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Synthesis and Characterization of Acidic Steel Slag as Anode Material of Lithium-Ion Batteries

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ABSTRACT

This study explores waste acidic steel slag as a potential anode material for next-generation high-performance Li-ion batteries (LIBs). The search for low-cost, sustainable electrode materials remains a great issue for state-of-the-art batteries. Acidic slag is the byproduct of steel refining and is rich in transition metal oxides (TMOs) with outstanding electrochemical characteristics. Our findings reveal that the proposed slag anode has high energy efficiency and good reversibility. The cell exhibited good cycling characteristics and capacity retention over 20 charge/discharge cycles with an average coulombic efficiency (CE) of above 90%, signifying high reversibility of the cycle. This study, therefore, simultaneously resolves the hazardous waste by-product of steelmaking and accelerates the evolution of LIB technology, presenting a low-cost and environmentally benign substitute for conventional anode materials.

Keywords: Li-ion batteries, Acidic Slag, Anode, Cycling and Coulombic Efficiency.

1. Introduction

Recent economic development has been rapid, which has resulted in increased industrial waste production, consequently creating enormous environmental and economic challenges. The proper management of this waste is essential to reduce the environmental contamination it contributes to, as well as to minimize the cost of its disposal. Proper disposal of waste contributes greatly to environmental mitigation and cost savings of its disposal. Such waste, which is considered as a liability, can be recycled to be a useful resource in the manufacturing of highly valuable products. This perspective shift has given rise to a lot of innovative methods to turn industrial waste into value-added, eco-friendly products [1-2].

One such example of industrial waste would be steel slag, which is a byproduct of the iron and steel production process. All over the world, several hundred million tons of steel slag are being produced every year [3]. Traditionally, steel slag has been used in cement and aggregate products; however, its recycling is viewed as an economic underachiever and environment unfriendly [4]. Excess slag is often landfilled, posing a waste management problem and an environmental nuisance.

The soaring global economy combined with the swift depletion of fossil fuels along with an increasing environmental concern necessitates the instant establishment of clean, efficient and sustainable energy sources. This also necessitates advancement in energy storage systems. Among these, rechargeable LIBs stand out as one of the most prospective options for wide-ranging application such as backing up an electric vehicle or stationary energy storage. This is thanks to their efficiency, long-life, high-energy concentration and eco-friendliness [5].

A developing technique that has the potential to solve both problems is direct utilization of acidic steel slag as an anode material in lithium-Ion batteries. Acidic slag also exhibits structural and chemical characteristics conducive to lithium storage and transportation due to its high active oxides content of SiO_2, Fe_2O_3, CaO, and Al_2O_3 [6]. These redox-active components confer significant potential in high-tech applications in energy storage systems. Acidic slag is a cheap by-product, which makes it an attractive substitute to graphite-based anodes, with the potential to drive the performance of LIBs forward.

The present paper explores the use of acid-washed steel slag directly as an anode material in LIBs without requiring complicated extraction of oxides. With the help of a simple and scalable process, we intend to provide two benefits simultaneously: the sustainable use of industrial waste and the development of battery technology. This study highlights the material properties of acid-washed slag, its characterization, and its performance as an anode material—bridging waste management and high-performance energy storage in a single approach.

1.1 Lithium Ion Battery

LIBs are among the most renowned and state-of-the-art energy storage technologies of recent times, mainly because of its advantageous properties of lithium ions as charge carriers. Lithium, the third lightest of elements on the periodic table, has extremely low reduction potential and a very small radius with only one proton contained in the nucleus. Therefore, LIBs are usually light on weight and also, they can store energy densely in a compact space. These characteristics make lithium ideal for a high-capacity energy storage such as portable electronics like laptops, mobile phones and electric vehicles.

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Due to this, their application in renewable energy storage is on the increase as they serve to balance supply of the flow of energy of the renewable sources and maintain a continuous supply of energy without interruption [7-8]. Moreover, the ongoing technological improvement of lithium-ion batteries has resulted in their utilization at grid-scale energy storage solutions, where their quick reaction and scalability are invaluable to stabilize the grid and manage energy properly [9].

However, the growing interest in high performance has prompted an intense search on novel electrode materials that exceed the capabilities of the traditional intercalation compounds. Particularly, the inherent drawback of the carbon-based anodes demands seeking of other alternatives [10]. Among these, transition metal oxides have been seen as particularly promising candidates because of their very large theoretical capacities that are many times larger than those of graphite and are largely based on a conversion reaction mechanism rather than intercalation process [10-15].

1.1.1 Mechanism of Lithium-Ion Battery

A typical LIB includes a battery cathode composed of common substances like lithium cobalt oxide (LiCoO2), a battery anode of graphite, and an electrolyte-filled polymeric separator that enables a transfer of lithium ions (Li+) without direct connection between the battery electrodes.

However, the process of conversion reaction with TMOs as anode, undergoes a different mechanism as compared to that of intercalation/deintercalation. Unlike intercalation which involves the in-and-out insertion of lithium into an existing host lattice with little structural change, conversion involves the destruction and re-formation of chemical bonds, resulting in a major rearrangement of the active material crystal structure and morphology through electrochemical cycling. This key difference enables conversion materials to reach theoretically much higher energy densities than conventional intercalation materials since crystallographic restrictions in the ratio of intercalated lithium atomic positions within a fixed host crystal structure do not apply [17]. The formation of new structures commonly observed in conversion reactions, such as the formation of lithium oxide incorporating metallic nanoparticles, is a complex combination of the morphological and electrochemical processes [18]. This complex process entails a loss of electrons on the transition metal oxide as it is reduced to its metal state and the formation of Li2O during discharge, which is oxidized and disintegrated during charge. A general representation of conversion reaction is:

$$MO_x + 2xLi^+ + 2xe^- \leftrightarrow M + xLi_2O$$

where MO_x is typically TMOs such as MnO_x, FeO_x, CuO_x, and NiO_x [19].

1.2 Acidic Slag

Slag may be either acidic or basic according to its composition and the flux used in some metallurgical processes. Acidic steel slag is a byproduct produced in steelmaking, particularly in those steel plants that use electric arc furnaces (EAF) or basic oxygen furnaces (BOF), where impurities and fluxes are separated as a liquid metal is distilled off. It is distinguished by a large proportion of acidic or amphoteric oxides, mainly silica (SiO2) and alumina (Al2O3), and a related low level of bases such as lime (CaO) or magnesia (MgO) [21]. Such oxides have high polymerization characteristics with the silicates forming viscous networks that make the slag more corrosive, besides being hard to work with under high temperatures [22]. Unlike simple slag, acidic slag contains more acidic oxides and is more acidic in character, usually the result of acid fluxes, such as quartz or other siliceous materials. The basicity index, which is defined as ratio between the basic oxides and the acidic oxides, is usually taken to describe the nature of the slag, with a value of less than 1.0 being acidic and above 1.0 being basic [23].

1.2.1 Compositions of Acidic Steel Slag

The composition of the acidic slags varies with the materials used and process conditions, but as it is usually high in acidic and amphoteric oxides, silicon dioxide (SiO2) and aluminum oxide (Al2O3), which can comprise more than 50% of its weight [24]. In addition to these, traces of iron oxides, including Fe_2O_3 and Fe_3O_4, may also be found in large amounts as a result of failure to separate the iron from the molten slag. The minor additions consist of a mixture of calcium oxide (CaO), magnesium oxide (MgO), manganese oxide (MnO), titanium dioxide (TiO2) in different proportions depending on the type of ore and additives used during steel making process [25].

Figure 1 shows the compositions of steel slags in various operations, with steel slag sometimes also containing up to 30% metallic iron by weight. Calcium oxide and iron oxide are the two most abundant chemical compositions of both EAF and BOF slags. The mineralogy of the steel slags is uneven due to variability in the chemical composition of the steel slags.

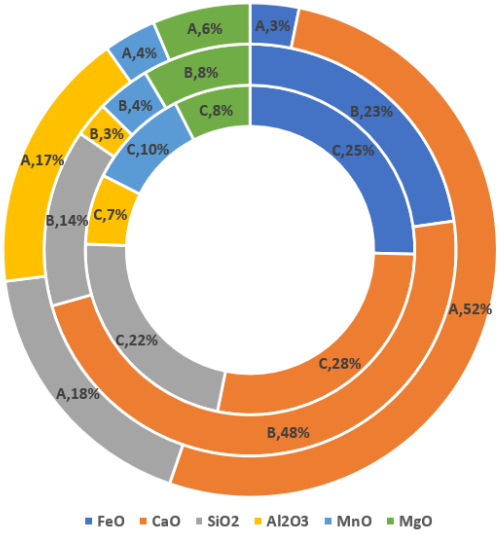

Fig. 1 Chemical compositions of steel slag from various literature. A) Ladle slag, B) BOF slag and C) EAF slag.

2. Methodology

The research methodology describes the logical proceeding that will be taken to convert waste steel slag to high-performance LIB anodes, which is presented in **Figure 2**.

Fig. 2 Schematic of coin cell preparation from acidic steel slag.

2.1 Anode Preparation

We collected waste acidic metal slags from a nearby steelmaking plant. The large amount of slag collected was crushed into smaller pieces using a crusher. The crushed slags were then further ground into fine particles via the application of a grinder. After that, the fine powders of acidic slags were screened using a sieve.

After separating the fine acidic slags, the material was prepared for anode fabrication by mixing acidic slag the active material, carbon black as a conductive additive, and polyvinylidene fluoride (PVDF) as a binder in a 70:15:15 weight ratio. The binder was then dissolved in a typical PVDF solvent, N-methyl-2-pyrrolidone (NMP), to give a homogenous slurry. This slurry was then stirred thoroughly with a mortar until all contents were homogeneously dispersed, resulting in a viscous slurry that was uniformly deposited on a copper foil (current collector). The surface-coated foil was thereafter dried in a vacuum oven overnight at 105℃ to evaporate the solvent, NMP, and ensure that the electrode is perfectly dry to be assembled in the cell.

2.2 Battery Assembly

After that the acidic slag coated copper foil was served as the anode, and a lithium cobalt oxide ($LiCoO_2$) cathode was used, which is a common choice in commercial LIBs due to its high energy density and stability. A porous 15 mm polyethylene separator was installed between the anode and the cathode to avoid the contact and allow the movement of lithium ions. The role of the separator on the safety of operation is associated with its contribution to the elimination of short circuits. 1M Lithium hexafluorophosphate (LiPF6) electrolyte dissolved in ethylene carbonate (EC) and diethylene carbonate (DEC) at a volume ratio of 3:7 was then introduced between the electrodes. This was added to improve transport of ions between the electrodes under the charge and discharge processes.

The anode, separator, and cathode were carefully stacked and assembled into a CR2032 type coin cell. After that, the cell was sealed using a hydraulic crimping machine. The coin cell was compressed at a pressure of 100 psi for the cell to be completely sealed and airtight. Following the assembly, an electrochemical test was carried out on the battery using a LAND-CT2001A battery analyzer (LANDt Instrument, USA) under the charge-discharge mechanism within 0.1-3.5 V with a constant current rate (CC) of 5mA on 20 cycles.

3. Result And Discussion

After the battery was connected to the LAND battery testing system, it was then permitted to undergo galvanostatic charging-discharging (GCD) cycles. a slow charging and discharging procedure that builds a stable solid electrolyte interphase (SEI) film at the anode. The SEI is a protective layer that forms spontaneously in the initial cycles and is essential to battery long-term performance as it prevents additional anode degradation by the electrolyte.

As revealed in the charge-discharge graph (**Figure 3**), the cell shows a stable and repeatable voltage profile across all cycles. The charging profile exhibits a very steep rise in the voltage but stabilizes as it reaches the upper cut-off voltage, whereas the discharging curves appear smooth reduction till it reaches the lower cut-off voltage (0.01V). Each cycle is separated by a 1-minute rest phase, which helped stabilize the electrode–electrolyte interface.

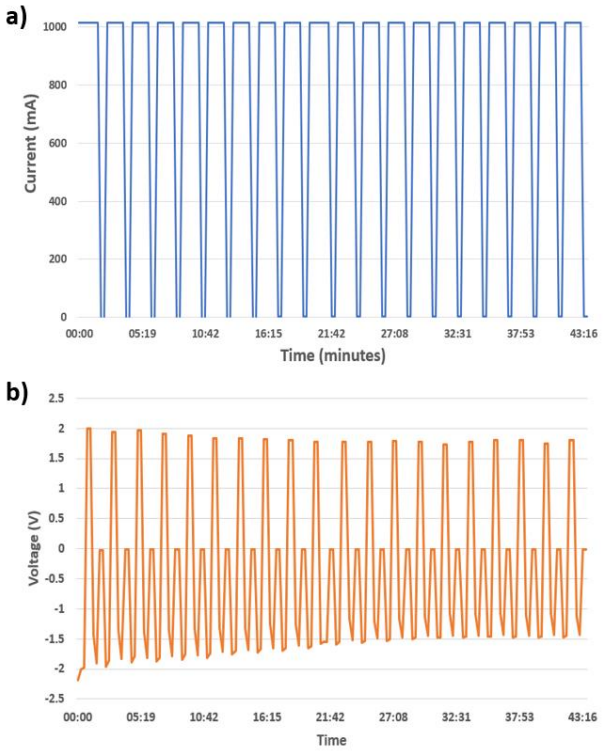

Fig. 3 a) Current and b) voltage profile of the battery over the first 20 cycles

The lack of sharp voltages or variations establishes that little internal resistance accumulation occurs inside and the stable lithiation and de-lithiation is obtained. The flat trend over 20 cycles indicate a strong structure retention of the anode material without strict engineering or surface treatments. The smooth appearance of the curve means that the anode is operating well and there is no sudden increase in the dendrite growth or thermal runaway that can cause a short circuit in the cell.

The cell was also shown to be stable and consistent through time. The coulombic efficiency starts at a value of around 45% in the first cycle as it is natural since the processes that take place in the first cycle cannot be completely reversible, such as the formation of solid electrolyte interphase (SEI), which causes an irreversible loss of lithium, incomplete activation of the electrode surface, and trapping of lithium ions in structural defects or solid matrices of the slag. The CE takes an upward jump in the second cycle to reach around 92% before gradually settling at a steady range of 89 to 91% in the next 14 cycles up to the 18th cycle (**Figure 4**).

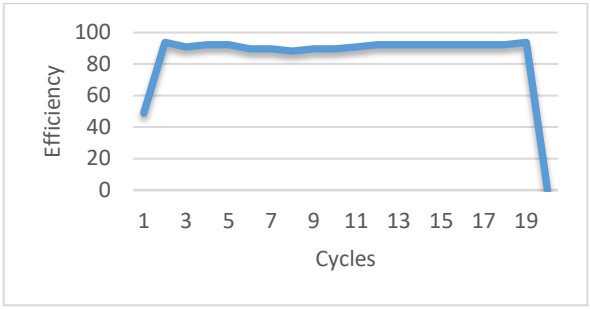

Fig. 4 Efficiency curve over the first 20 cycles

This constant behavior means that the redox reactions are now more reversible after conditioning, the SEI has been stabilized, and parasitic side reactions are now less critical, which results in predictable behavior during charge and discharge. Nevertheless, the clear reduction in efficiency at the 19th cycle is indicative of the start of a degradation event or perhaps some experimental disturbance like an incomplete cycle or loss of contact inside the cell.

The battery also showed consistent capacity over several cycles, thus stable performance over time. The charge capacity is a lot higher initially, beginning at a value about 35 mAh per cycle minimum but quickly fluctuating down to about 18-19 mAh per cycle near subsequent cycle number two, and then remains relatively constant throughout the remaining cycles (**Figure 5a**). This significant drop reflects the inefficiencies typical of the early cycle of lithium-ion cells, which can be due to irreversible losses of lithium to irreversible stability of part of the SEI during cycling, initial electrode activation losses, or side-reactions.

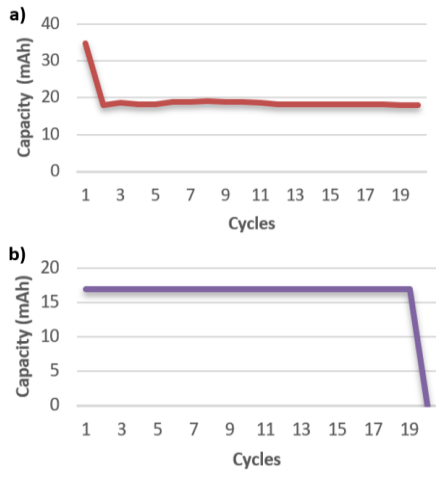

Fig. 5 a) Charge capacity and b) discharge capacity over the first 20 cycles.

By comparison, the discharge capacity has an initial capacity level of about 17 mAh (**Figure 5b**), which essentially lies on a flat and steady line throughout the test, indicating a steady extraction of lithium and efficient structural preservation of the electrode. But unlike the gradual trend of the charge curve, the discharge capacity suddenly drops to quite low in the 20th cycle, indicating some possible forms of degradation, like internal shorting, electrolyte dry-up, or electrical bad connection. This mismatch between charging and discharging behavior manages to assert the natural asymmetry in electrochemical processes during cycling, where it is natural that the initial charge loss will already take place due to irreversible processes, but that the discharge process will remain stable until the critical discharge limits are reached. All these trends indicate that the slag-based anode has good reversibility and structural integrity in the majority of the cycles, though it needs additional optimization to avoid end-of-life failures and to be enhanced.

4. Conclusion

The tested coin cell using the treated slag had a stable charge-discharge behavior and maintained high specific capacity after numerous cycles. Although the initial capacity is rather low, the anode performed consistently with a capacity loss of less than 3% over 18 cycles and an average Coulombic efficiency exceeding 90%, showing excellent electrochemical reversibility and minimal side reactions during the formation cycle.

The results suggest innovative use of acidic slag to replace anode materials in the next generation of batteries. The findings are encouraging, as they may just help solve some of the most essential problems in the field of waste disposal and energy storage. In addition to the earlier outlined benefits of the process, a significant improvement in terms of energy density and cycle life of the battery could be achieved, since the synthesized anode demonstrated enhanced charge capacity, improved cyclic stability, and reduced sensitivity to temperature as compared to the conventional carbon-based anode.

Despite the encouraging results, this study has certain limitations and is based on a few underlying assumptions. It is based on the assumption of chemical homogeneity of the slag composition, whereas in practice, the elemental content may be inconsistent since it is dependent on the origin of the steel production. The electrochemical measurements were conducted at a maximum of 20 cycles, and this constrains our perception of the long-term stability and rate capabilities of the anode. In addition, the coatings, like carbon deposit or anti-oxidation doping, act as a basis to help define future research pursuits.

Assuming that current limitations are overcome, it is hoped that acidic slags will have more applications as anodes to offer a cheaper and more environmentally friendly alternative. However, several technical constraints need to be addressed: developments in extraction and synthesis processes must be made on a larger scale, and it remains challenging to mitigate the volumetric expansion issue during battery cycling, although it has been achieved best with high porosity iron oxide, which still needs to be modified for practical application. In a nutshell, the research on the electrochemical behavior of acidic slag as anode of LIB offers a lot of insightful value, which can be used in future to advance the design and engineering of energy storage systems, including the recycling of wastes.

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