Rechargeable Zinc Air Batteries and Highly Improved Performance through Potassium Hydroxide Addition to the Molten Carbonate Eutectic Electrolyte

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The zinc air battery has been regarded as an efficient solution to renewable energy storage applications in the next generation. Zinc air chemistries are promising, though great challenges still remain to utilize their high energy, optimize efficiency and high discharge rate. Here, we demonstrate an improved zinc air battery by replacing NaOH, used in our previous study, with KOH as an additive to the molten Li0.87Na0.13K0.50CO3 eutectic electrolyte. Cycling tests showed a very stable performance through 150 charge-discharge cycles, exhibiting high coulombic efficiency (94%) and an average discharge potential of ∼1.08 V when charged at a constant current of 75 mA and discharged over a constant 1.00 V cutoff at 550 °C. Moreover, rate tests revealed a good performance even at high rates of cycling, maintaining a coulombic efficiency of over 90% while at 7.3°C. These results show marked improvements in the field of rechargeable zinc air batteries.

In modern industrialized society, the demand for electric energy is increasing at a rapid rate.1 Due to the consumption of fossil fuels to generate electricity, the issue of environmental pollution has attracted attention as a crisis of urgency.1–6 Renewable resources, such as wind and solar energy, have emerged as alternatives to fossil fuels.3–6 Nevertheless, there is still a need for a safe, reliable, and efficient energy storage method in order to optimize the conversion of energy in power plants.

Among existing approaches, the battery is the most likely to become a highly efficient solution for the next generation of renewable sources applications.7–9 Presently, metal air batteries are among the best candidates due to their high energy densities; specifically, zinc air batteries are noteworthy because of its safe performance, low production cost, etc.

Through decades of development, only primary zinc air cells have been implemented in a few fields, such as hearing aids, flash lights and alarm monitoring devices.10 Compared to primary zinc air batteries, electrically rechargeable zinc air batteries are much more attractive option.11 Unfortunately, these cells are trapped in the development stage because they still suffer from low cell voltage (∼1.0 V), low efficiency, and poor cycle performance, in addition to several technical problems with these cells, namely dendritic growth that causes morphology changes, corrosion and passivation of the zinc electrode during discharge, and low catalytic activity of the air electrode.12

While each of the three main components of the battery (the anode, the cathode, and the electrolyte) plays a vital part in the battery’s performance, the electrolyte might be the most important due to its role in the transport of ions throughout the cell. The conductivity of the electrolyte (which is inherently related to the resistance of the electrolyte), can be altered in order to tune the cell voltage of zinc air batteries.13

Alkaline aqueous solutions were adopted as the electrolyte very early on in the development of zinc air batteries. In most cases, 6–7 M KOH aqueous solutions are used to achieve the optimum ionic conductivity and reaction kinetics for both the oxygen reduction reaction (ORR) and the oxygen evolution reaction (OER).14 However, the open system still faces some unresolved problems, namely the reaction products with atmospheric CO2 and water decomposition, which leads to desiccation of the electrolyte.

Ionic liquids (ILs) are an attractive option for the replacement of conventional alkaline aqueous electrolytes; these liquids have negligible volatility, an extremely desirable property for electrolyte in air batteries,15 and are also non-flammable and extremely stable, making the battery much safer. Moreover, IL electrolytes have been shown to generate uniform, non-dendritic zinc depositions,16–20 while maintaining a small over potential for zinc redox chemistry.19 Although ILs could also provide wide electrochemical windows,21 their high viscosity may give rise to poor wetting of the air electrode and a quick voltage decrease during discharge of the zinc air battery.10

Recently, gel electrolytes were proposed as a replacement for liquid electrolytes. These electrolytes have many advantages over aqueous electrolytes, specifically that they do not leak, nor can they dry out in the same manner as an aqueous electrolyte. Also, they exhibit high ionic conductivity, good mechanical strength, as well as good electrochemical stability.22,23 However, these electrolytes still leave the electrode susceptible to dendrite formation.

We recently demonstrated a new kind of zinc air battery with a molten electrolyte consisting of Li0.87Na0.13K0.50CO3 eutectic with added NaOH.24 In our previous report, the foundation of a rechargeable zinc-air battery with a molten eutectic was established. At 550 °C, and under the aid of a nickel air cathode, the battery was charged at a constant current of 25 mA with an average charge voltage of 1.43 V. The battery could reach an average discharge voltage of 1.04 V and a reversible cycling coulombic efficiency above 90% when discharged with a constant load of 100 Ω to a cut off voltage of 0.8 V. However, the discharge process of the battery was divided into two voltage plateaus: a higher voltage plateau at ~1.06 V and a lower voltage plateau at ~0.82 V. The discharging capacity of the lower voltage plateau was as long as 33% of the total charging capacity, which strongly affects the performance of the zinc air battery.

In this paper, we substitute NaOH by KOH to significantly enhance the charge-discharge performance. The results showed that the lower voltage plateau was markedly shortened with merely no coulombic efficiency decay due to the addition of KOH. The battery exhibited a higher average discharge voltage of 1.08 V and a higher discharge rate capacity.
Materials and Methods

The chemicals and materials utilized for this study are as follows: lithium carbonate (Li2CO3, 97%), sodium carbonate (Na2CO3, anhydrous, 99.8%), potassium carbonate (K2CO3, anhydrous, 99.0%), potassium hydroxide (KOH, 85%), zinc oxide (ZnO, 99%), ammonium acetate (NH4Ac, 98%), 2 mm Ni wire (99.5%), Ni foil (99.95%), pure Pt foil (99.999%), 1.2 mm Fe wire (annealed), steel foil (316 steel), and pure alumina crucible (99.7%).

Cyclic voltammetry (CV) measurements were recorded using an electrochemical workstation (CS350, Wuhan Corr Test Instruments Co. Ltd., China). The working electrode was a 0.05 cm2 (0.2 cm × 0.25 cm) Pt foil, and a 5 cm2 (2 cm × 2.5 cm) piece of nickel foil was used as the counter electrode and reference electrode.

Powder X-ray diffraction (XRD) patterns were resolved by comparison to the library spectra of MDI Jade 5.0 to identify the phases of each sample using a Cu Ka source (Rigaku D/MAX-2200). Each sample was scanned from 2-theta 10° to 80°. The thermal decomposition of the ZnO and KOH mix was characterized by thermal analysis (PerkinElmer Diamond TG/DTA).

Morphologies and elemental analysis of the electrode were examined by a field emission scanning electron microscopy (FE-SEM, Zeiss Sigma) coupled with energy dispersive spectroscopy (EDS, Oxford Instruments X-Max). The samples were prepared for analysis by applying a gold sputtering treatment to the surface.

Charging polarization experiments of the batteries were carried out with a special configuration. The battery was assembled with two electrodes of vastly different sizes, the smaller of which (0.5 cm × 0.5 cm) was used as the working electrode. The larger electrode was twenty times the size of the working electrode, measuring 2 cm × 2.5 cm. The electrolytes were Li0.87Na0.63K0.50CO3 eutectic with either added NaOH or KOH. The current density ranged from 5 to 80 mA/cm2.

The battery configuration used a 4 cm2 steel foil cylinder 1 cm in height with a circumference of 4 cm, and as a counter electrode, a 20 cm2 nickel foil cylinder 2 cm in height with a circumference of 10 cm. The electrolyte consists of a molten Li0.37Na0.63K0.50CO3 eutectic with 3m (m = molal, moles per kilogram of electrolyte) KOH and 1m ZnO, and added together into an alumina crucible. The crucible was placed into the center of an 1800 W Band Heater and heated to 550 °C. Temperature was monitored by a thermocouple and controlled by a temperature controller (708P, YUDIAN automation technology Co. Ltd., China). When the electrolyte was heated to 550 °C, the electrodes are put into the electrolyte. The steel foil cylinder was located concentrically inside the nickel foil cylinder. Whereas the molten electrolyte covered the steel foil electrode, the immersed depth of the nickel foil cylinder in the molten salt was 1cm. The charge-discharge behaviors were recorded using a LANHE battery testing system (CT2001A, Wuhan LAND electronics Co. Ltd, China). Charging was carried out at 75 mA for 160 seconds. The battery was discharged over a 100 Ω load to a cut-off voltage of 0.8 V.

The discharge rate performance was carried out with different loads of 200 Ω, 150 Ω, 100 Ω, 80Ω, 40 Ω, and 20 Ω, and each load was used for 20 cycles. In each cycle, the battery was charged with a constant current of 75 mA for 160 seconds, kept in an open circuit for 60 seconds, and then discharged under a constant load to a cut-off voltage of 0.8 V.

Results and Discussion

In order to demonstrate the deposition of zinc in the molten electrolyte, electrolytic zinc deposition (charging) was carried out under a constant current of 100 mA for 4 hours at 550 °C. Steel foil (2 cm × 2.5 cm) was used as current collector, and nickel foil (2 cm × 2.5 cm) was used as a counter electrode. The charging curve is shown in Fig. 1. Following the charging, the steel foil was extracted from the electrolyte, cooled, and washed with water to remove the excess electrolyte. Then, the product on the current collector was treated with 15% NH4Ac solution for 30 minutes at 80 °C to dissolve the ZnO on the surface. The current collector with the product was flushed with deionized water before it was vacuum dried. After this treatment, the product was identified by EDS. Fig. 2b exhibited the uniform distribution of elemental zinc on the surface of the current collector. This implies that zinc can be deposited in molten Li0.87Na0.63K0.50CO3 eutectic with 3m KOH and 1m ZnO.

The morphology of the obtained zinc was observed by SEM. As shown in Figs. 3a and 3b, the zinc was distributed evenly on the surface of the current collector. It was very clear from Figs. 3c and 3d that the zinc exhibited a sheet-like appearance, being different from the...
molten KOH became transparent after about one hour at 550°C. We observed that ZnO dissolved gradually in molten KOH, and the ZnO and KOH at 550°C for two hours. In order to understand the chemistry of the battery, both ZnO and the mixture of ZnO and KOH are heated at 550°C for two hours. We observed that ZnO dissolved gradually in molten KOH, and the molten KOH became transparent after about one hour at 550°C. After the temperature dropped to room temperature, the mixture was ground down to fine powders. XRD analysis of the powder was confirmed by a powdered X-ray diffraction library. This implies that K2Zn3O4 was formed in the mixture of Li0.87Na0.63K0.50CO3 powder with 1mZnO and 3mKOH by TG/DTA of the mix of Li0.87Na0.63K0.50CO3 powders with 1mZnO and 3mKOH by electrolysis of potassium zinc oxide (K2Zn3O4), represented by Eq.2. It is suggested that the reactions involved in the battery are during charging, the K2Zn3O4 is converted to zinc metal and K2O, and O2, which releases to the air, while during discharging, zinc metal is electroreduction via Eq.2 in charging. The charge/discharge reactions occur as the combination of Eqs.2 and 3. Overall, this process can be summarized by Eq.4.

$$3\text{ZnO} + 4\text{KOH} = 2\text{K}_2\text{Zn}_3\text{O}_4 + \text{H}_2\text{O} \quad [1]$$

$$2\text{K}_2\text{Zn}_3\text{O}_4 \rightleftharpoons 6\text{Zn} + 3\text{O}_2 + 2\text{K}_2\text{O} \quad [2]$$

$$2\text{K}_2\text{Zn}_3\text{O}_4 + \text{H}_2\text{O} = 2\text{K}_2\text{ZnO}_4 + 2\text{Zn} + 3\text{O}_2 \quad [3]$$

$$\text{ZnO} \rightleftharpoons \text{Zn} + 1/2\text{O}_2 \quad [4]$$

Cyclic voltammetry (CV) was used to examine the reversibility of the Zn/Zn2+ redox reaction using electrolyte that consists of a molten Li0.87Na0.63K0.50CO3 eutectic with 3m KOH and 1m ZnO. Platinum foil (0.05 cm²) was used as the working electrode. The counter electrode was a 5 cm² nickel foil electrode which was also used as the reference electrode. CV measurements were recorded starting at 0 V, going down in the reduction section to −1.80 V at a sweep rate of 100 mV/second, as shown in Fig. 6. Starting at the potential of 0 V, to the negative side, a sharp peak current (peak A1) was observed at about −1.25 V. This peak could be attributed to the electrolysis of potassium zinc oxide (K2Zn3O4), represented by Eq.2. At −1.80 V, the cyclic voltammogram flips to the oxidation scan. In this section, an evident peak current (peak A1) was observed at about −0.39 V after three cycles. The peak A1 is attributed to the potassium zinc oxide formation process (Eq.2).
The relationship between current and voltage was measured in various electrolytes (either with added NaOH or added KOH) for the cathode against an oversized (∝ 20 surface area) anode, and vice versa, where the cathode was then oversized (∝ 20 surface area) compared to the anode. This provides a precise probe of the electrode efficiency, while avoiding the challenge of establishing a reproducible reference electrode for a three-electrode measurement in the molten carbonate medium. The charging process was conducted at the expected current density for three minutes to reach a steady state, and then the current density would be increased to the next step. As shown in the Fig. 7a, the charging polarization curves with an oversized steel anode barely changed with the addition of NaOH compared to the addition of KOH. The curves even coincided when the current densities exceeded 50 mA/cm², indicating that the various additions had negligible effects on the cathode. Conversely, Fig. 7b displays an obviously difference in the relationship between current density and voltage with an oversized nickel cathode when NaOH was added to the electrolyte vs KOH. With the addition of NaOH, the anode exhibited much higher charging potentials at similar current densities when compared with KOH, demonstrating that as current density increases, the charging potential increases much faster for an electrolyte containing NaOH than for one which instead uses KOH.

X-ray diffraction (XRD) was utilized to characterize the used air electrode. As shown in Fig. 8, the X-ray diffraction pattern of the cathode exhibited obvious differences before and after charge-discharge experiments. The X-ray diffraction pattern of the fresh air electrode showed three nickel peaks at 44.7°, 52.1°, and 76.6°, as shown in Fig. 8a. The used air electrode showed distinct peaks that were not present in the diffraction pattern of the fresh air electrode; these additional peaks appeared at 2-theta of 37.3°, 43.3°, 62.9°, and 75.4°, which match the library XRD spectra of NiO (MDI Jade 5.0, PDF 65-5745), and 2-theta of 22.0°, 37.3°, and 38.3°, matching the library XRD spectra of lithiated nickel oxide (Li₂NiO₂.91) (MDI Jade 5.0, PDF 31-0733). The formation of lithiated nickel oxide could be due to the presence of Li₂CO₃, one of the components of the electrolyte. Lithiated nickel oxide is generally used as a cathode material in air batteries due to its high electrical conductivity, high electrochemical activity, and low precursor cost.

The morphology of the used air electrode was observed as shown in Fig. 9. The SEM images of the used air electrode in Figs. 9a and 9b, clearly show the well dispersed nickel oxide and lithiated nickel oxide. Using the high magnification images in Figs. 9c and 9d, the size of nickel oxide and lithiated nickel oxide particles was calculated to be in the range of tens to hundreds of nanometers. The small size of these nanoparticles provides a large surface area for reactions, improving the reaction rate of the OER and ORR.

Figure 7. Charging polarization of different electrodes in the electrolytes of molten carbonate with various additions. (a) Polarization curve of cathode. (b) Polarization curve of anode.

The extended cycling performance of the zinc-air battery was also recorded, as is shown in the charge-discharge data in Fig. 10a. This test consists of charging the cell with a constant current of 75 mA for 160 seconds, followed by an open circuit process for 60 seconds, and then discharging under a constant load of 100 Ω to a cut-off voltage of 0.8 V. The battery cycled steadily for 150 cycles. As shown in Fig. 10b, the coulombic efficiency remained between 85% and 94%, and the voltage efficiency was very stable at 72%. Moreover, the coulombic efficiency and voltage efficiency reached 90% and 73%, respectively, in the first cycle rather than that after five cycles, as was originally reported in our previous paper. This is consistent with the formation of lithiated nickel oxide. As shown in Fig. 10c, the average charge voltage was 1.53 V, the average open-circuit voltage was 1.34 V, and the average discharge voltage was 1.08 V. This discharge voltage was higher than that in the previous paper, which used the NaOH electrolyte additive instead of KOH.

The individual voltage profiles of the 1st, 20th, 40th, 60th, 80th, 100th, 120th, 140th, and 150th cycles are compared in Fig. 11. As shown in
The battery is discharged over a 100 h load to a 0.8 V cut-off. Figure 10. Cycling behavior of the zinc-air battery using molten Li$_{0.87}$Na$_{0.63}$K$_{0.50}$CO$_3$ eutectic electrolyte with 3 m KOH and 1 m ZnO at 550°C. The battery configuration used a 4 cm$^2$ steel foil electrode (cylinder 1 cm high with a circumference of 4 cm), and a 20 cm$^2$ nickel foil electrode (cylinder 2 cm high with a circumference of 10 cm) as the counter electrode. The steel foil cylinder was located concentrically inside the nickel foil cylinder. The molten electrolyte covered the steel foil electrode, while the nickel foil cylinder in the molten salt was only submerged 1 cm. The charging progress is carried out at 75 mA for 160 seconds. The battery is discharged over a 100 Ω load to a 0.8 V cut-off. (a) Cycling characteristics. (b) The coulombic efficiency and voltage efficiency. (c) Average voltage of charging, open circuit, and discharging.

Fig. 11, the charging curves and discharging curves are extremely coincident, respectively, indicating that the battery behaved similarly in each cycle. It can be observed from the discharge curves in Fig. 11 that there were two voltage plateaus. The voltage plateaus could be attributed to the discharge of Zn by Eq. 2. However, the formed K$_2$Zn$_3$O$_4$ should decompose to K$_2$O and ZnO by Eq. 3 on the zinc electrode. ZnO acts as an insulator and increase the internal resistance of the battery, reducing discharge voltage plateau or leading to formation of the secondary voltage plateau. We observed that ZnO powders dissolved more rapidly into the molten Li$_{0.87}$Na$_{0.63}$K$_{0.50}$CO$_3$ eutectic with addition of KOH than that with addition of NaOH, reducing the internal resistance of the battery, or improving voltage plateau. The voltage plateau at ~1.08 V accounts for about 75% of the charge capacity (12 C), which is superior to our previous results with the battery using the NaOH electrolyte additive, which the main voltage plateau was 1.06 V, and its discharge capacity was 58% of the total charge capacity. The secondary discharge voltage plateau was also improved with the addition of KOH to the electrolyte instead of NaOH; the secondary voltage plateau was higher than that obtained with the NaOH electrolyte additive (0.85 V and 0.82 V, respectively). Also, the discharge capacity of the secondary voltage plateau was shorter using the KOH electrolyte additive instead of the NaOH additive, 16% versus 33%, respectively.

To investigate the discharge rate performance of the battery, in each cycle the battery was charged with a constant current of 75 mA for 160 seconds and kept open-circuit for 60 seconds. For obtaining different current densities, the battery was discharged under a series of loads, 200 Ω, 150 Ω, 100 Ω, 80 Ω, 40 Ω, and 20 Ω for 20 cycles each. In Fig. 12a, the coulombic efficiency and average discharge voltage are displayed. When discharged under loads of 200 Ω, 150 Ω, 100 Ω, 80 Ω, and 40 Ω, the coulombic efficiency remained above 85%; however, when the load decreased to 20 Ω, the efficiency decreased to ~65%. The average discharge voltage of each cycle under loads of 200 Ω, 150 Ω, 100 Ω, and 80 Ω decreased slightly with each decrease in resistance. When the load was decreased to 40 Ω and 20 Ω, though, the voltage fell to ~0.90 V and remained relatively constant for both resistances. Essentially, when the load was high, the battery performed well, maintaining a high discharge voltage. On the other hand, the battery performance dramatically decreased under low loads. This can be seen in the discharge curves in Fig. 12b, where under different current densities, the battery exhibited vastly different voltage plateaus. The highest voltage plateaus occurred when the loads were 200 Ω and 150 Ω, whereas the lower loads produced voltage plateaus that were not as desirable. The relationship between discharges load, rate, coulombic efficiency, and voltage can be seen in Table I.
Discharge rate performance of the battery using molten Li$_{0.87}$Na$_{0.63}$K$_{0.50}$CO$_3$ eutectic electrolyte with 3 m KOH and 1 m ZnO at 550°C. (a) Coulombic efficiency and average discharge voltage of all 120 cycles, 20 cycles for each load. (b) Individual discharge curves of each load for 200 Ω, 150 Ω, 100 Ω, 80 Ω, 40 Ω, and 20 Ω.

Table I. The detailed rate capacities for the rechargeable zinc air battery with Li$_{0.87}$Na$_{0.63}$K$_{0.50}$CO$_3$ eutectic electrolyte containing 1 m ZnO and 3 m KOH at 550°C. The indicated discharge current density is normalized by the 4 cm$^2$ anode surface.

| Load (Ω) | Average discharge current (mA) | Average discharge current density (mA/cm$^2$) | Rate (C) | Average coulombic efficiency (%) | Average discharge voltage (V) |
|----------|-------------------------------|--------------------------------------------|----------|-----------------------------------|------------------------------|
| 200      | 4.9                           | 1.2                                        | 1.5      | 87.6                              | 1.12                         |
| 150      | 6.6                           | 1.7                                        | 2.0      | 89.4                              | 1.10                         |
| 100      | 9.9                           | 2.5                                        | 3.0      | 86.8                              | 1.08                         |
| 80       | 12.5                          | 3.1                                        | 3.8      | 88.6                              | 1.06                         |
| 40       | 24.2                          | 6.1                                        | 7.3      | 91.2                              | 0.95                         |
| 20       | 44.6                          | 11.1                                       | 13.4     | 66.6                              | 0.92                         |

Conclusions

The improvement of the rechargeable zinc air battery with a molten Li$_{0.87}$Na$_{0.63}$K$_{0.50}$CO$_3$ eutectic electrolyte is reported. Cyclic voltammetry studies have shown the reversibility of the redox reactions of Zn$^{2+}$, and it has also been demonstrated that the addition of KOH decreased the polarization of the battery. SEM and XRD results indicated that nanostructure nickel oxide and lithiated nickel oxide particles are formed in situ on the air electrode in the Li$_{0.87}$Na$_{0.63}$K$_{0.50}$CO$_3$ eutectic electrolyte with KOH additive at 550°C. When charged at a constant current of 75 mA and discharged over a constant 200 Ω load to a 0.8 V cut-off voltage at 550°C, the zinc air battery with KOH electrolyte additive demonstrated an improvement over previous studies that used an NaOH electrolyte additive, displaying very stable cycling ability, performing 150 charge-discharge cycles with a coulombic efficiency of 94% and an average discharge voltage of 1.08 V.

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