Emerging presodiation strategies for long-life sodium-ion batteries

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Sodium-ion batteries (SIBs) have attracted increasing attention as electrochemical energy storage in academic research and industrialization due to abundant reserves, low cost, and excellent power performance. Unfortunately, the commercial application is impeded by the low initial coulombic efficiency (ICE) and limited cycle life owing to largely irreversible loss of Na⁺, so the presodiation techniques have been crucial strategies to compensate capacity loss. For emerging technology, some reviews have summarized the presodiation methods according to mechanisms and their corresponding performance with specific applications in the past few years. However, there has rarely been sorted based on electrode materials systematically though the normal sequence is that electrode materials were selected first and then optimized for improved performance happened. Therefore, this review focus on the presodiation process and the performance from the perspective of electrode materials. In addition, the perspective of how to promote the further development of presodiation was described. We believe this review will provide a novel sight between electrode materials in SIBs and presodiation strategies and contribute to the rapid development and application of SIBs in the future.

With the rapid consumption of traditional fossil fuels and increasing prominence of global environmental issues,¹ it is crucial to utilize green and renewable energy resources such as solar, wind, and geothermal.² Among them, alkaline rechargeable metal-ion batteries have been widely applied in electrochemical energy storage as a significant part of energy storage systems due to environmental friendliness.³ Sodium-ion batteries have attracted increasing attention as electrochemical energy storage due to abundant reserves, low cost, and better safety. SIBs are especially suitable for large energy storage systems in the future market,⁴ where the most promising characteristic for developing practical SIBs is ultra-long life. The ICE as an important parameter to present batteries’ performance, monitors the lifespan and rate performance of SIBs.⁵ And it is one of essential role standing for the reversible de-/intercalation of Na⁺ and how much the irreversible reaction occurs on the electrode materials, which ultimately affect the cycling life of batteries. However, unmodified batteries usually feature low ICE mainly due to unstable interface and irreversible reaction of electrodes. For example, unstable generation of solid electrolyte interphase (SEI) and side reactions on electrode surfaces cause continuous consumption of Na⁺ in electrolytes. Meanwhile, the insertion process is much higher than that delivered back by the electrode material during the desodiation process which results an insufficient sodium ion amount for sustainable cycle. Thus, how to improve ICE and maximize the cycling-life of batteries remains an important issue.⁶

To improve ICE effectively, lots of researchers have taken efforts to come up with lifting strategies, which were summarized briefly as follows: (1) Controlling the electrode materials through regulating the amounts of defects and doping heteroatom; (2) Reducing the electrolytes’ consumption by preparing the electrode materials with low surface area; (3) Developing the additives in electrolytes to stabilized the SEI; (4) Using the presodiation reagents or sodium-doped electrode materials to bring extra Na⁺ and compensate the irreversible consumption of Na⁺ during cycling. Among them, the effects of materials optimization are relatively limited when the ICE is improved to above 90%. It is also difficult to influence the formation of SEI precisely in subsequent cycles through directly operating on electrodes. Meanwhile, the method of adding additives is not extensively applied due to the lack of suitable additives. The presodiation is an effective strategy with engineering advantage for large-scale application to compensate for the initial irreversible capacity loss and improve ICE. After presodiation treatment, ICE can achieve nearly 100%. In addition, presodiation can not only be used in various electrode materials but also electrolyte for improving stability and performance. The presodiated anodes exhibit a reduced and flat potential curve during charge

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and discharge, which means an increased voltage range and higher energy density for full cell. For presodiated sodium-rich cathode, the Na⁺ in the lattice structure will not be consumed excessively with supplementation of additional sodium source, thus ensuring the stability of the cathode material and achieving excellent electrochemical performance in long cycle.⁷ For sodium-deficient cathode, additional Na⁺ need to be extracted from the electrolyte as the cathode cannot provide sufficient Na⁺ to cope with the adverse consequences of irreversible Na⁺ loss. Supplementing Na⁺ by presodiation reduces the loss of Na⁺ in electrolyte and thus maintains the stability of the electrolyte. It is worth noting that the stability of the liquid concentration has a significant impact on the extension of the battery life.⁸ Furthermore, the presodiation degree could be controlled precisely, which means the energy density could be greatly released with the improvement of ICE in SIBs. Therefore, a systematic summary of the presodiation strategy will effectively promote the practical advancement of SIBs.

Nowadays, most reported presodiation reviews usually principally summarized the presodiation strategies in the ways of following: (1) The usage of sodium metal and its alternatives; (2) The adoption of extra additives; (3) The operation between active sodium sources and SIBs. However, it is rare to compare the effects achieved by various presodiation methods on one specialized electrode material. Contemplating that to provide an effective and concise guidance to improve electrochemical performance, we propose a perspective of different parts of SIBs (anodes and cathodes) matched with main presodiation strategies (direct contact, electrochemical method and chemical synthesis method). In this review, the investigations about various influence factors of presodiation from a theoretical and experimental perspective are systematically explored. The conspicuous advantages and defects of presodiation are proposed and systematically evaluated as well. In the end, we put forward rational suggestions and prospects on the further research and potential challenges of presodiation strategy towards commercialization.

Presodiation of sodium-ion batteries

Presodiation of cathode is to supplement Na⁺ which would be transported to anode in advance, so as to prevent excessive irreversible consumption of the transported Na⁺ and ensure structure stable on cathode during cycle.⁹ As for presodiation of anode, it directly supplements irreversible loss of Na⁺, and in order to prevent the subsequent continuous consumption of Na⁺ due to unstable interphase, anode often needs to preform SEI with excellent quality after supplementing Na⁺ which can keep beneficial effects of presodiation.¹⁰ Based on above analysis, the presodiation of cathode is more inclined to simply supplement active Na⁺, and the requirement of presodiation is safe and efficient, which does not affect the stability of electrode structure and battery system. The presodiation of anode also needs to consider the long-term cycle after supplementing Na⁺, avoiding the consumption of active Na⁺ by the rupture and growth of SEI layer. Therefore, recent researches on cathode additives are biased towards the hardnessless to the battery system after supplementing Na⁺ efficiently, while the researches on anodes are biased towards forming high-quality interphase to maintain long life cycle after presodiation.

The reported presodiation technology is commonly classified into four methods, including: (1) Direct contact (DC); (2) Electrochemical presodiation (EP); (3) Chemical synthesis presodiation (CSP); (4) Adding additives. The strategy of direct contact can be divided into two main operations to supply Na⁺ directly, one is putting sodium metal and electrode together to produce sodium-rich electrode, another is adding tiny electrolyte between electrode and sodium metal to make short circuit. While electrochemical presodiation achieves Na⁺ embedded in the electrode material by assembling the half cell with sodium metal and discharge to a certain voltage, and then dismantle the battery and assemble the full cells with other counter electrodes. Chemical synthesis presodiation can obtain presodiated materials through chemical reaction in solvent so that Na⁺ could be embedded in the electrode material. Adding additional additives is introduced to replenish the active Na⁺ lost by the electrode during the initial cycle in advance.

It should be pointed out that each presodiation method has its practical application scenario based on specific academic and industrial requirements. Direct contact could simplify the disassembly process. However, it requires the process to be implemented in strict environments with H₂O<0.1 ppm and O₂<0.1 ppm due to high reactivity of sodium metal, which limits its large-scale commercial application. Meanwhile, it is worth mentioning that the adhesion of sodium metal on the electrode side may cause a series of safety issues and increase the difficulty of operation caused by the high viscosity of sodium metal. It is suggested to consider the pressure between electrode and sodium metal. Electrochemical presodiation could accurately control the presodiation degree through tuning the electric quantity and potential by assembling the half cell. However, the complicated process of assembling and disassembling half-cells requires extremely inert environments. Chemical synthesis presodiation can control the degree of presodiation by adjusting reagent concentration, soaking time or spraying dose. This method requires chemical reagents to have a sufficiently low redox potential to presodiate the electrode materials. The use of sacrificial additives has the advantage of being easy to implement and safety compared to other methods. Sacrificial additives needed to be chemically stable because they should not react with most solvents and binders during the cycles. The desodiation potential of the cathode and the additive must be aligned.

In view of adopting different presodiation strategies always leads to gap in electrochemical performance. It is vital to select suitable strategy to presodiate specific electrode material combining the main defect of material and outstanding advantages of strategy. So, the progress of presodiation in SIBs was summarized based on the main electrode materials, and a schematic overview is illustrated in Scheme 1.

Presodiation of Anodes

Presodiation can overcome the negative effects of anodes with their own defects effectively, such as carbon-based anodes’ high defects and oxygen-containing groups on material surface, conversion-type anodes’ high voltage platform and
poor reversibility and alloying-type anodes’ large volume expansion and so on. For carbon-based anodes, most researches focus on how to preform high quality interphase after supplementing Na\(^+\), which address low ICE caused by large specific surface areas and irreversible sites. The majority of presodiating conversion-type anodes pay more attention to conversion potential to minimize the adverse influence of side reactions. Alloying-type anodes have to deal with severe interphase problem caused by large volume expansion, which require flexible and high-rigidity SEI. It should be noted that this material face more safety issues during presodiation.

**Intercalation-type anode materials**

Intercalation-type anode materials are based on the reversible intercalation and extraction of Na\(^+\) in the lattice of a host anode material with layered structures which mainly include carbon-based materials and Ti-based materials.\(^{[11]}\) Carbon-based anode materials mainly include hard carbon (HC), soft carbon (SC), carbon nanotubes (CNTs), graphene and graphite, which generally have low ICE and poor cycling performance because of their high defects on material surface and specific surface areas which induce continuous consumption of electrolyte to form the unstable SEI and produce lots of irreversible Na-storage sites.\(^{[12]}\) While, although Ti-based anode materials could cycle in high rate and keep volume steady, their low specific capacity and ICE still hamper their development.\(^{[13]}\) Lots of researchers have done large amounts of work to compensate for the irreversible capacity loss in the initial cycle through presodiation. At the same time, to maintain the benefits of presodiation operated on anode side, it is crucial to build high quality SEI formed by Na\(^+\) supplemented mainly from presodiation.

For HC, it is necessary to combine Na\(^+\) supplementation with the incubation of high quality SEI due to its high defect concentration. In order to simplify above process, some researchers directly contacted the electrode with the sodium foil under a certain pressure in the presence of electrolyte, which made anode thermal react with the sodium metal spontaneously under micro short circuit. For example, Lim et al.\(^{[14]}\) developed a direct-contact approach to presodiate HC anode by pressing sodium metal to electrode with tiny electrolyte, the presodiation process was shown in Figure 1a. But it is not well-performed just passivate HC anode with normal electrolyte at room temperature. The unstable interphase will still prevent Na\(^+\) supplement by presodiation from being well utilised. So some researchers incubated robust SEI through direct contacting HC anode wetted by fluoroethylene carbonate (FEC)-rich warm (temperature of 50 °C) electrolyte with sodium metal,\(^{[15]}\) which was shown in Figure 1b. As a result, the assembled HC||[Na\(_3\)V\(_2\)(PO\(_4\))\(_3\)]\(_{x}\)F\(_3\)(NVPF)@CNTs@C full cells showed high ICE (~97%) with an excellent capacity retention of ~99% after 100 cycles. In comparison to room temperature, the 50 °C electrolyte increased the reaction kinetics between Na metal and HC and accelerated the full SEI formation on the surface of HC. However, it is difficult to control the degree of sodiation precisely by direct contact method whose effect largely depends on the reaction time. It is still better to take electrochemical presodiation if considering the presodiation degree and the formation of SEI primarily. The three-electrode strategy was adopted to presodiate HC anode by using sodium metal as the counter electrode in full cell for the first time.\(^{[16]}\) Electrodes and sodium metal were separated by separator and HC could be sodiated by sodium metal during the discharge process. The electrochemical performance of full cell displayed an improvement in ICE and capacity. In practical application, electrochemical presodiation is adopted by tuning the electric quantity and potential through assembling the half cell. Wang et al.\(^{[17]}\) proposed a presodiated phosphorus/Super P//HC anode by assembling half cell followed by the discharging process, which delivered higher reversible capacity and increased energy density. Compared to direct contact and electrochemical presodiation, chemical synthesis presodiation could add active Na\(^+\) in anode more efficiently and conveniently. For instance, sodium biphenyl (Na-Bp) was utilized to presodiate HC electrode (Na\(_x\)HC) by a
chemical reaction (Figure 1c). The large potential difference between HC (2.5 V vs Na+/Na) and Na-Bp (0.12 V vs Na+/Na) reagent makes presodiation spontaneous. HC accepts electrons from the BP radical while accommodating Na+ to form NaHHC, and the BP radical anion is converted to the initial state of biphenyl. At the same time, a reductive decomposition of electrolyte will happen to form SEI film on HC electrode surface due to its low potential when NaHHC is immersed in the electrolyte. The NaHHC[Na2V3(PO4)3] (NVP) full cell reached a high ICE of ~95.0%, an elevated energy density of 218 Wh kg−1 and capacity retention of ~100% after 1000 cycles. If suitable reagents were found, this method is a potentially industrialized approach to promote the production of presodiated electrode materials. Sun et al. developed sodium naphthalene (Na-Naph) solution to presodiate HC at the electrode level with promising commercial applications. Na-Naph can react with oxygen-containing functional groups on HC to make Na-containing functional groups due to its low redox potential and strong reducibility. Meanwhile, presodiated HC will react with electrolyte to preform SEI before assembling half cell, the presodiation process was shown in Figure 1d. The ICE was improved to 87% (vs. 67%) significantly and presodiated full cell showed dramatic increase in energy density (from 141 to 240 Wh kg⁻¹). Recently, sodium diphenyl ketone (Na-DK) was reported to realize dual-functional presodiation as a new chemical reagent for HC anodes. HC was immersed into Na-DK solution and got Na⁺ to fill its irreversible site. Then the robust NaF-rich SEI was formed on HC in 1.0 M NaPF₆ in diglyme through spontaneous chemical reaction, which favoured the interfacial reaction kinetics and stable Na⁺ insertion and extraction (Figure 1e). The presodiated HC delivered high ICE of ~100% and capacity retention of 82.4% after 6800 cycles unbelievably. This work offers a simple and efficient strategy to improve the interfacial reaction kinetics and cycle stability. Furthermore, in order to improve their complex batteries’ performance deeply, many researchers also regarded presodiating HC anodes as a crucial step. For instance, An et al. reported full cell with spray-dried NVPF/C cathode and presodiated HC anode, which fulfilled a remarkable reversible capacity. Yin and co-workers matched presodiated HC with their B-doped cathode material to prove their concept. SC has attracted attention due to its low cost, tunable interlayer distance, and favorable electronic conductivity.
However, its ICE is lower than HC due to its higher defects-rich structures.[23] It is important to modify and pretreat the SC anode to improve electrochemical performance. The phospho-doped soft carbon (P-SC) anode was assembled into half cell to get active Na\(^+\) and preform SEI in the discharging process, which showed high CE (over 90%) and long-cycle stability (Figure 2a).[24] The quality of SEI formed by electrochemical presodiation is not always good. In order to ensure the quality of preformed SEI, some researchers delivered a novel approach that carries out alumina coating preserves to build artificial SEI layer after presodiation. For example, Bubil et al.[25] established a thin alumina coating layer on presodiated amorphous SC by atomic layer deposition technology (Figure 2b). Presodiation keeps active Na sites in the SC from the adverse effects of the artificial SEI.

In contrast to HC and SC, the two-dimensional carbon like reduced graphene oxide (rGO), can utilize the carbon materials as much as possible and deliver a high rate performance, but their ICE are lower than above materials because their exposed surface react with electrolyte more severely.[26] To solve this problem, Na-Naph dissolved in dimethoxysythenane (DME) were used to presodiate rGO driven by their large potential differences in an ultrafast chemical method (10 s).[27] At the same time, Na\(^+\)-(DME)\(_x\) complexes in the solution have lower unoccupied molecular orbitals (LUMOs) than the Fermi level of rGO, which leads to form artificial SEI on rGO surface caused by the decomposition of electrolyte. The ICE of rGO increased to 96.8% due to the artificial SEI on its surface. Besides rGO could be used as electrode materials, the huge energy of rGO under microwave treatment was also used to carbonize cellulose nanofibers (CNFs) and presodiated CNFs through inducing NaOH assisted with 2,2,6,6-tetramethyl-piperdine-1-oxylradical (TEMPO),[28] which was shown in Figure 2c. The microwaved rGO-CNF anode delivered a high initial capacity of 558 mAh g\(^{-1}\) and the capacity of 340 mAh g\(^{-1}\) remains after 200 cycles. This work provided a unique method to presodiate carbon-based anodes for large-scale application.

Titanium-based materials have appropriate reaction potential, low lattice strain, good stability and low cost but they still have to face severe problem of low ICE.[29] Li and co-workers adopted a convenient way which immersing Na\(_2\)Ti\(_2\)O\(_7\) anode in the Na-Naph-DME liquid solution directly without presassembling/disassembling.[30] They controlled the degree of sodiation by changing the concentration of the liquid source. The ICE of the Na\(_2\)Ti\(_2\)O\(_7\) electrode was greatly improved from 65% to 100%. While improving ICE effectively, the electrode still keep a good rate capability due to undestroyed crystal structure. To show the presodiation efficiency for different presodiation methods clearly, the electrochemical performances of the carbon-based anodes and Ti-based anodes after presodiation was summarized in Table 1.

### Conversion-type anodes

Conversion-type anode materials are based on reversible replacement redox reactions between sodium-ions and transition-metal cations, which include transition-metal oxides, sulfides, selenides, nitriles, and phosphides that are of relatively low cost and relatively high capacity (such as Co\(_2\)O\(_4\), FeS\(_2\), MnO\(_2\), and NiCo\(_2\)O\(_4\)).[31] As evident from previous research, the conversion voltage in SIBs is lower than that observed in lithium-ion batteries (LIBs),[31] which indicate poor reversibility of conversion reactions. Although the theoretical capacity of conversion-type materials is usually much higher than intercalation-type materials, the decay of capacity during the cycle is faster due to the unstable SEI caused by the formation or early termination of the conversion reaction. So it is vital to provide a stable interface and control conversion

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**Fig. 2** Presodiation of soft carbon, reduced graphene oxide and Ti-based anodes. A Schematic of SEI formation during the presodiation process and cycle stability of the sodium dual-ion batteries.[24] Copyright 2018, American Chemical Society. B. Graphic illustration of the effect of the different preparation steps on the sodiation mechanism.[25] Copyright 2021, Elsevier. C. Schematic of the synthesis process and charge/discharge cycling performance of microwaved reduced graphene oxide cellulose nanofibers.[28] Copyright 2019, Wiley.
potential to take reversible replacement redox reactions.

For example, the reduction of Fe-based oxides to metal along with the related conversion reaction is dominant below 0.75 V. Large irreversible capacity is occurred in this region. In order to compensate for the irreversible sodium loss of the conversion reaction in advance, the carbon coated Fe$_3$O$_4$ anodes were presodiated up to Na$_{1.7}$Fe$_3$O$_4$ by direct contacting with sodium metal in the electrolyte of 1 M NaClO$_4$ in ethyl methanesulfonate (EMS)/FEC. The capacity retention and rate capability was improved significantly explained by shallow conversion reaction between Fe metal and Fe$_3$O$_4$. Similarly, the irreversible capacity of tin-based oxides was mainly observed above 0.9 V indicating large amounts of side reactions would happen during the first sodiation process such as reductive decomposition of electrolyte and SEI formation. Myung et al. minimized the initial capacity loss through direct physical contacting of SnO$_2$/rGO with sodium for 30 min in electrolyte. Correspondingly, electrodes were presodiated to 0.9 V to minimize the irreversible capacity. As for Co$_2$O$_4$, its main capacity is connected to the flat plateau at 0.12 V. The highest loss in capacity retention is related to the flat plateau occurring at low potential. Ruffo and co-workers used direct contact method by putting sodium metal and Co$_2$O$_4$ anodes wetted with electrolyte together for a while. The long lasting plateau at 0.12 V was suppressed in favour of a shorter process occurring at high potential (0.31 V), which delivered a higher capacity of 600 mAh g$^{-1}$ after 50 cycles (Figure 3a). Recently, metal nitrides have advantages of superior electrical conductivity and high ionic diffusion. The three-dimensional N-doped carbon foam (3DNCF) anode was presodiated via discharging-charging for 3 cycles in half cells. The Fe$_3$N/C/3DNCF showed a high ICE and good capacity retention because presodiation supplement Na$^+$ to preform SEI. At the same time, its reversible conversion from Na$_3$N and Fe to Fe$_3$N is stable in the range of 0.9-1.5 V after the activation process. Metal sulfides have been paid great researches due to enhanced mechanical stability, electrical conductivity, and better reversibility compared to their metal oxide counterparts. NiS$_2$ is a representative material with above mentioned advantages. Yang et al. presodiated heterostructured NiS$_2$/SnS$_2$ anodes between 0.01 and 3.0 V at 0.2 A g$^{-1}$ for three cycles. The full cell showed an extraordinarily high ICE of 94.3%. Typically, iron sulfides manifest prominent advantages such as natural abundance and environmental benignity. Our team prepared carbon-coated sodium-iron-disulfide (NaFeS$_2$/C) electrode material through chemical synthesis method, which mixing NaCl, Na$_2$S·9H$_2$O, FeCl$_2$·6H$_2$O and glucose together firstly and then annealing precursor at 750 °C for 3 hours under Ar atmosphere. Compared with Fe$_2$ without presodiation, NaFeS$_2$ electrode shows higher density of states (DOS), reactivity and reversibility of conversion reaction, showing better electrochemical performance. In addition, an ultrathin and NaF-rich SEI is formed on the surface of NaFeS$_2$/C electrode (Figure 3b). NaFeS$_2$/C electrode exhibits extremely high ICE (96.7%), extremely high-rate performance (136 mAh g$^{-1}$ at 100 A g$^{-1}$), excellent cyclic stability (343 mAh g$^{-1}$ after 2500 cycles at 5.0 A g$^{-1}$) (Figure 3c). In addition to applying the traditional strategies of presodiation to the electrode, multifunctional binders have been used to improve the electrochemical performance of electrode materials in recent years. For example, sodium polycrylate (PAA) as the binder could offer extra sodium source to compensate for the loss of active Na$^+$ during cycling. Based on this characteristic, Chen’s group selected PAA as the binder of FeS anode to solve low initial coulombic efficiency and serious capacity fading owing to the shuttle effect and volumetric expansion. The PAA binder enhanced ICE through contributing to form high-quality SEI and decreasing the decomposition of electrolyte. This work provides a novel presodiation

<table>
<thead>
<tr>
<th>Electrode materials</th>
<th>Pre-sodiation methods</th>
<th>Energy density (Wh kg$^{-1}$)</th>
<th>$1^{st}$ charge/discharge capacity (mAh g$^{-1}$)</th>
<th>Cyclability</th>
<th>ICE</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>HC</td>
<td>DC</td>
<td>~279/300</td>
<td>99% after 40 cycles at 0.1 C (full cell)</td>
<td>94% (vs. 73%)</td>
<td>1 M NaClO$_4$ PC/FEC(98:2wt%), 0.01–2 V</td>
<td>[14]</td>
</tr>
<tr>
<td>HC</td>
<td>DC</td>
<td>~250</td>
<td>~99% after 100 cycles at 50 mA g$^{-1}$</td>
<td>97%</td>
<td>1 M NaClO$_4$ EC/PC + 5 vol% FEC, 0.001–2.0 V</td>
<td>[15]</td>
</tr>
<tr>
<td>HC</td>
<td>EP</td>
<td>~126/93</td>
<td>72% after 100 cycles at 30 mA g$^{-1}$</td>
<td>73% (vs. 64.3%)</td>
<td>1 M NaClO$_4$ PC, 1.5–2 V</td>
<td>[16]</td>
</tr>
<tr>
<td>P/HC</td>
<td>EP</td>
<td>~135/110</td>
<td>capacity starts to decay after 30 cycles at 0.5 C</td>
<td>~79.1% (vs. 73.9%)</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>HC</td>
<td>CSP</td>
<td>300/301</td>
<td>~100% after 1000 cycles at 100 mA g$^{-1}$</td>
<td>~100% (vs. 70%)</td>
<td>1 M NaClO$_4$ PC/EC/DEC, 0–2 V</td>
<td>[17]</td>
</tr>
<tr>
<td>HC</td>
<td>CSP</td>
<td>~185/212</td>
<td>~81% after 100 cycles at 25 mA g$^{-1}$</td>
<td>87% (vs. 67%)</td>
<td>1 M NaClO$_4$ EC/DMC + 2 vol% FEC, 0.01–1.5 V</td>
<td>[18]</td>
</tr>
<tr>
<td>HC</td>
<td>CSP</td>
<td>~290/295</td>
<td>82.4% after 6800 cycles at 500 mA g$^{-1}$</td>
<td>99.2% (vs. 84.5%)</td>
<td>1.0 M NaPF$_6$, diglyme, 0.01–1.5 V</td>
<td>[19]</td>
</tr>
<tr>
<td>HC</td>
<td>DC</td>
<td>~130/133.9</td>
<td>80.1% after 100 cycles at 1 C</td>
<td>—</td>
<td>1 M NaFeS$_2$ in EC/DEC + 5 vol% FEC, 1.5–4.2 V</td>
<td>[20]</td>
</tr>
<tr>
<td>P-SC</td>
<td>EP</td>
<td>309/112</td>
<td>81.8% after 900 cycles at 1000 mA g$^{-1}$</td>
<td>36.2% (vs. 46.9%)</td>
<td>1 M NaPF$_6$, ED/DMC(6.4, v/v), 2.0–4.7 V</td>
<td>[21]</td>
</tr>
<tr>
<td>rGO</td>
<td>CSP</td>
<td>~315/325</td>
<td>68.4% over 1000 cycles at 0.5 A g$^{-1}$</td>
<td>96.8%</td>
<td>1 M NaPF$_6$, DME 0.005–2.5 V</td>
<td>[22]</td>
</tr>
<tr>
<td>rGO</td>
<td>CSP</td>
<td>~420/558</td>
<td>60.0% after 200 cycles at 100 mA g$^{-1}$</td>
<td>75% (vs. 59%)</td>
<td>1 M NaClO$_4$ EC/DMC.0.001–2 V</td>
<td>[23]</td>
</tr>
<tr>
<td>Ti</td>
<td>CSP</td>
<td>~185/190</td>
<td>~94% after 200 cycles at 100 mA g$^{-1}$</td>
<td>99% (vs. 65.7%)</td>
<td>0.001–2.5 V</td>
<td>[24]</td>
</tr>
</tbody>
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Presodiation of conversion-type anodes. a Sodiation/desodiation profiles of pre-treated (red) and pristine (black) cobalt oxide needles electrode, solid and dashed lines refer to first and second cycle respectively and cycling stability and coulombic efficiency of the two electrodes. Copyright 2016, Elsevier. b Schematic illustration of the possible Na⁺ storage mechanism of the NaFeS₂@C electrode and presodiation effects on SEI thickness and component. Copyright 2021, Elsevier. c Electrochemical performance of NaFeS₂@C and FeS₂@C electrode and charge/discharge curve of the initial cycle and cycle performance of NaFeS₂@C and FeS₂@C electrodes. Copyright 2021, Elsevier.

strategy based on selecting sodium-rich binder to improve ICE in SiBs. The electrochemical performance of different presodiation methods based on conversion-type was summarized in Table 2.

**Alloying-type anodes**

During the last decade, alloying-type anodes (Sn, Sb, Bi, Si, Ge and P) have attracted lots of attention due to their high specific capacities and low redox potential. However, alloying-type anodes still have to face enormous challenges, such as large volume expansion during charge/discharge, which may cause electrode pulverization, the rupture and continuous growth of SEI, and consequent capacity fading. As a result, it is crucial to pre-incubate high quality SEI with higher degree of flexibility and mechanical stability. Presodiation can solve above problems effectively by compensating the initial active Na⁺ loss and preforming SEI subsequently.

The volume expansion of Sn-based electrode reaches 420% during the phase transformation and the first cycle irreversible capacity loss is too high for practical application. Meanwhile, Sn could catalyze electrolyte’s decomposition due to its electron configuration of 4d¹⁰ 5s² 5p², which would release large amounts of gas at high open-circuit voltage (OCV). To alleviate this issue, Liu et al. found that Sn could lower their voltage platform through alloying reactions with sodium metal. As a result, they produced Sn-Na alloy by putting sodium metal foil and Sn metal foil together and then using roller to press (Figure 4a), which decreased the OCV and alleviate a serious gas problem at Sn anode. The method of direct contact is suitable because the metal anodes spontaneously undergo a thermodynamic reaction with sodium metal. However, the presodiation with different metal has certain environmental requirements, which increases the cost of electrode production and post-maintenance. Recently, some researchers melted Na and injected it into Sn nanocrystals within the hollow carbon nanofibers (Sn@HCNF) composites by coaxial electrospinning technique, as shown in

**Table 2.** Presodiation methods and electrochemical performance of conversion-type anodes for SiBs.

<table>
<thead>
<tr>
<th>Electrode materials</th>
<th>Pre-sodiation methods</th>
<th>Energy density (Wh kg⁻¹)</th>
<th>1st charge/discharge capacity (mAh g⁻¹)</th>
<th>Cyclability</th>
<th>ICE</th>
<th>Electrolyte, Voltage range (V vs. Na⁺/Na)</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>C-Fe₃O₄</td>
<td>DC</td>
<td>—</td>
<td>~200/225</td>
<td>157 mAh g⁻¹ after 50 cycles at 2 A g⁻¹</td>
<td>—</td>
<td>1 M NaClO₄, EMS/FEC, 0–2 V</td>
<td>[32]</td>
</tr>
<tr>
<td>SnO₂/rGO</td>
<td>DC</td>
<td>—</td>
<td>~620/608</td>
<td>80.2% after 100 cycles at 100 mA g⁻¹</td>
<td>46.5%</td>
<td>1 M NaF₆, PC/FEC, 0–3 V</td>
<td>[34]</td>
</tr>
<tr>
<td>Fe₃N@C/3DNCF</td>
<td>EP</td>
<td>~375/335.9 (full cell)</td>
<td>95.2% after 400 cycles at 0.5 A g⁻¹</td>
<td>89.5%</td>
<td></td>
<td>1 M NaPF₆, EC/DMC + 5 wt% FEC, 0.5–3.25 V</td>
<td>[36]</td>
</tr>
<tr>
<td>NiS₂@SnS₂</td>
<td>EP</td>
<td>753/798 (full cell)</td>
<td>80.4% after 150 cycles at 0.5 A g⁻¹</td>
<td>94.3%</td>
<td></td>
<td>1 M NaF₆ diglyme, 0.5–3.5 V</td>
<td>[38]</td>
</tr>
<tr>
<td>NaFeS₂@C</td>
<td>CSP</td>
<td>689</td>
<td>343 mAh g⁻¹ after 2500 cycles at 5.0 A g⁻¹</td>
<td>96.7% (vs. 68.7%)</td>
<td></td>
<td>1 M NaClF₆SO₄ diglyme, 0.3–3 V</td>
<td>[39]</td>
</tr>
<tr>
<td>FeS</td>
<td>binder</td>
<td>—</td>
<td>~580/660</td>
<td>88.52% after 100 cycles at 1.64 A g⁻¹</td>
<td>87.4%</td>
<td>1 M NaClF₆SO₄ diglyme, 0.1–3 V</td>
<td>[40]</td>
</tr>
</tbody>
</table>

DOI: 10.54227/elab.20230008
Figure 4b. Driven by the capillary effect, Na$_{15}$Sn$_2$ alloy intermediate was formed in the hollow fiber. The alloyed electrode had a capacity retention of 89.6% after 300 cycles at 0.5C. Chemical synthesis presodiation is also a good choice because it could realize homogeneous presodiation. Consequently, Sn powder was immersed into Na-Bp/THF (tetrahydrofuran) solution to supplement Na$^+$. Then the presodiated Sb anode can form a highly fluorinated SEI layer by decomposition of specific electrolyte. Above process was driven the redox potential of Na-Bp/THF (0.095 V vs. Na$^+/Na$) and Na$_2$Sn. The ICE of pretreated Sn anode increased from 70% to more than 94%. The key to preform high quality SEI with chemical synthesis presodiation is to satisfy the requirement of redox potential between sodium-rich reagent, electrode and electrolyte.

Sn has high theoretical specific capacity of 660 mAh g$^{-1}$ by the formation of full sodiation product Na$_3$Sb and suitable redox potential. Based on the redox potential of Sb-based anode's sodiation product, Sn$_2$ anode material was immersed in Na-Bp reagent similar to Sn-based anode, which eliminate large irreversible Na losses and form a dense and highly fluorinated SEI. The comparison of redox potentials is Na-Bp (0.12 V), Na$_2$Sb (0.6 V) and FEC (1.0 V). In this work, ICE of the presodiated Sn$_2$ anode increased from 75% to 100% and with high capacity retention of 85% after 300 cycles at 4C. As a result, it is vital to form a NaF-rich SEI in this process. Bian and co-workers coupled the electrolyte containing 10 vol.% FEC with the presodiated Sn$_2$ anode, which induced a robust NaF-rich SEI on the surface. The nucleation barrier and voltage platform could be decreased through alloying Sn and Sb species with Na. Recently, a voltage-induced presodiation was used in Sb nanoparticles encased in hierarchical ZnO microflowers with coated carbon (Sb@ZMF/C) to decrease the nucleation overpotential, which the process was shown in Figure 4c. The presodiated Sb@ZMF/C was obtained by assembling Na foil and polyvinyl alcohol gel onto the Sb@ZMF/C in diglyme solvent and then discharging to 0.01 V at 0.1 mA cm$^{-2}$. The presodiated anode ICE up to 95.7%, the capacity retention rate was 90.1% after 400 cycles. During the presodiation process, Sb nanocrystals preferentially alloy with Na$^+$ to form a Na-Sb Na-philic phase, while the ZMF/C layer regulates volume expansion and uniform Na$^+$ flux.

Red phosphorus (RP) exhibit potentially ultrahigh theoretical capacity of 2596 mAh g$^{-1}$ by forming Na$_3$P. However, RP exhibits low electrical conductivity and a large volume expansion (300%) during sodiation/desodiation. Recently, some researchers have done lots of work to solve by presodiation. For example, the presodiated RP/CNT@G composite, ICE of pretreated electrode increased from 28.26% to 37.97%. Meanwhile, the P/C composite anode was immersed into Na-Bp/THF solution to achieve controllable presodiation treatment. The ICE of the pretreated electrode was increased from 64% to 94%, accompanied with the formation of more stable NaF-rich SEI. A summary of alloying-type anodes concentrating on presodiation methods, specific capacity, ICE, cycling stability, and electrolyte was listed in Table 3.

Presodiation of Cathodes

The development of research into presodiation of cathode has enormous advantages and needs that distinguish it from anode. (1) Enhancing capacity and stabilizing structure for cathode itself. (2) Improving the performance of full cells while cathode supplies Na$^+$ and has a smaller specific capacity than anode. (3) The researches of sacrificial additives mostly focus on cathode side because both of them release Na$^+$ during the desodiation potential. (4) Safer is a highlighted aspect which is demonstrated by a higher sodiation potential (vs. Na$^+/Na$) as compared to anode material. Sodiated anodes normally have higher reactivity as their sodiation potentials are adjacent to the Na deposition potentials. (5) Higher prospects for industrial applications because cathode could always maintain their initial capacity longer than anode stored in the same atmosphere. Meanwhile, every kind of cathode material has its inherent defects, such as P2-type...
transition metal oxides suffer sodium-deficient, polyanion compounds have low specific capacity and prussian blues are stuck by unstable interphase. It is crucial to specifically distinguish presodiation for each cathode material.

**Transition metal oxides**

Layered materials, with flexibility, versatility and excellent electrochemical performance, are expressed by the general formula of Na$_{x}$MO$_{2}$ (M = transition metal).\(^{[51]}\) According to the different Na$^+$ environments around, layered materials can be divided into P-type and O-type. Among them, O3 and P2 are the two common structures for SIBs. O3-type has a higher initial Na content and can deliver a higher capacity. P2-type has a larger Na layer spacing, which enhances transportation of Na$^+$ and maintains the integrity of the layered structure. The sodium-deficient cathode material allows limited amount of Na$^+$ to deintercalate from the structure while the Na$^+$ are inserted into anode. Namely, the resulting capacity is dependent on the first charge capacity. Thus, the extractable amount of Na$^+$ at the first charge is a critical parameter for cathode materials.

The capacity of cathode is always lower than anode, which limits the performance of full cell capacity. Presodiation could compensate for initial capacity loss of transition metal oxides to maximize the reversible capacity. The method of direct contact was used in Na$_{0.6}$Fe$_{0.5}$Mn$_{0.3}$O$_2$ cathode to compensate for the capacity loss in the initial cycle.\(^{[14]}\) Electrochemical presodiation was adopted in the Na$_{0.6}$Fe$_{0.5}$Mn$_{0.3}$O$_2$ cathode, which was in the discharge state for 1 cycle.\(^{[52]}\) Meanwhile, the partial substitution of Mn with Fe and Ni in P2-Na$_{0.6}$Fe$_{0.5}$Ni$_{0.3}$O$_2$ decrease the structural degradation by the multistep phase transition. The assembled full cell showed excellent cycle performance. The similar method was also used to presodiate the P3-type NaPO$_3$ coated Na$_{0.6}$[Mn$_{0.5}$Co$_{0.1}$O$_{2}$] cathode.\(^{[53]}\) Compared to P2-type, the capacity retention of P3-type is always inferior due to its lower crystallinity. In addition to presodiation, the adoption of coating NaPO$_3$ also improves ionic conductivity and suppresses the crack formation on cathode surface. Furthermore, in order to realize the commercial application of presodiation to transition metal oxides materials faster, it is crucial to develop a facile method for using. Xi et al.\(^{[54]}\) obtained Na$_{0.66}$Mn$_{0.2}$O$_2$ cathode by immersing Na$_{0.44}$Mn$_{0.2}$ (NMO) in Na-Bp solvent, which transforms the Na-poor phase into Na-rich phase. Na-Bp can reduce Na$_{0.44}$Mn$_{0.2}$O$_2$, which driven by the huge potential difference between Bp/Bp$^-$ couple (0.09 V) and Na$_{0.44}$Mn$_{0.2}$O$_2$ (2.81 V). The chemical presodiation overcomes the disadvantage of low initial charge capacity and provides strategic direction for the modification of sodium-deficient transition metal oxides in SIBs.

In addition to using traditional presodiation methods to improve the performance of transition metal oxides electrodes, many studies were also devoted to developing novel methods to compensate for the capacity loss. For instance, Ding et al.\(^{[55]}\) used a simple and special quenching treatment to presodiate Na$_{0.6}$Cu$_{0.1}$Ni$_{0.3}$Fe$_{0.2}$Mn$_{0.3}$O$_2$ material. As shown in Figure 5a, Mn$^{3+}$ tends to get oxidized at a lower voltage, therefore, the material with high Mn$^{3+}$ and Na$^+$ content obtained by the quenching treatment could achieve the purpose of self-presodiation by oxidizing Mn$^{3+}$ to release Na$^+$. It was worth noting that the surface of the cathode material prepared by quenching treatment had less Na$_{0.3}$CO$_3$, which improved its cycling performance. As a result, the electrode achieved a capacity of 132.6 mAh g$^{-1}$ at the first charge, with a capacity retention of 89% after 500 cycles at 1 C rate. Nowadays, the flexible devices applied in SIBs have attracted attentions, but it is difficult to find the suitable cathodes to meet the requirements of application. Herein, a novel chemical synthesis presodiation was used to form Na$_{0.4}$Co$_{0.2}$O$_2$ nanosheet arrays with expanded interlayer spacing and flexibility. And the formation progress and mechanism of the presodiated P2/P3 nanosheet arrays by "solvolithod sodation" was illustrated in Figure 5b.\(^{[56]}\) The P2/P3 heterostructured Na$_{0.4}$Co$_{0.2}$O$_2$ was obtained by immersing α-Co(OH)$_2$ nanosheet arrays into NaOH dissolved in ethanol firstly and then being heated with two steps (first in Teflon-lined stainless steel autoclave at 120 °C for 12 h and then at 600 °C for 6 h under Argon atmosphere). The electrode achieves a reversible specific capacity of 126.2 mAh g$^{-1}$ and a high capacity retention of 92.4% after 300 cycles at 1 C. This work offered an opportunity to construct transition metal oxides cathodes for flexible devices in SIBs.

Compared with above three mentioned traditional methods, adding additives to transition metal oxides cathode can maintain high capacity retention with easy operation. As for

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**Table 3. Presodiation methods and electrochemical performance of alloying anodes for SIBs.**

<table>
<thead>
<tr>
<th>Electrode materials</th>
<th>Pre-sodiation methods</th>
<th>Energy density (Wh kg$^{-1}$)</th>
<th>$1^{st}$ charge discharge capacity (mAh g$^{-1}$)</th>
<th>Cyclability</th>
<th>ICE</th>
<th>Electrolyte, Voltage range (V vs. Na$^+/Na^-$)</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>P/C</td>
<td>CSP</td>
<td>~800/850</td>
<td></td>
<td>~94%</td>
<td></td>
<td>1 M NaPF$_6$ EC/DEC + 5 wt% FEC, 0.01–2 V</td>
<td>[42]</td>
</tr>
<tr>
<td>Sn</td>
<td>CSP</td>
<td>~800/850</td>
<td></td>
<td>&gt;94%</td>
<td></td>
<td>1 M NaPF$_6$ EC/DEC + 5 wt% FEC, 0.01–1.5 V</td>
<td>[42]</td>
</tr>
<tr>
<td>Sn@HCNF</td>
<td>DC</td>
<td>~100/95 (full cell)</td>
<td>72.36% after 100 cycles (full cell)</td>
<td>75%</td>
<td></td>
<td>1 M NaPF$_6$ EC/DEC + 5 wt% FEC, 0.01–1.5 V</td>
<td>[43]</td>
</tr>
<tr>
<td>Sn@HCNF</td>
<td>DC</td>
<td>311.4</td>
<td>89.6% at 0.5 C after 300 cycles</td>
<td></td>
<td></td>
<td>1 M NaPF$_6$ diglyme, 2.8–4.2 V</td>
<td>[46]</td>
</tr>
<tr>
<td>Sb</td>
<td>CSP</td>
<td>540/540</td>
<td>85% at 4 C after 300 cycles</td>
<td>100%</td>
<td></td>
<td>1 M NaPF$_6$ EC/DEC + 5 wt% FEC, 0.05–2 V</td>
<td>[47]</td>
</tr>
<tr>
<td>Sb</td>
<td>EP</td>
<td>277</td>
<td>96% after 100 cycles at 0.2 C</td>
<td></td>
<td></td>
<td>1 M NaClO$_4$ PC + 10% FEC, 1.5–3.6 V</td>
<td>[48]</td>
</tr>
<tr>
<td>Sb@ZMF/C</td>
<td>DC</td>
<td>251.5</td>
<td>90.1% at 0.1 C</td>
<td></td>
<td></td>
<td>1 M NaPF$_6$ diglyme, 0.01–4.2 V</td>
<td>[49]</td>
</tr>
<tr>
<td>RP/CNT@rGO</td>
<td>EP</td>
<td>176</td>
<td>~650/240</td>
<td>37.97%</td>
<td></td>
<td>1 M NaPF$_6$ EC/DMC/EMC + 10 wt% FEC, 2–4.5 V</td>
<td>[50]</td>
</tr>
</tbody>
</table>

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Ref: 10.54227/elab.20230008
using sodium additives, there are two main ways, one is mixing a small amount of high-capacity material in the process of fabricating cathode materials, the other is spraying or coating additive thin layer on the cathode surface. The mainly reported sodium additives include the following: NaNiO2,[57] NaP2,[58] NaN,[59] NaNiO2,[60] Na2CO3,[61] Na2C2O4,[62] Na2C2O4,[63] Na2O2,[64] Na2O,[65] NaNO3,[66] ethylenediaminetetraacetic acid tetra-sodium salt (EDTA-4Na),[67] diethylenetriamine penta-acetic acid salt (DTPA-5Na).[68] During the first charging progress, the excess Na+ released by the cathode additives can migrate to the electrolyte and anode irreversibly after oxidation and decomposition at certain potential. For example, NaNiO2 was added to NaCrO2 cathode for the first time,[57] the irreversible phase transition of NaNiO2 in the first cycle provided additional Na+ ions to the anode. The charge and discharge capacities increased to 132.1 and 107.5 mAh g⁻¹ and the capacity retention up to 95.4% after 150 cycles (Figure 5c). Meanwhile, NaP2 was firstly proved to solve the problem of the cathode sodium deficiency, it can serve as an additional sodium reservoir to compensate for the anode consumption due to the formation of SEI.[58] Adding NaP sacrificial salt to P2-Na1[Fe0.5Mn0.5]O2 powders could increase the energy density of full cell with 7%. However, the gas of phosphine will be produced by adding NaP, which will cause serious security issues. Compared to NaP, NaN is more stable in air which means it could be used conveniently. Mar-

Fig. 5 Presodiation of transition metal oxides cathodes. a Redox behaviours of transition metal cations and oxygen anions of the layered O3- Na3TMO2 (TM = transition metal ion(s)) cathode and schematic illustrations of the quenching treatment for self-presodiation cathode compounds.[55] Copyright 2021, Wiley. b Schematic illustration of the synthesis procedure of the P2/P3-Na0.7CoO2 nanosheet arrays on the carbon cloth and the formation mechanism of the P2/P3 heterostructure via the solvothermal sodiation.[56] Copyright 2020, Elsevier. c Crystallographic structures of Na2NiO2 (space group: Cmc21) and full-cell voltage curves and cycle performance of the Sb/C composite anode and the NaCrO2 cathode with and without 10% Na2NiO2 substitution.[57] Copyright 2015, American Chemical Society. d Schematic diagram of preparing the electrode through spraying Na2O2-containing acetonitrile slurry and initial galvanostatic charge/discharge voltage profiles.[58] Copyright 2021, American Chemical Society.
tinez et al.\textsuperscript{[59]} added NaN\textsubscript{2} to the NaN\textsubscript{0.67}Fe\textsubscript{0.33}Mn\textsubscript{0.33}O\textsubscript{2} cathode to alleviate sodium deficiency issue. NaN\textsubscript{2} decomposition in the process of electrochemical cycle of N\textsubscript{2} can from the reduction in battery, so have less effect on the energy density. It is worth noting that, unlike NaP\textsubscript{2} additives, which have no obvious effect on the cycling performance of the battery, NaN\textsubscript{2} additives may have a negative effect on the cycling performance of the battery. Although the additive of NaN\textsubscript{2} could supply extra Na\textsuperscript{+} efficiently, it is still difficult to be used widely due to its explosive nature and toxicity. In P2 NaN\textsubscript{0.67}Al\textsubscript{0.33}Mn\textsubscript{0.33}O\textsubscript{2} composites, NaN\textsubscript{2}O\textsubscript{4} could act as a sacrificial salt, providing Na\textsuperscript{+} to increase the reversible capacity (~140 mAh g\textsuperscript{-1}).\textsuperscript{[60]} NaN\textsubscript{2}O\textsubscript{4} can also act as a chemical means to stabilize the P2 phase. But the introduction of NaN\textsubscript{2}O\textsubscript{4} will liberate CO\textsubscript{2} which was revealed by ex situ mass spectroscopy. Especially, O\textsubscript{2} has been proven to be produced when Li\textsubscript{2}CO\textsubscript{3} decomposes as additive in cathode during cycling.\textsuperscript{[69]} It has to remove the gaseous products, otherwise it will cause severe problems to sodium metal. Recently, NaN\textsubscript{2}O\textsubscript{3} was tested as Na sacrificial template in full cell, which decomposed at high voltage and generated Na\textsuperscript{+} and CO\textsubscript{2}.\textsuperscript{[61]} The NaN\textsubscript{2}O\textsubscript{3} additive increases the specific capacity of the composite in the first cycle and does not alter the reaction mechanism of the cathode material. However, the Coulomb efficiency is greatly affected by the decomposition of organic salts, indicating the existence of a serious irreversible decomposition mechanism, which indicates the need to design new salts in the future to improve full-cell performance. NaN\textsubscript{2}O\textsubscript{3} with high oxidation potential (~4.0 V vs. Na\textsuperscript{+}/Na\textsuperscript{0}) delivered high capacity retention and stable cathode-electrolyte interphase (CEI) when added to P2 NaN\textsubscript{0.67}Mn\textsubscript{0.33}Fe\textsubscript{0.17}Ti\textsubscript{0.17}O\textsubscript{2} cathodes. The formation of thin CEI layer in the presence of sacrificial sodium salt may be the result of salt decomposition, and the CO\textsubscript{2} produced by salt decomposition causes cracks in the formation of CEI layer. At the same time, the alcohol oxide formed on the anode electrode diffuses through the electrolyte and reacts with the CO\textsubscript{2} formed on the cathode electrode to fill the cracks of CEI. In addition, the reaction product can inhibit the further accumulation of CEI, thus controlling its thickness. Therefore, the gas produced at the expense of sodium salt is not a major problem in the battery manufacturing process. As a cathode additive, the capacity utilization ratio of NaN\textsubscript{2}O\textsubscript{4} is more than 99% (theoretical capacity is 400 mAh g\textsuperscript{-1}) and NaN\textsubscript{2}O\textsubscript{3} has no side effect on the electrochemical performance.\textsuperscript{[62]} The oxidation potential of NaN\textsubscript{2}O\textsubscript{4} can be reduced from 4.41 V to 3.97 V by introducing high specific surface area conductive additives, so that NaN\textsubscript{2}O\textsubscript{4} can be used as a sacrificial sodium source. Meanwhile, it is important to explore a series of new safer additives. NaN\textsubscript{2}O\textsubscript{3} was found that it could be stored stably under a dry atmosphere and has no obvious negative effect to the cathode. NaN\textsubscript{2}O\textsubscript{4} was sprayed on the Na\textsubscript{2}Ni\textsubscript{0.33}Ni\textsubscript{0.33}Fe\textsubscript{0.17}Ti\textsubscript{0.17}O\textsubscript{2} (NMT) cathode surface as a sacrificial sodium source (Figure 5d).\textsuperscript{[63]} The results showed that the energy density of the HC||VP||carbon cathode was increased from 134 to 175 Wh kg\textsuperscript{-1} and with capacity retention of 84.1 after 80 cycles at 0.5 C. Recently, the study of NaN\textsubscript{2}O\textsubscript{4} has shown that it could compensate sodium loss without by-products efficiently. The NaN\textsubscript{1.55}Ni\textsubscript{0.33}Ni\textsubscript{0.33}Cu\textsubscript{0.16}Mn\textsubscript{0.33}O\textsubscript{2} electrode and NaN\textsubscript{2}O\textsubscript{4}/Ru@graphene (NaN\textsubscript{2}O\textsubscript{4}/Ru@G) electrode were assembled into a composite cathode, in which NaN\textsubscript{2}O provides recyclable Na\textsuperscript{+} through Ru@G electrocatalyst, the specific desodiation process.\textsuperscript{[65]} Based on this technology, the ICE could reach up to 90%, the energy density was 295 Wh kg\textsuperscript{-1} and with capacity retention of 74% after 100 cycles at 0.5 C.

To further develop commercial applications, some researchers proposed additives that produce inert oxidative decomposition and less gas. For instance, EDTA-4Na was added into P2 NaN\textsubscript{0.67}Al\textsubscript{0.33}Mn\textsubscript{0.33}O\textsubscript{2} electrode directly and its decomposition provided Na\textsuperscript{+} and electroconducting C\textsubscript{N} deposits on the surface of the P2 NaN\textsubscript{0.67}Al\textsubscript{0.33}Mn\textsubscript{0.33}O\textsubscript{2} electrode.\textsuperscript{[66]} The reversible capacity of EDTA-4Na/NaN\textsubscript{0.67}Al\textsubscript{0.33}Mn\textsubscript{0.33}O\textsubscript{2}/HC full cell system increased to 152 mAh (g-oxide)\textsuperscript{-1} at the first discharge with a coulombic efficiency of 89% and a capacity retention of 77% after 200 cycles. Similarly, DTPA-5Na was added to the NaN\textsubscript{0.67}Mn\textsubscript{0.33}O\textsubscript{2} active materials directly to make cathode as a sacrificial sodium supply source.\textsuperscript{[68]} Compared to EDTA-4Na, DTPA-5Na is cheaper. The oxidation and decomposition of DTPA-5Na also produce conductive C\textsubscript{N} compounds in the composite electrode, thus slightly increasing the capacity. Table 4 summarized some relevant data of transition metal oxide cathodes assembled with various desodiation methods and additives.

### Polyatomic compounds

Polyatomic-type electrode materials, NaN\textsubscript{2}M\textsubscript{y}(XO\textsubscript{x})\textsubscript{N} (X = S, P, Si, As, Mo, etc.; M = transition metal), possess a series of tetrahedron anion units (XO\textsubscript{x})\textsuperscript{n+} and their derivatives (X\textsubscript{y}M\textsubscript{y-m+1}O\textsubscript{y}n), in which strong covalent bonds exist in M\textsubscript{y}O\textsubscript{x} polyhedral.\textsuperscript{[70]} Polyatomic compound cathode materials have shown excellent electrochemical performance, high ionic conductivity, and high thermal stability in SIBs. This material has shown long cycle performance due to its NASICON structure, which is composed of a highly covalent 3D scheme. It creates large interstitial spaces through which Na\textsuperscript{+} can easily diffuse.\textsuperscript{[71]} However, the development of these materials is limited by relatively low energy density and typical low conductivity.

To solve above problems, Mirza et al.\textsuperscript{[72]} changed NaN\textsubscript{3}V\textsubscript{5}(PO\textsubscript{4})\textsubscript{3} (NaN\textsubscript{3}VP) to a NaN\textsubscript{3}V\textsubscript{5}(PO\textsubscript{4})\textsubscript{3} (NaN\textsubscript{3}VP) cathode by using NaN\textsubscript{3}VP and sodium metal as counter and reference electrodes in three-electrode system. NaN\textsuperscript{+} was inserted to obtain NaN\textsubscript{3}VP, represented by one plateau at 1.46 V from 2.5 to 1.0 V (vs. Na\textsuperscript{+}/Na). The NaN\textsubscript{3}VP was desodiated below 2.2 V to compensate for the ICE of the anode. There is no residue produced by NaN\textsubscript{3}VP after desodiation that will reflect excellent electrochemical performance. The conversion between NaN\textsubscript{3}VP and NaN\textsubscript{3}VP is accompanied by one sodium, which causes the improvement of energy density. Meanwhile, considering the improvement from NaN\textsubscript{3}VP to NaN\textsubscript{3}VPN and finally to NaN\textsubscript{3}V\textsubscript{5}(PO\textsubscript{4})\textsubscript{3} (NaN\textsubscript{3}VP), the energy density will be improved higher with the reversible extraction of two sodium atoms. As a result, the NaN\textsubscript{3}VP was desodiated in the form of NaN\textsubscript{3}VPN which greatly improved coulombic efficiencies of sodium plating/stripping cycles (Figure 6a).\textsuperscript{[71]} The NaN\textsubscript{3}VP|carbon-coated Al full cell achieved an ultrahigh energy density of 400 Wh kg\textsuperscript{-1} and impressive capacity retention of 93% after 130 cycles in anode-free batteries. At the same time, benefiting from the desodiated NaN\textsubscript{3}VPN cathode, this work offers a sodium-free anode strategy to overcome safety issues. Recently, Sun et al.\textsuperscript{[73]} desodiated NaN\textsubscript{3}VPN electrode with 5% addition of ultrafine NaN\textsubscript{3}S/C (< 2 nm) and 5% carbon black achieved a
high first-cycle charge capacity of 128 mAh g⁻¹ (Figure 6b). The induced carbon framework constrained polysulfide dissolution into the electrolyte and provided an electronic pathway for the insulating Na₂S and S. There is no negative effect on electrochemical performance of cathode with the addition of Na₂S/C.

Adding sacrificial additives is also an effective method to presodiate polyaniomonic compound cathodes with the advantages of feasibility and low cost. For instance, NaCrO₃,⁶³ was firstly proposed as a sodium supplement additive on the cathode to compensate for the irreversible loss of sodium. The irreversible phase transition of NaCrO₃ not only provides an irreversible capacity of up to 230 mAh g⁻¹, but also ensures that the additive does not affect the reactivity of the battery subsequently. The high reversible capacity allows effective sodium supplementation with only a small amount of NaCrO₃. With the development of additives with cathode, organic compounds have been paid more attention due to structural diversity and environment friendly nature.⁶⁶ Shanmukaraj et al.⁷⁷ added Na₂C₀O₃ to Naₓ(VO)₂(PO₄)₂F cathode (Figure 6c), NaₓC₀O₃ oxidizes/decomposes to Na⁺ ions and CO₃ at a potential difference of 3.6 V, providing an additional specific capacity of 256 mAh g⁻¹ for the cathode in the initial cycle. The electrode with 30 wt % of Na₂C₀O₃ showed an increase in charge capacity similar to that of explosive NaN₃ with Na-carboxymethyl cellulose (CMC) binder and polyvinylidine fluoride (PVDF) binder. Following this idea, the sodium content of Na₂C₀O₃ with higher capacity was proposed.⁷⁸ Na₂C₀O₃ additive was obtained by sodiating the carbonyl groups of NaₓC₀O₃, which caused significant improvement in performance. Recently, Zhang et al.⁷⁹ proposed commercial sodium citrate as a novel sacrificial additive, which sodium citrate could deliver a considerable capacity of 312 mAh g⁻¹ with the capacity utilization of 97% for Na compensation. The mechanism of releasing Na⁺ in charged sodium citrate doped electrode was illustrated by X-ray photoelectron spectroscopy, which showed the electrochemical oxidation of the sodiated carboxyl group represented by the decomposition of the COONa group. Meanwhile, lithium citrate and potassium citrate are also proven to decompose electrochemically, showing that sodium citrate analogues can be extended as cathode additives for compensation with large potential. The electrochemical performance of polyaniomonic cathodes is listed in Table 5.

**Prussian blues**

Prussian blue (PB), also known as ferrocyanide, a coordination compound Fe₄(Fe(CN)₆)₃. PB cathode material has the advantages of open channel for rapid migration of Na⁺,⁸⁰ simple preparation and stable structure, so it has broad application prospects.⁸¹ However, fast capacity decay caused by unstable CEI is still a problem for PB cathode materials.⁸² Many researchers tried many ways to improve this situation of PB cathode material, while presodiation can incubate high quality CEI in a convenient way among these strategies. Ye et al.⁸³ in-situ constructed a NaF-rich CEI on the surface of PB cathode by chemical presodiation. The PB cathode was immersed in Na-Bp/DME solution and then the CEI was in-situ constructed by spontaneous redox of sodium metal and PVDF binder. This process could be achieved by relying on the reaction between cathode and presodiation solution's

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**Table 4.** Presodiation methods and electrochemical performance of transition metal oxides for SIBs.

<table>
<thead>
<tr>
<th>Electrode materials</th>
<th>Pre-sodiation methods/Additives</th>
<th>Energy density (Wh kg⁻¹)</th>
<th>1st charge/discharge capacity (mAh g⁻¹)</th>
<th>Cyclability</th>
<th>ICE</th>
<th>Electrolyte, Voltage range (V vs. Na⁺/Na)</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>P₂-NaₓFe₂O₄Fe₀₃MnₓO₂O₂</td>
<td>DC —</td>
<td>~200/160</td>
<td>99% after 40 cycles at 0.1 C (full cell)</td>
<td>—</td>
<td>1 M NaClO₄, PC/DEC (98:2), 1.5–4.3 V</td>
<td>—</td>
<td>[14]</td>
</tr>
<tr>
<td>P₂-type NaₓFe₀₃(OH)ₓFe₀₃(TiO)₂</td>
<td>EP 542</td>
<td>220(discharge)</td>
<td>81% after 500 cycles at 260 mA g⁻¹</td>
<td>—</td>
<td>0.5 M NaPF₆, PC/EC (98:2), 1.4–4.2 V</td>
<td>—</td>
<td>[52]</td>
</tr>
<tr>
<td>P₃-Naₓ[Feₓ,MnₓO₄]NaNₓO₂</td>
<td>EP</td>
<td>~155/155 (full cell)</td>
<td>81% after 200 cycles at 25 mA g⁻¹</td>
<td>—</td>
<td>0.5 M NaPF₆, PC/EC (98:2), 1.9–4.4 V</td>
<td>—</td>
<td>[53]</td>
</tr>
<tr>
<td>Naₓ₂MnO₂</td>
<td>CSP —</td>
<td>115.7/110.9</td>
<td>94.1% after 200 cycles at 2 C</td>
<td>—</td>
<td>1 M NaClO₄, EC/DEC + 5% FEC, 2–4 V</td>
<td>—</td>
<td>[54]</td>
</tr>
<tr>
<td>Naₓ₄(Feₓ,Mnₓ)O₂</td>
<td>CSP —</td>
<td>133/128</td>
<td>92.4% after 300 cycles at 1 C</td>
<td>—</td>
<td>1 M NaClO₄, PC/EC + 5% FEC, 2–4 V</td>
<td>—</td>
<td>[55]</td>
</tr>
<tr>
<td>NaCO₃</td>
<td>NaₓNiO₂</td>
<td>132.1/107.5</td>
<td>58.3 mAh g⁻¹ after 30 cycles at 2 C</td>
<td>81.4%</td>
<td>1 M NaClO₄, EC/DEC/FEC (4:3:4:3), 2–3.6 V</td>
<td>—</td>
<td>[56]</td>
</tr>
<tr>
<td>P₂-Naₓ[Feₓ(Mnₓ)O₄]O₂</td>
<td>NaₓP —</td>
<td>247/155</td>
<td>~85% after 20 cycles at 0.1 C</td>
<td>—</td>
<td>1 M NaClO₄, EC/DMC, 0–4.5 V</td>
<td>—</td>
<td>[57]</td>
</tr>
<tr>
<td>P₂-Naₓ[Feₓ(Mnₓ)O₄]O₂</td>
<td>NaₓC₀O₃</td>
<td>172.6 (vs. 129.2)</td>
<td>85% after 200 cycles at 1 C (full cell)</td>
<td>76%</td>
<td>—</td>
<td>—</td>
<td>[63]</td>
</tr>
<tr>
<td>P₂-Naₓ[Feₓ(Mnₓ)O₄]O₂</td>
<td>NaₓO₂</td>
<td>175</td>
<td>~221/115</td>
<td>96.8% after 100 cycles at 0.1 C</td>
<td>—</td>
<td>1 M NaPF₆, PC/EC/DEC + 5% FEC, 2.5–4.5 V</td>
<td>—</td>
</tr>
<tr>
<td>O₃-Naₓ</td>
<td>NaₓO</td>
<td>~112/80 (full cell)</td>
<td>74% after 100 cycles at 0.5 C</td>
<td>90%</td>
<td>—</td>
<td>1 M NaClO₄, EC/DEC + 5 vol% FEC, 1–4.2 V</td>
<td>—</td>
</tr>
<tr>
<td>Liₓ[Naₓ(Mnₓ)O₄]</td>
<td>NaNO₂</td>
<td>~210/217</td>
<td>98%</td>
<td>0.5 M NaPF₆, PC/EC (98:2), 1.5–4.3 V</td>
<td>—</td>
<td>—</td>
<td>—</td>
</tr>
<tr>
<td>P₂-Naₓ[Feₓ(Mnₓ)O₄]O₂</td>
<td>EDTA–4Na</td>
<td>~175</td>
<td>92% after 20 cycles (vs. 50%)</td>
<td>99%</td>
<td>0.5 M NaPF₆, PC/EC (98:2), 2–4 V</td>
<td>—</td>
<td>[67]</td>
</tr>
<tr>
<td>Naₓ₂MnO₂</td>
<td>DTPA–5Na —</td>
<td>~280/245</td>
<td>83% after 500 cycles at 0.2 C (full cell)</td>
<td>96%</td>
<td>—</td>
<td>—</td>
<td>[68]</td>
</tr>
</tbody>
</table>

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active Na⁺. The CEI@PB|Na cell displays excellent cycle performance with a capacity retention of 68.9% after 3000 cycles. This work has greatly increased the feasibility of commercial PB materials for large-scale application with an easy method.

Summary and Perspective

In this review, we summarize the presodiation strategies and corresponding electrochemical performance of electrode materials in SIBs. The electrode materials involved in presodiation are divided into anode materials and cathode materials, which are subdivided into intercalation-type materials (HC, SC, rGO, Ti-based, etc.), conversion-type materials (Fe₆N, SnS₂, Co₃O₄, etc.), alloying-type materials (Na-Sn, Na-Sb, Na-P, etc.), transition metal oxide materials (NaₓFeₓOₓMnxO₂, Naₓ-xMnxO₂, NaCrO₂), polyanionic compound materials (Na₃Vₓ(PO₄)ₙ, Na₃VₓOₓ(PO₄)ₓF) and prussian blue materials. Based on the properties and defects of electrode materials, adopting suitable presodiation strategy could maximize the cycling-life and capacity effectively from direct contact with sodium metal, electrochemical presodiation, chemical synthesis presodiation and adding additives with active Na⁺. Herein, we analyze the most effective presodiation for each material. Intercalation-type and alloying-type materials are more suitable for chemical synthesis presodiation due to their higher requirements for interphase after supplementing sodium ions, which allows for an efficient combination for both of them. Choosing direct contact method is also a good choice for alloying-type materials, because the metal anodes are easier to undergo a thermodynamic reaction spontaneously with the sodium metal. Conversion-type materials are better suited to electrochemical presodiation because of the sensitive relationship between capacity and cycling-life to conversion potential. From the perspective

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**Table 5.** Presodiation methods and electrochemical performance of polyanionic compounds for SIBs.

<table>
<thead>
<tr>
<th>Electrode materials</th>
<th>Presodiation methods/Additives</th>
<th>Energy density (Wh kg⁻¹)</th>
<th>1st charge/discharge capacity (mAh g⁻¹)</th>
<th>Cyclability</th>
<th>ICE</th>
<th>Electrolyte, Voltage range (V vs. Na⁺/Na)</th>
<th>Refs</th>
</tr>
</thead>
<tbody>
<tr>
<td>Na₃V₃(PO₄)₃</td>
<td>EP</td>
<td>265</td>
<td>~125/110</td>
<td>98% after 200 cycles at 1 C</td>
<td>95%</td>
<td>1 M NaPF₆, EC/DEC, 2–4.3 V</td>
<td>[72]</td>
</tr>
<tr>
<td>Na₃V₂(PO₄)₃</td>
<td>DC</td>
<td>400</td>
<td>~225/160</td>
<td>93% after 130 cycles at 2 C</td>
<td>---</td>
<td>1 M NaPF₆, diglyme, 1.0–3.8 V</td>
<td>[73]</td>
</tr>
<tr>
<td>Na₃V₃(PO₄)₃</td>
<td>Na₃S/C</td>
<td>263</td>
<td>~130/105</td>
<td>300 cycles without an obvious capacity degradation</td>
<td>---</td>
<td>1 M NaClO₄, EC/DEC + 5 vol% FEC, 2.5–4 V</td>
<td>[74]</td>
</tr>
<tr>
<td>Na₃V₂O₃(PO₄)₂F</td>
<td>NaCrO₂</td>
<td>201.5 (vs. 122.2)</td>
<td>~252/130</td>
<td>80% after 50 cycles at 0.5 C (full cell)</td>
<td>---</td>
<td>1 M NaClO₄, EC/DEC + 5 w% FEC, 2–4.2 V</td>
<td>[75]</td>
</tr>
<tr>
<td>Na₃V₂(PO₄)₃F</td>
<td>Na₃CeO₃</td>
<td>210.8 (vs. 154.5)</td>
<td>~180/115</td>
<td>95.7% after 100 cycles at 20 mA g⁻¹</td>
<td>---</td>
<td>1 M NaClO₄, PC + 5% FEC, 2–4.3 V</td>
<td>[76]</td>
</tr>
<tr>
<td>Na₃V₂(PO₄)₃/rGO</td>
<td>Sodium citrate</td>
<td>241 (vs. 187)</td>
<td>~180/110</td>
<td>~92.7% mAhg⁻¹ after 50 cycles at 50 mA g⁻¹</td>
<td>---</td>
<td>1 M NaClO₄, PC + 5 vol% FEC, 2–4.4 V</td>
<td>[78]</td>
</tr>
</tbody>
</table>

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of industrial application, if there are suitable and harmless additives, adding additives is the most convenient and efficient method for cathode. In addition to this, taking into account the structure of each cathode material, the P2-type layered materials require presodiation that can effectively add active Na\(^{+}\) to compensate for its sodium-deficient structure, while polyanionic compounds are more suitable for electrochemical presodiation in order to control the de/sodiation in the range of voltage precisely. Prussian blues are more inclined to chemical synthesis presodiation due to their requirement for high-quality interphase. Meanwhile, in order to better advance the research of presodiation, the following aspects can be promoted:

(1) Considering the high reactivity of sodium, lower-cost, safer and smaller-size sodium alloy additives should be developed as soon as possible to compensate the irreversible sodium losses in the initial cycle efficiently.

(2) Electrochemical presodiation could achieve large-scale application through adopting a continuous roll-to-roll process in the future. To carry forward the advantage of controlling the degree of presodiation precisely, it is feasible to import an external circuit and add electrolyte additives. In addition, it is vital to keep the presodiated electrode stable in air.

(3) The challenge of chemical synthesis presodiation lies on exploring more active sodium reagent with a sufficiently low redox potential to presodiate wide variety of electrode materials. It should be also taken thought about the compatibility between chemical reagents and electrode binders. Meanwhile, the electrode reacted with chemical reagents also need to improve the air stability.

(4) More attention should be paid to investigate whether the additives can eliminate the effect of used leftovers on subsequent electrochemical performance and stop generating gas during cycles. Additionally, it is necessary to comprehensively evaluate the presodiation strategy in combination with the specific full cells.

Furthermore, we propose prospects and suggestions for the future development of presodiation technology from aspects of manufacture, characterization, equipment, recycling and high-throughput screening in following part. The summary and prospects for the presodiation technology in sodium-ion batteries are illustrated in Figure 7.

(1) To select well-fit electrode materials precisely in SIBs, it should combine the factors of application environment and specific electrolyte in system before presodiation. For example, polyanionic compounds are applied in cold environment as cathode materials mostly because of good thermal stability. And alloying-type materials should not be selected to apply in aqueous electrolyte systems as the anodes. To address the intrinsic defects of various electrode materials, corresponding strategies should be taken to enhance the performance of electrode in conjunction with presodiation, such as NVP with poor electronic conductivity, which can be doped with carbon materials with good conductivity while adopting presodiation. It is vital to design presodiated electrode materials which can meet the requirements of market for large-scale manufacture.

(2) In order to manufacture presodiated electrode materials for large-scale, it is also essential to design process control equipments of presodiation. Process control equipments can monitor the state of the electrode materials in the presodiation process and feedback to the terminal equipments to adjust in real time, which could control the degree of presodi-
ation automatically and accurately and address the safety issues better.

(3) At present, there is no characterization technology that can reflect the presodiation process better. Scientific researches on presodiation are always based on the final results of electrochemical performance and electron microscopy images, which greatly slow down the research progress of presodiation. So there is an urgent need to design characterization techniques that can reflect presodiation influence directly or indirectly based on existing electrochemical theory.

(4) The establishment of recycling presodiated materials in SiBs at scale has great strategic significance with large-scale application in the future. The recycling of presodiated electrode materials not only alleviates the constraints imposed by raw material scarcity and enhances environmental sustainability, but also supports a safer and more resilient raw material supply chain. Meanwhile, they are inherently different from other common electrode materials and have a higher added value for recycling.

(5) With the development of high-throughput computing based on DFT theory, high-throughput screening has played an increasingly important role in the exploration of emerging materials in recent years. In the field of presodiation, new solvents for chemical synthesis presodiation and active sodium additives applied in cathodes can be developed by calculating the HOMO-LUMO energy level of solvents, additives and electrolyte systems. At the same time, combining the structure of electrodes and properties of the electrode-electrolyte interface can explore suitable new presodiated electrodes with high-throughput screening technology. A large number of chemical reagents meeting the basic parameter requirements will be selected by high throughput screening, from which some reagents will be sent to do higher-order parameter refinement and characterization, and the correlation between characterization results and calculated data will be analyzed by machine learning, providing a reliable guideline for the development of finding new presodiation reagents.

■ ACKNOWLEDGEMENTS

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■ CONFLICT OF INTEREST

The authors declare no conflict of interest.

■ AUTHOR'S CONTRIBUTIONS

Jiacheng Chen mainly wrote the manuscript and conducted the figures; Xindan Li assisted in the writing of original manuscript and drew the table of contents; Liwei Mi gave suggestions in the process of revising manuscript and polished the manuscript; Weihua Chen guided the direction and led the whole process of writing. All authors had approved the final version.

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