Supporting Information

An Efficient Rechargeable Aluminium–Amine Battery Working Under Quaternization Chemistry

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Experimental Section/Methods

Materials: Triphenylamine (N1, 98%) was ordered from Alfa Aesar. 1,4-bis(diphenylamino)benzene (N2, 97%) and 1,3,5-tris(diphenylamino)benzene (N3, 97%) were purchased from Sigma Aldrich. 4,4',4''-tris(diphenylamino)triphenylamine (N4, >97%) and 4,4',4''-tri-9-carbazolyltriphenylamine (N4', >98%) were procured from Tokyo Chemical Industry. 1-Ethyl-3-methylimidazolium chloride ([EMIm]Cl, 97%) was product of Acros Chemicals. Alginic acid sodium salt (Alg, medium viscosity) was purchased from Sigma Aldrich. Super P carbon additive, glass fiber (GF/C), tungsten foil (0.05mm, 99.95%) and AlCl3 (99.999%) were bought from Alfa Aesar. Carbon cloth (CC, ELAT) was purchased from FuelCellStore. All the chemicals were used directly without further purification.

Preparation of Al electrolyte and amine electrodes: The Al electrolyte was prepared in an Ar-filled glove box with H2O and O2 content below 0.1 ppm. AlCl3 was mixed slowly with [EMIm]Cl in a specific molar ratio (1:1, 1.3:1 or 2:1) and the resultant electrolyte was denoted as IL-x, where x represents the molar ratio (1, 1.3 or 2). The mixture was stirred in the glove box for another 5 h, producing transparent liquid. Amine electrodes were prepared according to the following procedures. Amine power (N1, N2, N3, N4 and N4') was mixed with Super P and Alg binder in a weight ratio of 8:1:1 with water as the solvent. The resultant slurry was casted onto a hydrophilic carbon cloth or W foil (current collector). The amine electrodes were dried at 80°C for 8 h under vacuum. The amine loading was controlled in the range of 3.5~4 mg cm⁻². The electrode thickness under a N4 loading of 4 mg cm⁻² was determined as ~220 µm. In the high loading measurement, the amine loading was further increased to 6 and 12.9 mg cm⁻². For the pouch cell demonstration, the electrode calendering was applied for high electrode density with thin W foil as the current collector.

Material characterization: the (N4)ₙ samples at different charging states (charged or discharged) or after continuous charge-discharge cycling test were disassembled from Swagelok cells in the glove box followed by rinse with substantial extra dry methanol (99.8%, via molecular sieve) and vacuum drying (~20min). The (N4)ₙ samples then were stored in glove box before different characterizations. The mass spectrometry analysis was performed on a Bruker Autoflex Speed MALDI-TOF MS (Bruker Daltonics, Bremen, Germany) using trans-2-[3-(4-tert-butylphenyl)-2-methyl-2-propenylidene]malononitrile as matrix. N4 molecule was first extracted by dissolving fresh N4 electrode into dichloromethane for the measurement, while (N4)ₙ electrode was directly mixed with matrix for the measurement because (N4)ₙ is not
soluble in dichloromethane. \( \text{N}4' \) molecule was subjected to a similar procedure to \( \text{N}4 \) electrode because \( \text{N}4' \) in \( \text{N}4' \) electrode before or after cycling can be totally extracted by dichloromethane. FTIR spectra were recorded on theBruker Optics ALPHA-E spectrometer with a universal Zn-Se attenuated total reflection accessory in the 600–4000 cm\(^{-1}\) regime. The XRD patterns were collected on a PANalytical X’Pert Pro diffractometer with Cu-K\(\alpha\) radiation. SEM images and elemental mapping images were recorded on a field-emission scanning electron microscope Zeiss Gemini 500 equipped with an Oxford X-max\(^\text{N}\)-150 EDX detector. The charged and discharged \((\text{N}4)_n\) samples were shifted from glove box to SEM chamber under short air exposure (5-10s). \(^{27}\text{Al}\) NMR spectra of liquid electrolyte was acquired on sealed samples at 78MHz (7 T, 300MHz \(\text{H}1\)) using Bruker Avance I spectrometer equipped with HR BBI 2-channel 5mm NMR probe. Chemical shift referencing is to \(\text{Al}^{3+}(\text{H}_2\text{O})_6\text{Cl}_3\) \(\delta^{27}\text{Al} \) (ppm) = 0. Solid-state \(^{27}\text{Al}\) NMR spectra was acquired at 130MHz (500MHz \(\text{H}1\)) using Bruker Avance III spectrometer equipped with MAS 3-channel 1.9mm NMR probe, MAS rate was 25 kHz. Chemical shift is secondary referenced to Gibbsite \(\delta^{27}\text{Al} \) (ppm) = 11. The charged \((\text{N}4)_n\) sample after wash and dry was scratched off substrate (carbon cloth or W foil) and loaded into the NMR probe in glove box.

**Electrochemical characterization:** Two-electrode Swagelok cells with W rod were assembled in glove box to evaluate the electrochemical performance of \(\text{Al-amine} \) batteries. A circular amine electrode (1 cm diameter) was stacked with a glass fiber separator and \(\text{Al} \) foil anode, and then wetted by \(\text{Al} \) ionic liquid electrolyte. A steel spacer and spring were added afterward and sealed well for electrochemical measurement. The pouch cell was also assembled in glove box with a piece of amine cathode, a glass fiber and an \(\text{Al} \) foil. After injection of \(\text{Al} \) ionic liquid electrolyte, the pouch cell was sealed under vacuum. The galvanostatic charge-discharge measurements were carried out on a LAND CT2001A battery test system at room temperature. The specific capacity and current densities were calculated on the amine mass in cathode. A voltage window of 0.2–1.85 V was used. In the high loading case (12.9 mg cm\(^{-2}\)), the voltage window was extended to 0.1–2 V to compensate the polarization. The CV measurements were carried out on the CHI 760E electrochemical workstation at a scan rate of 1 mV s\(^{-1}\). The electrochemical impedance spectra were performed at a 10 mV ac oscillation amplitude in the frequency range of 100 kHz to 1 Hz. GITT experiment was performed in Al-(\(\text{N}4\))\(_n\) battery with a galvanostatic charge pulse current of 0.1 A g\(^{-1}\) for 5 min followed by an open-circuit relaxation of 2h.
In situ electron spin resonance (ESR)/ultraviolet–visible–near infrared (UV–vis–NIR) absorption spectroelectrochemistry: The spectroelectrochemical experiments were performed in the optical ESR cavity (ER 4104OR, Bruker Germany). ESR spectra were recorded by the EMX X-band CW spectrometer (Bruker, Germany). An NMR teslameter (ER036TM, Bruker, Germany) was used for precise g value determination. Variable-temperature EPR measurements were performed using an integrated variable temperature controller. The UV–vis–NIR spectra were measured using the Avantes spectrometer AvaSpec-2048×14-USB2 with the CCD detector and AvaSpec-NIR256-2.2 with the InGaAs detector applying the AvaSoft 7.5 software (Avantes, The Netherlands). A light source Avantes Avalight-DH-S-BAL was used. Both, the ESR spectrometer and the UV–vis–NIR spectrometer are linked to a HEKA potentiostat PG 390 which triggers both spectrometers. Triggering is performed by the software package PotMaster v2 × 90 (HEKA Electronik, Germany). Each UV–vis–NIR spectrum was collected relative to that of the neutral (uncharged) compound at the initial potential. For standard in situ ESR/UV–vis–NIR spectroelectrochemical experiments, a three-electrode EPR flat cell was used. A laminated gold mesh (Goodfellow, UK) as working electrode, an AgCl-coated silver wire as pseudo reference electrode, and a platinum wire as counter electrode were used in spectroelectrochemical experiments. All potentials are given versus Fe/Fe²⁺ redox couple as an internal standard in the spectroelectrochemical experiments. 0.1 M TBAPF₆ in dichloromethane was used as a supporting electrolyte. The cell assembling and the measurements were done under an inert (nitrogen) atmosphere.

Self-discharge investigation: The self-discharge behavior of Al-amine batteries was investigated by subjecting the fully charged Al-amine battery at open circuit for specific time (0~168h) under different temperature (-25~100 °C). The charge current is 0.1 A g⁻¹. After the standing period under specific temperature, the Al-amine battery was fully discharged at a current of 0.1 A g⁻¹ under the same temperature. The residue discharge capacity is collected and compared with the charge capacity.

DFT calculation: A density functional quantum simulation of N₄ was implemented with the Gaussian16 software package.[¹] The geometry optimizations for N₄ monomer, trimer and heptamer samples were carried out with Becke's three-parameter hybrid exchange functional and the Lee–Yang–Parr correlation functional (B3LYP) and the 6-311G (d,p) basis set. The redox processes of N₄ building block with 1~4 electrons were also simulated through closed-shell and open-shell structural calculation of N₄ using unconstrained B3LYP and 6-311G (d,p)
basis ser. Besides, HOMO, LUMO and SOMO mapping of all the systems were visualized and exported with GaussView 6.0 software. The state energy of open-shell (dication diradical) $\text{N}_4^2(\ast\ast)$ and close-shell (quinone diiminium) $\text{N}_4^2\ast$ was calculated using a (U/R) B3LYP/6-31G(d) method.
Figure S1. Photographs of Swagelok cell and accessories (W rod, stainless steel (SS) rod, spacer, spring, sealing rings et al).
Figure S2. Cyclic voltammetry (CV) curves of Al ionic liquid (IL-1.3) on W electrode. The scan rate is 1 mV s$^{-1}$. The IL-1.3 showed a maximum voltage window of 2.6 V.
Figure S3. CV curves of carbon cloth (CC), tungsten (W) foil current collector, Super P loaded on CC and super P loaded on W foil. Alg-Na binder was used to prepare super P electrodes (super P: Alg = 80:20 in mass ratio). The loading of Super P is 2 mg cm\(^{-2}\). The scan rate is 1 mV s\(^{-1}\).
Figure S4. Charge-discharge profiles and CE of amine electrodes. (A-C) Charge-discharge curves of N1, N2 and N3 electrodes at different cycles. (D) CE evolution of N4 electrodes during cycling.
Figure S5. Photograph of the separators of N1, N2, N3 and N4 cells after cycling performance in Figure 2A.
Figure S6. SEM images of N4 electrode before (A, B) and (C, D) after cycling test. Insets are photographs of the corresponding N4 electrodes.
Figure S7. MALDI-TOF mass spectrum of pristine N4.
Figure S8. MALDI-TOF mass spectrum of alginate sodium binder.
Figure S9. Calculation of the relative energies in vacuum and optimized structures of (N4)$_n$ used in the study obtained using DFT method.

The redox potential of organic compounds closely correlates with energy levels of the lowest unoccupied molecular orbital (LUMO) and the highest occupied molecular orbital (HOMO) and can be quantified by HOMO energy in an oxidation reaction, while LUMO energy in a reduction reaction. In order to reduce the complexity of calculation, the LUMO-HOMO of N4 monomer, trimer ((N4)$_3$) and heptamer ((N4)$_7$) instead of all the oligomers was calculated using density functional theory (DFT). As shown in Figure S9, the HOMO of (N4)$_3$ (-4.56 eV) and (N4)$_7$ (-4.32 eV) was higher than that of monomer (-4.63 eV), indicating that (N4)$_3$ and (N4)$_7$ have a smaller ionization potential and lower oxidation potential than the N4 monomer. This agrees well with the experiment results shown in Figure 2C. In addition, the gap between the HOMO and LUMO (3.80, 3.47 and 0.56 eV for N4, (N4)$_2$ and (N4)$_7$), represented as $\Delta E$ and related to the electronic conduction, are reduced due to better $\pi$-electron conjugation in the oligomers.
Figure S10. Characterization of cycled (N4)n electrode. (A) FTIR spectra. (B) X-ray diffraction (XRD) pattern (b) comparison of pristine and cycled (N4)n electrode. XRD measurement suggests that the formed (N4)n possesses an amorphous structure lacking long-range order.
Figure S11. Electrochemical behavior and characterization of N4'. (A) Cycling performance of N4’ cells at 0.1 A g⁻¹. (B) Charge-discharge curves of Al-N4’ battery. (C) CV curves of N4’. (D) MALDI-TOF mass spectroscopy of pristine and cycled N4’.
**Figure S12.** EDX mapping of the charged (N4)$_n$ electrode.
Figure S13. SEC measurements of N4 in 0.1 M TBAPF₆ dichloromethane electrolyte. (A) CV curves of N4 within a two-electron oxidation. The scan rate ranges from 0.01-1 V s⁻¹. Inset is dependence of the peak current (first oxidation) on the square root of the scan rate. (B) Evolution of ESR spectra as a function of potential (-0.43 to 0.46 V). (C) UV–vis–NIR spectra of N4 under different potentials. (D) Intensity variation of ESR signal and adsorption band at 440 nm as a function of potential. (E) The proposed reaction process of N4 under a two-electron oxidation. Electrolyte is 0.1 M TBAPF₆ dichloromethane electrolyte.
The peak currents are proportional to the square root of the scan rate (Figure S13A), suggesting that the fast two-electron oxidation of N4 is controlled by mass transfer. The in situ UV–vis–NIR spectra of N4 measured during the first oxidation show the absorption bands at 440 and 1115 nm. The first band shows the same intensity variation trend as the ESR signal intensity during CV scans (Figure S13C, D). Both absorption bands can be attributed to the electronic transition of the N4++. The band at 1115 nm blue shifts and a new band at 540 nm appears during the second oxidation. These features can be associated with the formation of N4++. 
Figure S14. Calculation of the open-shell (dication diradical N4\(^{2(+)}\)) and closed-shell (quinone diiminium N4\(^{2+}\)) state energy. The calculation method used is (U/R) B3LYP/6-31G(d). Closed-shell N4\(^{2+}\) shows a lower energy than open-shell N4\(^{2(+)}\) counterpart, suggesting higher stability of closed-shell N4\(^{2+}\).
Figure S15. Bond length information of N4^{2+}. Optimized structure starting from open-shell (N4^{2+}) and closed-shell (N4^{2+}) states shows the same bond information, where C_{26}-C_{27} (1.37511 Å) and C_{25}-C_{26} (1.4256 Å) bonds both are present in the inner ring and exhibit the inherent characteristics of the quinone structure. It indicates transformation of open-shell N4^{2+} to closed-shell N4^{2+} (quinone diiminium). So quinone diiminium is the most stable form of N4^{2+}. 
Figure S16. CV curves of N4 in 0.1 M TBAPF₆ dichloromethane electrolyte. The scan rate is 0.05 V s⁻¹. The reactions are controlled to be three (black) and four electrons (red), respectively.
Figure S17. Evolution of ESR spectra as a function of potential (-0.5 to 1.2 V).
Figure S18. Oligomerization initiated by N4 radical trication species. The formed H⁺ may exist in the form of HCl in the electrolyte, which can react further with Al metal to produce AlCl₃ and H₂.
Figure S19. ESR signal of cycled (N4)$_n$ electrode that charged to 0.7 V (vs Al).
Figure S20. Electrochemical impedance spectrum (EIS) of Al-(N4)n battery at different SOC. (A) EIS during charging. (B) EIS during discharge. (C) Contact resistance variation during charging and discharging. IL-1.3 was used.

The intersection of Nyquist plots with x axis at \( Z'' = 0 \) is Ohmic resistance (\( R_s \)) from the electrolyte and electrodes.
Figure S21. EIS of Al-(N4)$_n$ battery at different SOC. (A) EIS during charging. (B) EIS during discharge. (C) Contact resistance variation during charging and discharging. IL-2 was used.

To exclude the effect of variable Al electrolyte composition on the conductivity during battery operation, where the maximum ionic conductivity was obtained at IL-1.2,$^{[3]}$ excess IL-2 was used so that charging process would not significantly change the electrolyte composition. However, similar $R_s$ variation was noticed (Figure S22C).
Figure S22. Proposed conductivity mechanism for (N4)$_n$ electrode during oxidation.
Figure S23. GITT characterization of the \((\text{N}4)_n\) cathode at a current density of 0.1 A g\(^{-1}\). The discharge capacity is a little higher than charge capacity, which is due to self-recharge function of Al-(\text{N}4)_n battery.
Figure S24. Charge-discharge curves of Al-(N4)$_n$ battery at different current rates.
Figure S25. Charge-discharge curves of Al-(N4)$_n$ battery in IL-2. (A) Cycling performance at 0.1 A g$^{-1}$. The voltage windows used are 0.2~1.85 V, 0.1~1.8 V and 0.05~1.8 V for the first cycle, second cycle and other cycles, respectively. (B) Charge-discharge curves at different cycles. (C) Comparison of electrochemical performance of Al-(N4)$_n$ battery in IL-1.3 and IL-2. The current rate is 0.1 A g$^{-1}$. (D) Self-discharge test of Al-(N4)$_n$ battery in IL-2. The battery was first fully charged at 0.1 A g$^{-1}$, followed by standing at open circuit for 10h, 1 day and 2 days, respectively. Then the battery was fully discharged at 0.1 A g$^{-1}$.

The voltage window changing was due to the fact that IL-2 electrolyte decreased the working voltage of Al-(N4)$_n$ cell as compared to IL-1.3 (1.05 V vs 1.1 V), similar to Al-graphite battery[4]. To exert all the capacity of (N4)$_n$, we downshift the voltage window of Al-(N4)$_n$ cell in IL-2.
Figure S26. Self-discharge test of Al-(N4)$_n$ battery under different temperatures (from -25 to 100 °C). The battery was first fully charged at 0.1 A g$^{-1}$, followed by standing at open circuit for 10h, 1 day and 2 days, respectively. Then the battery was fully discharged at 0.1 A g$^{-1}$. IL-1.3 was used.

It was noticed that high potential reactions appeared for Al-(N4)$_n$ cell at 75 and 100°C. This is not well understood at the current stage. The result may relates to physiochemical properties change of IL electrolyte and also structure evolution of amine cathodes at high temperature. More investigation is still needed here.
Figure S27. Self-discharge behavior of Al-N4 battery at room temperature. The battery was first fully charged at 0.1 A g⁻¹, followed by standing at open circuit for 10h, 1 day and 2 days, respectively. Then the battery was fully discharged at 0.1 A g⁻¹. IL-1.3 was used.
Figure S28. Self-discharge test of Al-graphite battery under different temperatures (from -25 to 75 °C). The standing time at open circuit is 10 h, 1 day and 2 days, respectively. Before the discharge measurement, the battery was first fully charged. The current was 0.1 A g⁻¹. A voltage window of 0.5-2.45 V was used at 25 °C and below, while 0.5-2.39 V and 0.5-2.33 V were applied at 50 and 75 °C to suppress side reaction at high voltage. IL-1.3 was used.
Table S1. Theoretical capacity comparison of amine compounds based on one electron transfer per N center.

| Amine compounds | Molecular weight | Theoretical capacity (mAh g\(^{-1}\)) |
|-----------------|------------------|--------------------------------------|
| N1              | 245.32           | 109.2                                |
| N2              | 412.52           | 130.0                                |
| N3              | 579.73           | 138.7                                |
| N4              | 746.96           | 143.5                                |
### Table S2. Cycling stability comparison of rechargeable Al batteries.

| Battery            | Capacity retention                                      | Reference                                      |
|--------------------|---------------------------------------------------------|------------------------------------------------|
| Al-(N4)$_m$        | 93.5% after 4000 cycles at 1 A g$^{-1}$                | This work                                     |
| Al-Aquinone        | 59% after 5000 cycles at 2 A g$^{-1}$                  | Nat. Energy 2018, 4, 51.                       |
| Al-tetradiketone   | 73% after 300 cycles at 0.1 A g$^{-1}$                 | Nat. Commun. 2021, 12, 2386.                  |
|                    | 76% after 8000 cycles at 1 A g$^{-1}$                  |                                                |
| Al-PAQS            | <75% after 500 cycles at 0.5 C                          | Energy Storage Materials 2020, 24, 379.        |
| Al-polypyrene      | 53% after 1000 cycles at 0.2 A g$^{-1}$                | Adv. Mater. 2018, 30, 1705644                  |
Energy Density Calculation

The energy density of Al-amine batteries were calculated based on the mass of active materials and electrolyte. Due to the fact that Al is deposited during charging\[^5\] and Al plating/stripping is quite efficient in Al ionic liquid,\[^6\] Al-free configuration has been demonstrated.\[^7\] So the mass of Al anode was not considered during energy density calculation of Al-(N4)\(_n\) battery.

The reactions occurred in Al-(N4)\(_n\) battery are as follows:

\[
\begin{array}{|c|c|}
\hline
\text{Electrode} & \text{Reaction} \\
\hline
\text{Anode} & 4\text{AlCl}_{3}^+ + 3\text{e}^- \rightarrow 7\text{AlCl}^+_4 + \text{Al} \\
\text{Cathode} & 4\text{AlCl}^+_4 + \text{N4} \rightarrow \text{N4(AlCl)}_{4} + 4\text{e}^- \\
\hline
\text{Overall-1} & 16\text{AlCl}_{3}^+ + 3\text{N4} \rightarrow 16\text{AlCl}^+_4 + 4\text{Al} + 3\text{N4(AlCl)}_{4} \\
\text{Overall-2} & 16\text{AlCl}_{3} + 3\text{N4} \rightarrow 4\text{Al} + 3\text{N4(AlCl)}_{4} \\
\hline
\end{array}
\]

According to the overall-2 reaction, the minimum mass of AlCl\(_3\) (\(m_{\text{AlCl}_3}\)) can be calculated as:

\[
m_{\text{AlCl}_3} = m_{\text{N4}} \div 746.95 \div 3 \times 16 \times 133.34 = 0.952m_{\text{N4}},
\]

where \(m_{\text{N4}}\) is the mass of N4.

In the Al ionic liquid, the mass ratio of active AlCl\(_3\) is related to electrolyte composition, which is denoted as the molar ratio of AlCl\(_3\)/EMImCl (1<n≤2). The mass ratio of active AlCl\(_3\) in electrolyte (wt\%) is calculated as:

\[
\text{wt}\% = \frac{(n - 1) \times 133.34}{n \times 133.34 + 1 \times 146.62}
\]

The minimum mass of ionic liquid electrolyte is:

\[
m_{\text{AlCl}_3+EMImCl} = m_{\text{AlCl}_3} \div \text{wt}\%
\]

Therefore, the energy density of Al-(N4)\(_n\) battery can be calculated based on the following formulas:

\[
\text{Energy density (wh/kg)} = \frac{C_C \times m_{\text{N4}} \times U}{m_{\text{N4}} + m_{\text{AlCl}_3+EMImCl}} = \frac{C_C \times U}{1 + \frac{m_{\text{AlCl}_3+EMImCl}}{m_{\text{N4}}}} = \frac{C_C \times U}{1 + \frac{m_{\text{AlCl}_3}}{m_{\text{N4}} \times \text{wt}\%}}
\]

Where \(C_C\), and \(U\) represent capacity of cathode and average working voltage.

In IL-2, the \(C_C\), and \(U\) are 134.1 and 1.05 V. The energy density is 35.6 Wh/kg, which is higher than 27-33 Wh kg\(^{-1}\) of Al-graphite/graphene battery and lower than 50 Wh kg\(^{-1}\) of Al-phenanthrenequinone battery.\[^8\]
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