



Ultrasound-assisted synthesis of sodium powder as electrode additive to improve cycling performance of sodium-ion batteries



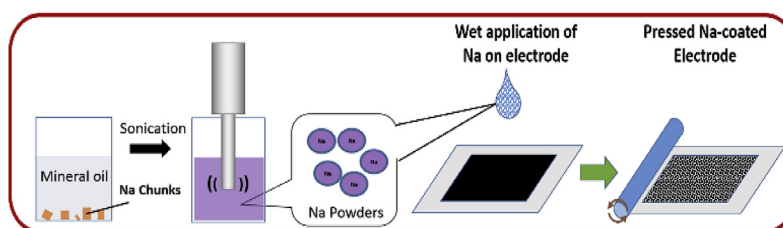
Jialiang Tang, Daniel Kyungbin Kye, Vilas G. Pol*

Davidson School of Chemical Engineering, Purdue University, West Lafayette, IN, 47907, United States

HIGHLIGHTS

- Sodium powder is prepared by ultrasonication of bulk sodium metal in inert solvent.
- Sodium powder in hexane is easily applied onto electrodes as pre-sodiation agent.
- Improved cycling in both half- and full-cells is observed in pre-sodiated cells.
- Reactivity of sodium powder to air and water remains a challenge.

GRAPHICAL ABSTRACT



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ABSTRACT

Excessive solid electrolyte interphase (SEI) buildup in the formation cycles or later cycles consumes electrolyte, depletes alkaline ion availability, and increases cell polarization; the reduction in the amount of available alkaline ions upon cycling often causes low capacity and poor capacity retention in full cells. To compensate the Na loss from SEI formation, we have developed a sodium-powder based pre-sodiation technique that could potentially be applied to both anode and cathode materials with minimal modification to conventional battery making process. The synthesis of sodium powder is accomplished via ultrasonic dispersion of molten sodium metal in mineral oil. When suspended in hexane, the sodium powder can be easily applied onto electrodes as pre-sodiation additive. In the half cell study with glucose derived carbon (GC1100), pre-sodiation decreases the initial open circuit cell potential (~ 1 V drop) and reduces the first cycle irreversible Coulombic efficiency (from 19.3% to 8%). In the full cell study with GC1100 and NaCrO_2 , pre-sodiation leads to $\sim 10\%$ improvement in cycling capacity and $\sim 5\%$ increase in energy density. Decreased cell polarization is also observed in pre-sodiated cells.

1. Introduction

Battery researchers around the world have been developing sodium-ion batteries (SIBs) as an alternative technology to rechargeable lithium-ion batteries (LIBs). SIBs could potentially cost less than LIBs and be produced in larger scale for grid energy storage, owing to the natural abundance of sodium resources [1,2]. The analogous electrochemistry of SIBs to LIBs also enabled the rapid development of many Na-ion cathodes (e.g., Na-based layered metal oxides and polyanionic

compounds) [3], anodes (e.g., tin-based alloying materials and hard carbon) [4,5], and electrolyte systems (e.g., NaClO_4 and NaPF_6 salt in combination of linear and cyclic carbonate solvent) [1,2,6].

Similar to LIBs, excessive and uncontrolled solid electrolyte interphase (SEI) growth on the anode surface remains a major challenge to SIBs. SEI formation results from the unavoidable decomposition of electrolyte at lower potential during initial cycles (< 1 V) [7,8]. Excessive SEI buildup throughout cycling consumes electrolyte, depletes available alkaline ions, and increases cell polarization [9]. Non-SEI

* Corresponding author.

E-mail address: vpol@purdue.edu (V.G. Pol).

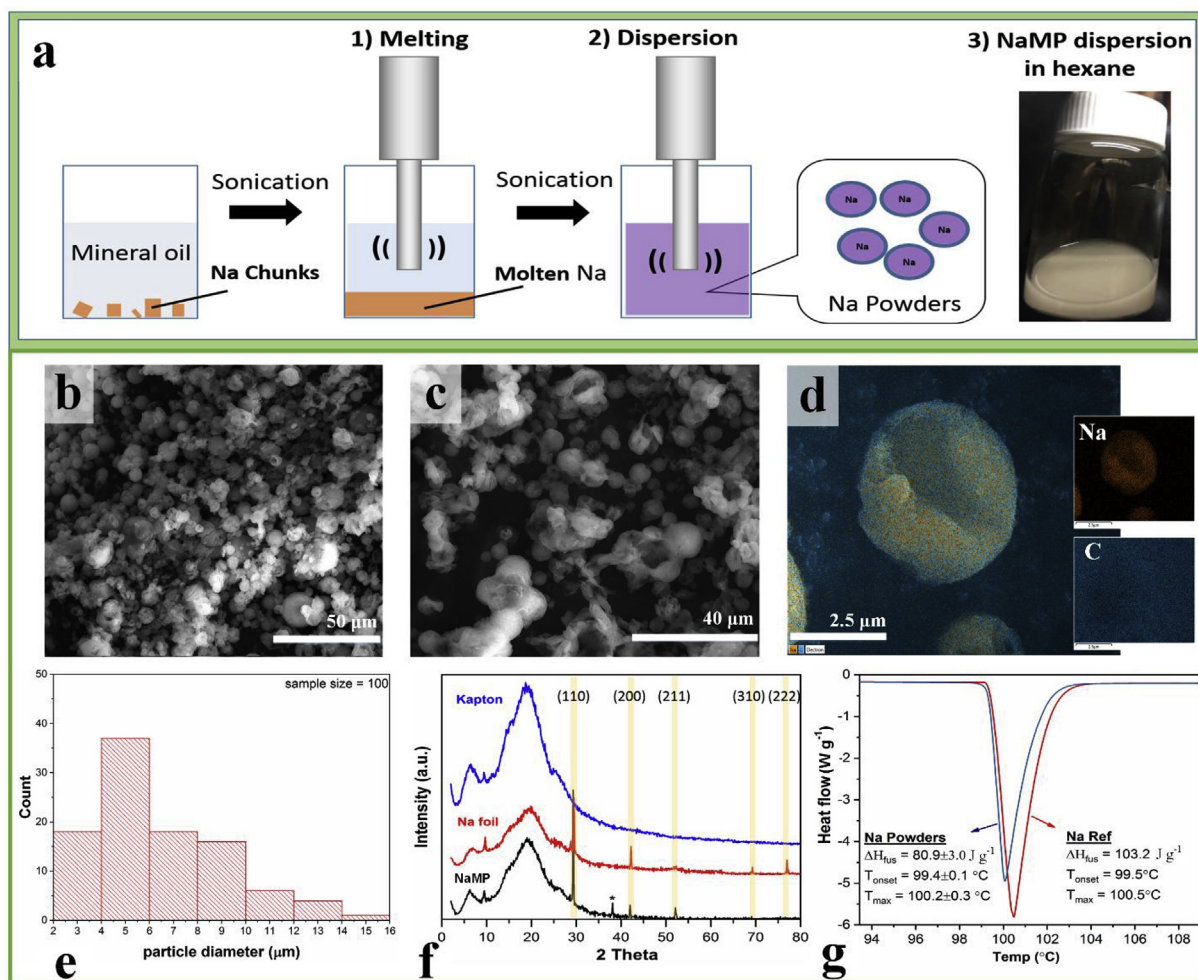


Fig. 1. Synthesis and characterization of sodium powder. a) Pulsed ultrasonic melting and dispersion of sodium metal in mineral oil, and actual photo of sodium powder stored in hexane after removal of mineral oil. b-c) SEM images of sodium powder at different scales; d) EDX mapping of a single sodium powder; e) corresponding particle size distribution of NaMP; f) XRD patterns of Kapton tape, Na foil, and NaMP; g) DSC analysis of NaMP and Na reference.

related capacity losses due to irreversible adsorption of Na at graphene defect sites and irreversible intercalation between graphene layers also contributes to low Coulombic efficiency in the first few cycles [10]. In our recent works exploring various carbon anode materials for SIBs, the first cycle Coulombic inefficiencies are 50% for scalable carbon sheets [11], 25.1%–41.6% for carbon particles [12], 60.3% for interconnected carbon network comprising electronegative fluorine [13], 32.7% for microporous carbon [14], and 38.0% for solid dense carbon spheres [15]. Moreover, many review articles have described high-capacity anode and cathode materials that suffer from poor first cycle Coulombic efficiencies and continuous capacity fade [2,6,16–20]. This indicates the genuine need for developing anode pretreatment processes to enhance reversible capacities in SIBs for their large scale adaptation.

These issues with SEI growth are exasperated in full cells where cathodes contain a limited amount of alkaline ions that are available for reversible cycling. The reduction in the amount of available alkaline ions upon cycling often results in low capacity and poor capacity retention in full cells as compared to half-cell performance [6]. Aurbach et al. recently highlighted the importance of anodic pretreatment (via electrochemical pre-sodiation/pre-lithiation to supply additional alkaline ions) to enhance reversible capacities in SIB full cells (hard carbon// $\text{NaNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$) and LIB full cells (hard carbon// $\text{LiNi}_{0.5}\text{Mn}_{0.5}\text{O}_2$) [21].

In light of these issues associated with SEI growth, several non-electrochemical pretreatment techniques of anodes have been

developed. For LIBs, thermal alloying of Li with Si nanoparticles [22,23], Li-metal free pre-lithiation of Si in an electrolytic cells [24], and poly-methyl-methacrylate (PMMA) protected lithium metal as pre-lithiation agent [25] have shown promise in mitigating the loss of Li due to SEI buildup. More noticeably, stabilized lithium metal powder (SLMP) developed by *FMC Corporation* has demonstrated feasibility as a commercial solution to prelithiate the anodes or cathodes without significant modification of the current battery manufacturing process [26,27]. As for SIBs, Tarascon's group recently reported a pre-sodiation technique via direct ball milling of sodium metal with cathode/anode materials under inert environment [28]. Another study using 10 wt% NaN_3 powder as cathode additive also achieved 60% increase in reversible capacity [29].

In this work, we report a new pre-sodiation technique using sodium powder that can be applied to anode and potentially cathode materials in a facile method. The synthesis of sodium powder is accomplished via pulsed ultrasonic dispersion of molten metallic sodium in mineral oil in an inert atmosphere. When cleaned and suspended in hexane, the sodium powder can be easily applied onto carbon electrodes for pre-sodiation. Reduction in the 1st cycle capacity loss and enhancement in reversible capacity are observed with sodium powder application on carbon anodes in this study.

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