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## Novel Li<sub>2</sub>FeSiO<sub>4</sub>/C Composite: Synthesis, Characterization and High Storage Capacity

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Figure S1. Schematic illustration of the synthesis procedure.



**Figure S2.** TG/DTA plots and the coupled mass spectra of the precursor: (a)TG, (b) DTA, (c) DTG, (d) m/z = 44, (e) m/z = 28, and (f) m/z = 18.

The decomposition process of the precursor (namely the as-obtained xerogel) was studied by TG-MS as shown in Fig. S2. TG curve, corroborated by MS analysis, can be divided into three regions i.e., from room temperature to 150 °C, 150-500 °C, and 500-800 °C (labeled as R1, R2, and R3, respectively). In region R1, the weight loss of 5 wt% below 150 °C is related to the dehydration of physically adsorbed water in precursor. In region R2, there is a significant weight loss of 65 wt%, which were ascribed to the decomposition processes of organic groups from acetate ion or citric acid,<sup>40</sup> proved by the coupled MS analysis (Fig. S2 d). In a high temperature range of R3, a large endothermic peak was observed at around 590 °C without obvious weight loss (Fig. S2b), which corresponded to the crystallization of Li<sub>2</sub>FeSiO<sub>4</sub>; the slight release of CO at 790 °C (Fig. 2c) may relate to the reaction between the Li<sub>2</sub>FeSiO<sub>4</sub> and its compact carbon coating layer.

Based on the analysis of TG-MS, we can choose the precalcination (ca. 400 °C) and calcination (ca. 600 °C) temperatures efficiently in our synthesis process. Herein, we selected 400 °C in stead of 500 °C as the precalcination temperature because a slower heating rate of 2 °C min<sup>-1</sup> was adopted in our material synthesis process compared to that of 10 °C min<sup>-1</sup> in TG-MS experiment. In fact, a slower heating rate is known to lead to an apparent shift of the decomposition steps to a lower temperature.