ , C65, polyvinylidene fluoride (PVdF, Sigma-Aldrich, USA) in the weight ratio of 96:2:2, respectively. All powder materials were dispersed in N-methylpyrrolidone (NMP, Merck) solvent in a container. After mixing for 20 minutes, the homogeneous slurry was cast on an aluminum foil and dried in the same condition as the anode. Then, the cathode film was punched into a round shape (12 mm diameter). The mass loading of the cathode was around 18 mg.cm<sup>-2</sup>.

# 2.3. Coin cell assembly process

In the CM method, the anodes were brought face to face with Li foil for a period, then taken out and assembled in the full-cell configuration with the NMC622 cathodes. On the other hand, in EM treatment, the anodes were pre-lithiated by half-cell with lithium metal as counter electrode using the electrolyte of 1.2 M LiPF<sub>6</sub> dissolved in EC: EMC (3:7, v/v) and a polypropylene separator (PP, Sigma-Aldrich) to achieve the pre-lithiation process. Then, these anodes were disassembled from half-cell and used again for matching in full-cell. All the steps were carried out in an Ar-controlled glovebox (MBRAUN, Germany). After the pre-lithiation process, these anodes were assembled in full-cell with NMC622 cathode material.

#### 2.4. Testing model

In the EM method, the anodes assembled in half-cells were tested to activate by Galvanostatic Cycling with Potential Limitation (GCPL), performed on CT2001A Lanhe Battery Testing System (China). In detail, the activated test was evaluated at current density C/10 ( $1C = 400 \text{ mA.g}^{-1}$ ) from 0.01 to 2.0 V. In full-cell configuration, the cells were cycled at a constant current density of C/3 ( $1C = 150 \text{ mA.g}^{-1}$ ) at 30 °C with a cut-off voltage range of 2.65 – 4.3 V using a battery testing system Lanhe.

## 3. Results and discussion





Figure 1. XRD patterns of  $SiO_2/C$  composite material prepared from rice husks.

As shown in **Figure 1**, the structure of silica carbon material prepared from rice husk as raw material was characterized by X-ray diffraction. The synthesis process of  $SiO_2/C$  anode material was reported elsewhere. [23] XRD patterns of silica carbon material exhibited broad peak spreading from 20 – 27° confirming the presence of amorphous silica and carbon. In addition, the diffraction peak of 43 – 44° is represented for the amorphous carbon. [9] As a result,  $SiO_2/C$  synthesized has no impurities which are compatible for anode application.

## 3.2. Electrochemical properties



**Figure 2.** (a) Voltage profile at first cycle, (b) Voltage profile at 80th cycle, (c) Cycling performance, and (d) Coulombic efficiency of GR100 and baseline samples with NMC622 full-cells.

The battery performance of 100GR and the baseline with NMC622 full-cells are shown in **Figure 2**. The 100GR full cell exhibits an initial discharge capacity of 140.27 mAh.g<sup>-1</sup>, while the baseline only displays an initial value of 48.16 mAh.g<sup>-1</sup> (**Figure 2** a). After 80 cycles, the 100GR full cell delivered a specific capacity of 51.35 mAh.g<sup>-1</sup> (**Figure 2** b), which was reduced by 63.4% compared to the first cycle. Meanwhile, the baseline one displays a capacity of 32.50 mAh.g<sup>-1</sup>, which only decreased by 32.9% compared to the first cycle. These results could be explained by the porous and amorphous structure of SiO<sub>2</sub>/C facilitated a considerable amount of Li<sup>+</sup> ions from the NMC cathode to intercalate in by shortening the Li<sup>+</sup> ion diffusion pathway, resulting in the significant charging capacity. [10] However, the superior specific surface area of SiO<sub>2</sub>/C material reacted strongly with electrolyte to consume Li<sup>+</sup> ions for SEI layer formation, which indicated "active lithium loss". Hence the discharged capacity of the baseline is lower than the 100GR.

Furthermore, the cycling performance and Coulombic efficiency of the baseline were more stable than 100GR (**Figure 2** c, d). Although the 100GR sample possesses a much higher discharged capacity than the baseline sample, capacity retention is lower (**Figure 2** c). Minh et al. reported that the 100GR||NMC622 full-

cell using the same electrolyte solution (1.2 LiPF<sub>6</sub>, EC: EMC 3:7 v/v) with N/P ratio of 1.24 delivered a discharged capacity of only 140.5 mAh.g<sup>-1</sup> and remained 9.8 % after 30 cycles, respectively. [25] Moreover, the previous study reported that the well-designed graphite||NMC622 batteries retained about 50% initial capacity during the cell lifespan. [14] This rapid capacity fading of graphite anode full-cell may associate with active lithium loss due to the growth of the SEI layer or physical delamination of the graphite particles. [26] Aiping Wang et al. showed that the decomposition of ethylene carbonate (EC) during the cycling process would thicken the SEI layer and consequently decrease capacity retention. [14] In order to enhance the long-cycling performance of the baseline sample, the CM and EM methods were carried out to optimize the full-cell capacity.

Regarding the pre-lithiation  $SiO_2/C$  using the direct contact method (CM) with lithium foil for a defined time, it can be expected that an adjustable amount of Li<sup>+</sup> can be used for compensating active lithium loss during the first cycles for SiO<sub>2</sub>-based anode without promoting the Li dendrite formation. Figure 3 shows the battery performance in various contact times, including 10, 20, 30, and 40 minutes. The CM30 sample shows the highest capacity, while the baseline sample only displays the lowest capacity in the first and 80<sup>th</sup> cycles (Figure 3 a, b). The first reversible capacity of CM30 is 99.08 mAh.g<sup>-1</sup>, which declines by 27.94% after 80 cycles, while these values for the baseline sample are 48.43 mAh.g<sup>-1</sup> and 32.50 mAh.g<sup>-1</sup>, respectively. According to cycling performance and Coulombic efficiency (Figure 3 c, d), the CM30 sample continually witnesses the highest capacity during the testing time and maintains stable Coulombic efficiency of around 98% for 80 cycles. Furthermore, increasing the contact time enhances not only the discharged capacity but also improves the coulombic efficiency. Indeed, by increasing the contact time, the initial Coulombic efficiency increases from 84.42% (CM10 sample) to the highest of 93.23 % for the CM30 before declining by the time in CM40. Recently, similar results were demonstrated by Gerrit Michael Overhoff et al. with Si/C||NMC811 full-cell by varying the contact times. [21] As a result, the amount of Li<sup>+</sup> ion was compensated to enhance battery performance by the CM method. Nevertheless, increasing the contacting time (over 30 minutes) leading to the deposition of lithium metal on the surface of the anode as well as the thickness of the SEI layer are the main reasons to explain the fast decrease of performance in the CM40 sample.



**Figure 3.** (a) Voltage profile at first cycle, (b) Voltage profile at 80<sup>th</sup> cycle, (c) Cycling performance, and (d) Coulombic efficiency of baseline, CM10, CM20, CM30, and CM40 samples with NMC622 full-cells.

FT-IR Spectroscopy was further performed to evaluate the formation of the SEI layer on the anode surface by analyzing the vibrational state of chemical bonds. **Figure 4** exhibits similar qualitative spectra but differentiates quantitatively in peak intensity, indicating the heterogenous formation of the SEI layer. In general, there are three main frequency regions observed, and each of these regions is associated with typical group frequencies:  $4000 - 3000 \text{ cm}^{-1}$  assigned for the O – H stretching,  $3000 - 1750 \text{ cm}^{-1}$  for the C – H stretching, and  $1700 - 800 \text{ cm}^{-1}$  for the C = O stretching regions, as earlier reported by Guorong V. Zhuang et al. [27] The wavenumber region of  $2800 - 2000 \text{ cm}^{-1}$  might represent the CO<sub>2</sub> molecular absorbed by LiOH or Li<sub>2</sub>O, which happens during sample reservation and testing process (**Figure 4**). In addition, the SEI layer was reported to be composed of Li<sub>2</sub>CO<sub>3</sub> and lithium alkyl carbonate (ROCO<sub>2</sub>Li) compounds due to the decomposition of ethyl carbonate (EC) electrolyte. [28] Thereby, IR fingerprints of these samples proved the present of Li<sub>2</sub>CO<sub>3</sub> on the electrode surface by the broad band of 920 – 800 cm<sup>-1</sup> and the double peak at 1520 – 1380 cm<sup>-1</sup> wavelength. It can be observed that the peak intensity of Li<sub>2</sub>CO<sub>3</sub> also increases with the longer contact duration, which is consistent with the explanation of the thick SEI layer. Additionally, three wavelength regions including 1820 – 1740 cm<sup>-1</sup>, 1100 – 1000 cm<sup>-1</sup>, and 780 – 600 cm<sup>-1</sup> represent organic carbonate from ROCO<sub>2</sub>Li compounds was observed and also increased with the contact time. [28]



Figure 4. FT-IR of pre-lithiated samples in different contact times with CM method.



**Figure 5**. (a) Voltage profile at first cycle, (b) Voltage profile at 80<sup>th</sup> cycle, (c) Cycling performance, and (d) Coulombic efficiency of baseline, EM1C, EM2C, EM3C, and EM4C samples with NMC622 full-cells.

Figure 5 shows a significant improvement in electrochemical performance by the EM method. Specifically, the three samples presented significantly higher capacity than the baseline sample, and the capacity decreased following the order: EM4C < EM2C < EM3C, respectively. Obviously, the sample EM4C delivers the highest initial capacity of 137.06 mAh.g<sup>-1</sup> (Figure 5 a) and maintains 97.87 mAh.g<sup>-1</sup> in the 80<sup>th</sup>cycle (Figure 5 b). Moreover, the EM4C continually illustrates stable coulombic efficiency, around 99% for 80 cycles. Meanwhile, regarding the sample EM2C and EM3C, there is no significant difference in the capacity behavior, showing a low initial value of 48.43 mAh.g<sup>-1</sup>, and the capacity declines to 32.50 mAh.g<sup>-1</sup> after 80 cycles (Figure 5 d). By activating anodes with the EM method, an artificial SEI layer is formed on anode's surface and becomes a stable SEI layer. Indeed, the full-cells using these anode pre-lithiated by EM method do not lose the capacity to stabilize the SEI layer. Furthermore, the EM method can enhance the mobility of Li<sup>+</sup> ions to optimize the lithiation mechanism, which makes the charge transfer process more accessible since the Li<sup>+</sup> ions are not trapped. [29-32] Therefore, the initial Coulombic efficiency and battery performance increase when increasing the cycles for pre-lithiation.

Table 2. Battery performance of 100GR, baseline, CM30, and EM4C sa	ample
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	Discharge capacity in the $1^{\rm st}$ cycle (mAh.g^-1)	Discharge capacity in the $80^{\rm th}$ cycle (mAh.g^-1)	Capacity retent
100GR	140.27	51.35	36.6
Baseline	48.43	32.50	67.1
CM30	99.08	71.40	72.1
EM4C	137.06	97.87	71.4



Figure 6. (a) Cycling performance and (b) Energy density in the 80<sup>th</sup> cycle.

The battery performance of four samples, including 100GR, baseline, CM30, and EM4C (**Table 2**) is shown in **Figure 6**. Overall, the discharged capacity of all samples decreases after testing time. Indeed, the 100GR sample drops dramatically in discharge capacity, from 140.27 to 51.35 mAh.g<sup>-1</sup> (**Figure 6** a). Meanwhile, the battery performance increase when using the pre-lithiation method. In detail, the CM30 and EM4C show the capacity of 99.08 and 137.06 mAh.g<sup>-1</sup> in the first cycle, respectively, which is increased around 2-3 times compared to the baseline sample. Therefore, the energy density of the EM4C is much higher than the others, which is 98.68 mWh.g<sup>-1</sup>. As a result, the EM method effectively enhanced the battery performance.

# 4. Conclusion

In conclusion, the pre-lithiation method could effectively enhance the battery performance compared to CM. In particular, the EM4C sample shows the highest battery performance with the discharged capacity of 137.06 and 97.87 mAh.g<sup>-1</sup> in the first and 80<sup>th</sup> cycle, while the CM30 demonstrates a capacity of 71.40 mAh.g<sup>-1</sup> in the 80<sup>th</sup> cycle, respectively. In addition, the EM4C exhibits remarkable cycling performance, with a retention capacity of 71.4% after 80 cycles at a C/3 current density compared to the other samples. The remarkable improvement in the performance of SiO<sub>2</sub>/C||NMC622 full-cell using the EM technique can be attributed to a stable artificial SEI layer on the anode surface which enhance the mobility of Li<sup>+</sup> ions and remained enough Li<sup>+</sup> ions stored capacity for stabilizing the SEI layer. Therefore, the EM is a promising pre-lithiation method for the silicon or silicon-based anode material to enhance battery performance in LIB applications.

### Author contributions

Xuan My Nguyen Thi: performed the analysis, collected the data, wrote the paper. Kha Minh Le: conceived and designed the analysis, data interpretation, edited the paper. Quan Phung: contributed to analysis tool. Duc Quang Truong: contributed to analysis tool. Hoang Van Nguyen: contributed to analysis tool, edit the paper. Quynh Nhu Nguyen: data interpretation, wrote, reviewed and format the paper. Man Van Tran: contributed to analysis tool. Phung My Loan Le: conceived and designed the analysis, reviewed the paper.

#### **Conflicts of interest**

There are no conflicts to declare.

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