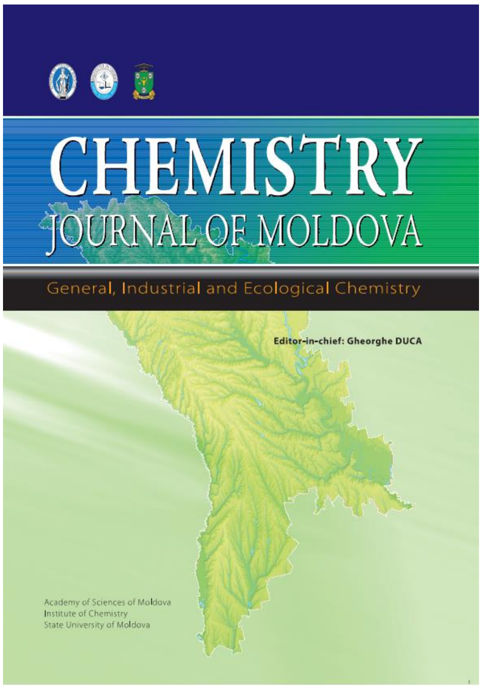

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ANTI-CORROSIVE PROPERTIES OF VARIOUS ORGANIC AND INORGANIC INHIBITORS IN ELECTROLYTE FOR ZINC-AIR BATTERIES

Priya Garg[a], Sudhish Kumar Shukla[a*], Pradeep Kumar Varshney[b]

[a]*Department of Sciences, School of Sciences, Manav Rachna University, Faridabad-121004, Haryana, India*
[b]*Swami Rama Himalayan University, Dehradun-248016, Uttarakhand, India*
[*]*e-mail: sudhish.shukla@gmail.com, +91-9555961834*

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[a] *Department of Sciences, School of Sciences, Manav Rachna University, Faridabad-121004, Haryana, India*
[b] *Swami Rama Himalayan University, Dehradun-248016, Uttarakhand, India*
**e-mail: sudhish.shukla@gmail.com, +91-9555961834*

Abstract. Zinc-air batteries offer a promising alternative for portable energy storage. However, the corrosion of zinc in alkaline electrolytes poses a significant challenge. This study explores efficacy of various organic and inorganic inhibitors, including citric acid, potassium iodide and sodium alginate as anti-corrosive additives in 7M KOH. A comparative analysis of experimental and theoretical constant-voltage discharge measurements and corrosion rates reveals that corrosion inhibition of zinc acetate and organic acid is more efficient in reducing dendrite formation. The active sites of organic acids, specifically oxygen atoms of carbonyl groups, adsorb onto the zinc surface, forming a composite protective film. This film effectively shields zinc from corrosion while allowing unimpeded battery reactions. Moreover, the addition of citric acid results in a maximum inhibition efficiency of 55.5%, which is higher than the efficiencies achieved with potassium iodide and sodium alginate additives. Surface analysis confirms that citric acid promotes the formation of a uniform protective film on the zinc surface. These findings indicate that the citric acid additive significantly enhances the anti-corrosive properties of the zinc film in the alkaline electrolyte of zinc-air batteries.

Keywords: anti-corrosive additive, alkaline electrolyte, electrolyte additive, sustainable energy, zinc-air battery.

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Introduction

Electrochemical batteries are playing a crucial role in this paradigm shift. The development of advanced battery technologies is vital to accelerating the global transition [1]. Secondary zinc electrode-based energy storage systems offer a promising solution for lightweight, high-performance applications. Zinc anodes boast numerous benefits, including low cost, low toxicity, and exceptional electrochemical properties [2]. Their advantages make zinc anodes an attractive choice for alkaline battery systems, catering to applications requiring efficient, reliable and eco-friendly energy storage [3]. Despite this progress, the zinc electrode still faces significant challenges, particularly with regard to its limited cycle life [4]. This limitation is primarily attributed to zinc corrosion, which is exacerbated by hydrogen evolution [5], as well as the high dissolution rate of zinc in traditional aqueous alkaline electrolytes [6]. Additionally, issues with dendrite formation and zinc electrode shape change further compromise the electrode's performance. Furthermore, the bifunctional air electrode also poses challenges, requiring stability and performance to support substantial cell voltage fluctuations during cycle life, due to its considerable overpotential towards both oxygen reduction and evolution [2]. During the charging process, the re-deposition of zinc-active material occurs non-uniformly, leading to localized hotspots of high current density. This uneven zinc redistribution on the electrode surface triggers morphological changes, including dendritic growth and diffusion-controlled deposition. Over time, dendrite formation can cause short circuits, ultimately compromising the electrode's capacity retention and cycle life [7]. In recent years, numerous efforts have been devoted to developing, modifying and optimizing zinc electrodes to minimize the solubility of zinc discharge products. The majority of these attempts have involved incorporating additives into either the electrode or the electrolyte [5,6,8-12]. However, this approach has a significant drawback: certain anode additives can reduce the initial availability of zinc-active species, thereby negatively impacting battery capacity and specific energy [10,13].

To mitigate the issue of dissolved zinc diffusing into the alkaline electrolyte, alternative strategies are necessary. One effective approach in zinc-based batteries involves modifying the alkaline electrolyte to reduce zinc solubility. Pursuing extended cycle life has led to innovations such as adjusting the electrolyte's alkalinity and ionic strength [14], thereby minimizing shape change and dendrite formation [12,14-17]. Concurrently, researchers have explored alternative formulations for zinc electrodes by incorporating innovative binders, such as conductive organic and inorganic additives. This strategic approach aims to surmount anode limitations, thereby enhancing the system's reversibility and ultimately, its cycle life [4,17-20]. The incorporation of additives has been shown to mitigate material accumulation on the anode by enhancing the dissolution of zinc deposits during each cycle. This enables the maintenance of a stable zinc ion concentration. Furthermore, additives facilitate the reduction of $Zn(OH)_2$ solubility by adsorbing onto active hydrogen evolution sites, thereby promoting early ZnO precipitation [21]. Electrolytes comprising NH_4Cl, KCl, $SnCl_2$, $SnCl_4$, $BiCl_3$, $HgCl_2$, K_2SO_4, Na_2SO_4, and Na_2SO_3 exhibit neutral properties, making them suitable for mitigating corrosion and providing high buffering capacities. These electrolytes also offer wide working potential windows of less than 2V, thereby reducing dendrite formation and minimizing electrolyte carbonation [22-25]. The incorporation of potassium iodide (KI) gives rise to a novel Zn-air/iodide hybrid battery (ZA/IB) concept. In this configuration, the added KI substitutes the traditional oxygen evolution reaction (OER) with iodide ion oxidation. This strategic substitution significantly reduces the potential gap between charging and discharging, thereby substantially enhancing the overall round-trip efficiency of the battery [26]. For several decades, researchers have explored the potential of organic weak acids, including carboxylic and amino carboxylic acids, as well as their salts, as additives in aqueous zinc electrolytes. These additives aim to enhance the quality of zinc deposition and suppress hydrogen evolution reaction (HER) [27-30]. Notably, these acids often exhibit polyprotic properties, yielding negatively charged conjugate bases capable of interacting with positively charged counter-ions. Furthermore, the solubility of zinc-organic salts is typically sufficient to prevent ZnO precipitation. The diverse range of organic weak acids offers flexibility in electrolyte design, enabling tailored optimization of zinc deposition and HER suppression. Citric acid, one of the most ubiquitous organic acids, is extensively utilized in various industrial and everyday applications, boasting an impeccable safety record. Despite its widespread use, the impact of citric acid treatment on the zinc anode's surface morphology, specifically in the context of zinc metal anodes, remains an unexplored area of research [30-32].

Therefore, it is of great significance to design novel, efficient and low-cost inorganic, organic and natural inhibitors and further investigate their corrosion inhibition mechanisms.

In this work, three kinds of novel inorganic, natural and organic corrosion inhibitors (citric acid, potassium iodide and sodium alginate) were investigated. The inhibition effects of the above inhibitors in electrolyte to reduce dendrite formation in alkaline Zinc-air batteries were investigated. The electrochemical behaviour and battery performance were studied by weight loss method, cyclic voltammetry and electrochemical impedance spectroscopy (EIS). The surface morphology and adsorption characteristics were investigated using scanning electron microscope (SEM).

Experimental

Preparation of electrode material

The pure metallic zinc sheet (>99.2% purity) was purchased from SPECIAL METALS, MUMBAI with a thickness of 0.2 mm. Zinc metal was fabricated into square-shapes samples with an area of 1×1 cm^2.

Electrolyte solution preparation

To conduct electrochemical tests on the zinc samples, a customized electrolyte solution was prepared. This solution consists of distilled water mixed with 7M potassium hydroxide (KOH) and various organic and inorganic additives. These additives are added to the solution to enhance corrosion resistance. The preparation of the electrolyte solution involved thoroughly mixing the KOH with 0.1 M citric acid, 0.35 M KI and 10 g sodium alginate with deionized water, followed by stirring the mixture for 5 minutes using a mechanical stirrer. This ensures the uniform dissolution of the salts and yields the required electrolyte solution for the electrochemical tests.

Corrosion rate test

To evaluate the corrosion rate in a 7 M KOH solution over a 60-minute duration (Table 1), standardized sheet samples of predetermined dimensions were submerged in various additive-containing solutions. Subsequent to immersion, the samples underwent a thorough rinsing process, followed by a precise weight measurement to determine the weight loss incurred during exposure [33]. The corrosion rate was calculated utilizing the Eq.(1).

$$\mathrm{CorrosionRate}(\mathrm{mgcm}^{-2}h) = \frac{87.6\times W}{\mathrm{DAT}} \qquad (1)$$

where, W- weight loss in milligrams;
D - Density in grams/ch^3;
A - surface area in cm^2;
T - time in hours.

The inhibition efficiency (IE) was subsequently calculated using Eq.(2) [34].

$$\mathrm{IE}(\%) = \frac{W_0 - \mathrm{Wi}}{W_0} \times 100 \qquad (2)$$

where, W_0 - weight loss of the blank sample;
Wi - weight loss of the sample with inhibitor.
Surface coverage θ = IE / 100.

A comprehensive study was conducted to evaluate the corrosion rates of zinc metals in 7M potassium hydroxide (KOH) solutions, employing various additives, including citric acid, potassium iodide (KI), and sodium alginate. The corrosion rates were meticulously measured at distinct time intervals (30, 60, and 90 minutes) to elucidate the temporal effects of corrosion. The results revealed that zinc films treated with citric acid as an inhibitor exhibited a significantly lower corrosion rate in 7M KOH solution after 90 minutes, outperforming the corrosion resistance of zinc films treated with KI, blank solution, and sodium alginate. These findings suggest that zinc films treated with citric acid possess superior corrosion protection properties in alkaline environments, highlighting its potential as a viable corrosion mitigation strategy.

Electrochemical studies

Electrochemical measurements were performed using an Origa Flex electrochemical workstation, employing a conventional three-electrode cell configuration. The setup consisted of a platinum counter electrode, a saturated calomel reference electrode, and a zinc working electrode. The zinc electrode was prepared by encapsulating it in epoxy resin, leaving an exposed surface area of 1 cm². All three electrodes were immersed in the electrolyte solution, comprising 7 M potassium hydroxide. Electrochemical Impedance Spectroscopy (EIS) measurements were carried out over a frequency range from 0.01 Hz to 100 kHz. The initial open-circuit potential was recorded, with a signal amplitude of 5 mV. Cyclic Voltammetry (CV) and Linear Sweep Voltammetry (LSV) measurements were also performed.

These measurements provided valuable insights into the electrochemical properties of the zinc electrode.

Results and discussion

The anticorrosive properties of the electrochemical system were enhanced by the addition of various additives, as demonstrated by the different characterisation methods employed.

Weight-loss study

By using the weight loss method, corrosion rate, inhibition efficiency and the surface coverage have been calculated in the absence and presence of various additives and the results are reported in Table 1. It was found that the corrosion rate decreases, while inhibition efficiency increases in the presence of the investigated additives. It is evident from Table 1 that citric acid exhibited the best inhibition efficiency and a lower corrosion rate among the tested additives. Furthermore, citric acid provided greater surface coverage, thereby reducing the exposure of the metal surface and suppressing the metal dissolution in alkaline medium.

Electrochemical Studies

The results were crucial in understanding the electrode's behaviour in the specified electrolyte solution. The data obtained were used to optimize the electrode's performance.

Cyclic voltammetry (CV)

A cyclic voltammetry analysis was conducted to assess the metal anode's electrochemical stability. The anticorrosive properties of the electrochemical system were enhanced by the addition of various additives, as demonstrated by the different characterization methods employed. The redox process initiates with single-electron reduction and oxidation steps.

Table 1

Inhibition efficiency of various additives in 7M KOH over 90 min.

Additives	*Weight Loss (in Grams)*	*Corrosion Rate (in mgcm^{-2}h^{-1})*	*Surface Coverage*	*Inhibition Efficiency (%)*
Blank	0.0009	0.011×10^{-3}	-	-
Citric acid	0.0004	0.003×10^{-3}	0.555	55.5
Potassium iodide	0.0006	0.005×10^{-3}	0.330	33.0
Sodium alginate	0.0005	0.004×10^{-3}	0.444	44.4

The cathodic peak potential (Epc) corresponds to the complete reduction of dissolved oxygen, while the anodic peak potential (Epa) marks the full oxidation of the zinc substrate at the electrode interface [35]. This analytical technique provides valuable information regarding the anode's electrochemical properties, providing a deeper understanding of its behaviour. This technique provides valuable information regarding the electrochemical behaviour and redox characteristics of the anode. The cyclic voltammograms of the blank electrolyte and electrolytes containing citric acid (CA), KI and sodium alginate (SA) additives in 7M KOH are presented in Figure 1.

In a 7 M KOH alkaline solution, Zn films containing various additives were scanned at a rate of 50 mV/s over a potential range from 0.6 to -1.8 V. During the first forward scan, the sample containing CA as an additive exhibited a small cathodic peak around +1.6 V corresponding to the reduction process occurring at the Zn electrode surface, followed by a gradual decrease in the current response. Upon reversal of the potential sweep, an anodic peak appeared around −1.6 V, indicating the oxidation process at the electrode interface. For the sodium alginate (SA) sample, a cathodic peak was observed near −0.4 V, while the corresponding anodic peak appeared at a similar potential, after which the current density gradually decreased [36]. In contrast, no difference observed between cathodic and anodic peak potentials was observed for the KI-containing electrolyte. Cyclic voltammograms of Zn film in 7M KOH did not show well-defined cathodic or anodic peaks, whereas Zn film containing Citric acid exhibited a pronounced cathodic peak around 1.6 V. Below are presented some possible electrochemical reactions occurring at the Zn electrode in 7M KOH containing citric acid (CA) as additive.

Zinc-citrate complex formation:

$$Zn^{2+}\,(aq) + C_6H_8O_7\,(aq) \rightarrow Zn(C_6H_5O_7)\,(s) \qquad (3)$$

In Eq (3) Citric acid ($C_6H_8O_7$) reacts with zinc ions (Zn^{2+}) to form a zinc-citrate complex. This complex may reduce zinc oxidation and consequently improve battery performance.

Citric acid oxidation:

$$C_6H_8O_7\,(aq) + 2OH^-\,(aq) \rightarrow C_6H_6O_7^{2-}\,(aq) + 2H_2O\,(l) \qquad (4)$$

Citric acid is oxidized by hydroxide ions (OH^-), resulting in the formation of a citrate ion ($C_6H_6O_7^{2-}$).

Zinc Oxidation with Citric Acid:

$$Zn\,(s) + C_6H_8O_7\,(aq) + 2OH^-\,(aq) \rightarrow$$
$$\rightarrow Zn(C_6H_5O_7)\,(s) + H_2O\,(l) \qquad (5)$$

Zinc metal reacts with citric acid and hydroxide ions to form a zinc-citrate complex and water.

Oxygen Reduction with Citric Acid:

$$O_2\,(g) + C_6H_8O_7\,(aq) + 2H_2O\,(l) + 4e- \rightarrow$$
$$\rightarrow C_6H_6O_7^{2-}\,(aq) + 4OH^-\,(aq) \qquad (6)$$

Oxygen is reduced in the presence of citric acid, resulting in the formation of citrate ion and hydroxide ions.

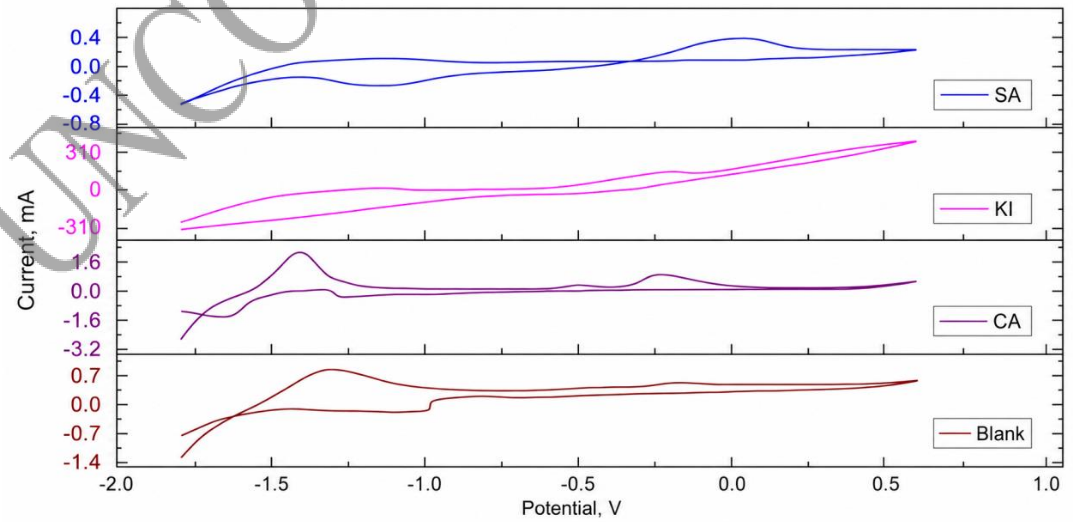

Figure 1. Cyclic Voltammetry (CV) curves zinc electrodes in 7M KOH in absence and presence of various additives at a scan rate of 50 mV/s

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The potential difference between the anodic and cathodic peaks is greater for the Zn film containing citric acid than for other additives in 7 M KOH. This result implies that Zn film with CA as additive exhibits higher electrochemical stability towards the oxidation-reduction processes in 7 M KOH solution.

Electrochemical impedance spectroscopy (EIS)

The optimization of additives in zinc-air batteries is crucial for enhancing performance, durability, and understanding the underlying electrochemical mechanisms. Electrochemical Impedance Spectroscopy (EIS) measurements were conducted in 7 M KOH to investigate the effects of several additives on the battery's electrochemical behaviour. The EIS measurements revealed complex impedance spectra, comprising three distinct semicircles. The high-frequency capacitive semicircle is attributed to the double-layer capacitance formed at the electrode-electrolyte interface. The mid-frequency inductive semicircle indicates a relaxation process associated with the corrosion reaction, while the low-frequency capacitive semicircle is linked to the diffusion-controlled corrosion process [37,38]. A larger loop diameter in the Nyquist plot is a hallmark of enhanced corrosion resistance, emphasizing the critical importance of tailoring anode composition to achieve optimal durability. Conversely, a smaller loop diameter is indicative of reduced corrosion resistance, underscoring the need for further optimization strategies or the exploration of alternative coating materials to mitigate corrosion-related degradation. By carefully calibrating anode composition and surface modifications, researchers can significantly enhance the corrosion resistance of zinc-air batteries, ultimately leading to improved performance, prolonged lifespan, and increased reliability [39]. The Nyquist plot (Figure 2) reveals a significant enhancement in the capacitive loop upon the introduction of additives, as evidenced by Electrochemical Impedance Spectroscopy (EIS) measurements in 7 M KOH.

Notably, citric acid emerges as a superior additive, exhibiting exceptional electrochemical activity compared to other additives. Further insights from the Bode plots (Figure 3) demonstrate that the zinc anode modified with citric acid achieves maximum impedance at lower frequencies in the 7 M KOH solution, indicating improved corrosion resistance. A meticulous analysis of the EIS spectra provides valuable information on corrosion kinetics, including charge transfer resistance, double-layer capacitance, and diffusion coefficient. By elucidating the complex interplay between additives, anode composition, and electrolyte properties, improved strategies for enhancing the efficiency, lifespan, and reliability of zinc-air batteries can be developed.

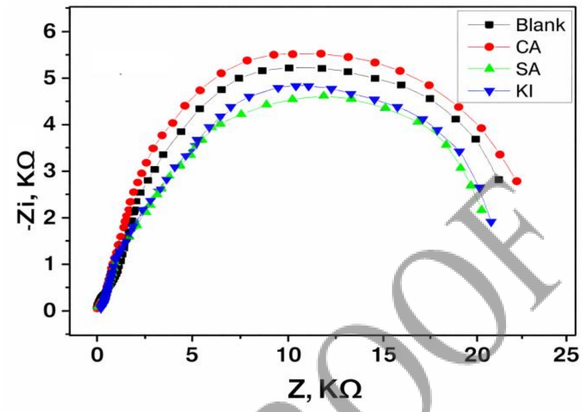


Figure 2. Nyquist plot of various additives in 7 M KOH.

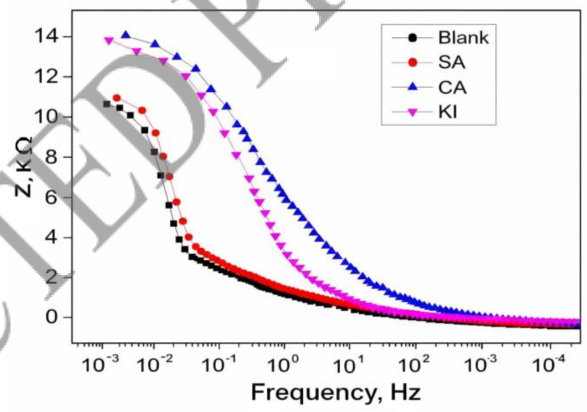


Figure 3. Bode plot for various additives in 7 M KOH.

Linear sweep voltammetry (LSV)

Linear sweep voltammetry (LSV) experiments were conducted to evaluate the electrical window (EW) of zinc (Zn) electrodes with various additives. The LSV tests were performed in a 7 M alkaline KOH electrolyte solution at a scan rate of 1 mV/s, with a potential range of -2 to -0.5 V. The cathodic stability of the Zn electrode in 7 M NaOH was determined to be approximately -0.76 V versus the standard hydrogen electrode (SHE). Notably, the Zn film with citric acid (CA) as additive in 7 M KOH exhibited the largest electrochemical stability window (ESW) at +0.5 V in 7 M KOH for the hydrogen evolution reaction (HER) in Figure 4. This remarkable stability is attributed to the formation of a protective film on the Zn surface. To ensure suitability as a current collector in Zn-Air batteries, the Zn-CA additive system in 7 M KOH must demonstrate stability against anodic dissolution. This requires careful evaluation of the electrode's electrochemical properties, including corrosion resistance and electrochemical stability. The Zn-CA additive system in 7 M KOH has ability to withstand anodic dissolution within the battery's operational voltage range. A comprehensive understanding of these properties is essential for optimizing the electrolyte performance and longevity. The results of this study provide valuable insights into the development of high-performance ZABs.

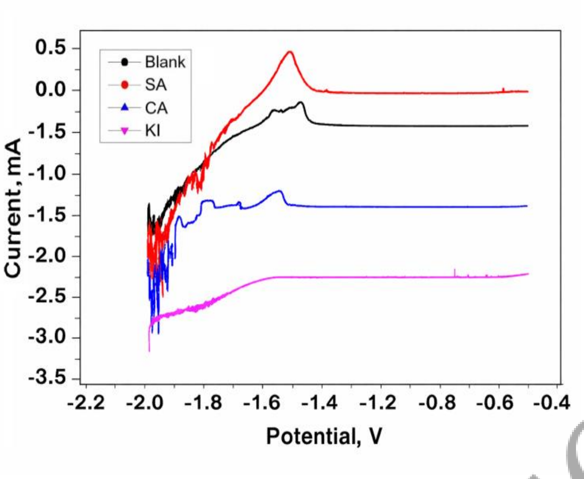
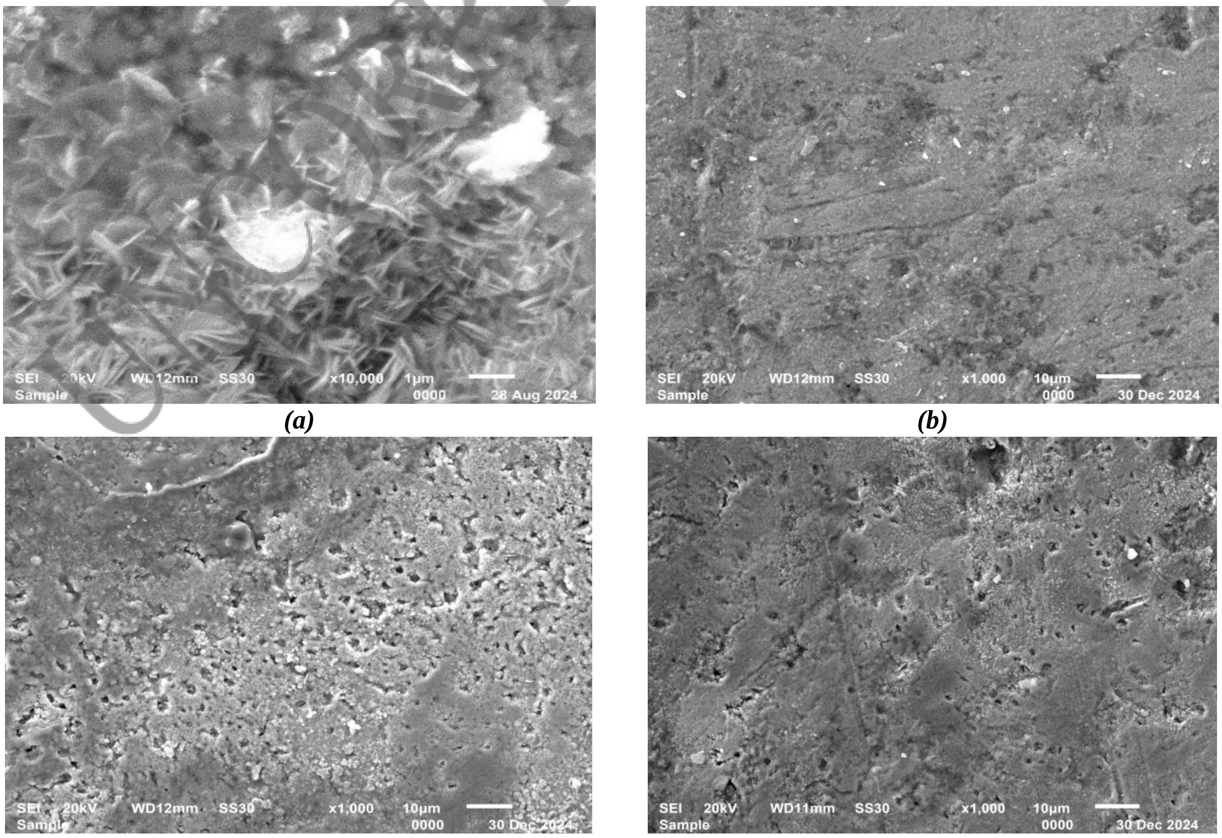


Figure 4. Linear sweep voltagram (LSV) curve for various additives in 7 M KOH at a scan rate of 1mV/s.

Surface characterization

The SEM images of zinc-air batteries with various additives in 7 M KOH reveal distinct surface morphologies. Following a 3600-second corrosion test in 7 M KOH, SEM imaging revealed pronounced degradation of the pure Zn electrode (Figure 5*(a)*). The surface exhibited widespread pitting corrosion, characterized by the formation of wide, shallow pits with a relatively compact morphology. The pit morphology suggests a high rate of zinc dissolution, facilitated by the alkaline environment. Furthermore, the anodic oxidation of Zn during the corrosion test led to the growth of needle-shaped dendrite structures, indicative of localized corrosion and zinc dissolution. The presence of these dendrites implies a significant increase in the electrode's surface area, potentially exacerbating corrosion. These findings underscore the inherent susceptibility of pure Zn electrodes to corrosion in alkaline environments, highlighting the need for surface modifications, alloying, or protective coatings to enhance corrosion resistance and ensure long-term durability. Among the additives, citric acid stands out as the most effective in Figure 5*(b)*, exhibiting uniformly distributed zinc oxide nanoparticles with significantly reduced zinc dendrite formation. This results in improved surface morphology and enhanced anode stability.



(a) *(b)*

(c) *(d)*

Figure 5. SEM images of Blank *(a)*, Citric acid *(b)*, KI *(c)* and Sodium alginate *(d)* in 7 M KOH.

In comparison, potassium iodide-treated sample, Figure 5*(c)*, exhibited white patches distributed across the surface, indicating the presence of well-dispersed iodide ions on the zinc surface, which contributed to reduced zinc corrosion rate and improved anode stability. Meanwhile, the sodium alginate-treated sample, Figure 5*(d)*, shows uniformly distributed alginate polymer chains, which improve zinc oxide dispersion and enhance anode stability. However, citric acid demonstrates superior performance in terms of zinc oxide dispersion, corrosion resistance, and overall anode durability.

Conclusion

The effects of different additives in Zinc-Air Batteries (ZABs) were compared, with citric acid (CA) emerging as the most effective additive. CA exhibited the largest electrochemical stability window (ESW), forming a protective film on the zinc surface that prevented corrosion and reduced anodic dissolution. This resulted in enhanced discharge capacity and cycle life, as well as improved electrochemical performance with reduced impedance and increased conductivity. In contrast, potassium iodide (KI) showed moderate performance by forming a complex with zinc ions and partially supressing corrosion and anodic dissolution, although its effectiveness remained lower than that of CA. Sodium alginate (SA) exhibited comparatively limited benefits, forming a hydrogel-like layer that moderately reduced corrosion and anodic dissolution; however, it showed relatively high impedance and low conductivity. Overall, CA was identified as the most effective additive for ZAB systems, offering improved electrochemical stability, enhanced discharge capacity, and extended cycle life.

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