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Controlling hydrogen evolution on iron electrodes

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ABSTRACT

Aiming to develop a cost effective means to store large amounts of electric energy, NiFe batteries were produced and tested under galvanostatic conditions at room temperature. Multiple regression analysis was conducted to develop predictive equations that establish a link between hydrogen evolution and electrode manufacturing conditions, over a wide range of electrode/electrolyte systems. Basically, the intent was to investigate the incidence of lithium hydroxide and potassium sulphide as electrolyte additives on cell performance. With this in mind, in-house built Fe/FeS based electrodes were cycled against commercially available nickel electrodes on a three electrode cell configuration. A 3×4 full factorial experimental design was proposed to investigate the combined effect of the aforementioned electrolyte additives on cell performance. As a consequence, data from 144 cells were finally used in conducting the analysis and finding the form of the predictive equations. Our findings suggest that at the level of confidence $\alpha = 0.05$, the presence of relatively large amounts of the soluble bisulphide would enhance the performance of the battery by reducing electrolyte decomposition.

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Introduction

Fossil fuels are, by definition, non-renewable energy sources that have fulfilled our energy needs during modern times. However, if we keep using them non-stop as before, in the long run, we will, inevitably, exhaust our planetary resources, for they can't be replenished (or at least, it would be unpractical). Renewable sources, on the other way, have the potential to provide large amounts of energy without exhausting them. It is imperative to find cost effective ways to store energy coming from these sources [1]. The knowledge of these aspects has prompted an increasing demand of energy from renewable sources [2–7].

Unfortunately, the availability of renewable sources (such as temporary energy profiles, availability of sun light, sufficient

supply of water, etc.) has restricted their use. In essence, the natural incompatibility between energy generation and demand must be addressed otherwise a large scale utilization of renewable sources would be unviable. Moreover, the use of renewable sources such as wind or solar would produce electricity, which is not easy to store, while reducing carbon dioxide production [8,9]. Basically the best way to store electricity is to convert electricity into a non-electric form of energy (chemical, kinetic, potential, etc.), once electricity is needed, the non-electric form of energy is reverted back into electricity for further use. Basically, sustainable energy storage has been identified as a global challenge that requires solution [6,10].

Nickel-iron cells are secondary batteries that were successfully commercialized back in the early 20th century but fell out of favour with the advent of cheaper lead acid

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batteries. There are many reasons favouring the use of NiFe cells as cost effective solutions to store grid-scale amounts of energy, such as: low cost of raw materials, environmentally friendliness, tolerance to electrical abuse, long life (in the order of thousands cycles of charge and discharge) and compatibility with PV's [11]. Moreover, it has been recognized that this technology would be suitable for relatively low specific energy applications ($30\text{--}50\text{ W h kg}^{-1}$) [12]. As a consequence, there are good reasons to foresee a large scale utilization of this technology; but there is a plethora of challenges to overcome first, such as increasing the cell efficiency, preventing electrolyte decomposition and therefore the evolution of hydrogen, and increasing energy and power densities [13,14].

One of the reasons for the attractiveness of NiFe cells is related with the abundance of the raw materials required for their construction. Iron is not only relatively easy to shape into different forms, but it is also the fourth most abundant element in the Earth's crust; the liquid core of our home planet is thought to be mainly composed of iron [15,16]. Although, less abundant, nickel is believed to be the second most abundant element in the Earth's core; in addition, large deposits of nickel ore can be found in many countries including Brazil, Russia, Philippines, Canada, Australia, Indonesia, etc. [17,18]. Even less abundant than nickel, bismuth is considered the 64th most abundant element in the Earth's crust [19,20], but only small amounts of this element are required in the production of iron electrodes. So a shortage of any of the aforementioned elements is not going to take place any time soon. Unfortunately, the process of extraction of metals such as iron renders huge amounts of carbon dioxide [21]. Recyclability of battery components, as with lead acid batteries, would provide a way to mitigate this issue [22].

Essentially, from the iron electrode point of view, the main process taking place during its charging process is the reduction of ferrous ion (Fe^{2+}) to metallic iron (Fe^0); in the same manner, the oxidation of metallic iron to ferrous ions take place during the discharging process of the iron electrode. Eq. (1) illustrates the charging and discharging (forward and backward reactions respectively) processes of an iron electrode under alkaline conditions [14,23,24].



It is well known, however, that during the charging of an iron electrode (under alkaline conditions), water is decomposed to yield hydrogen. Therefore, part of the energy, that was originally intended to be stored in the battery, is finally wasted in the parasitic evolution of hydrogen. In other words, hydrogen evolution accounts for a drastic reduction in the overall performance of the battery, as indicated by Eq. (2) [24].



Many attempts have been made in order to mitigate or even prevent the evolution of hydrogen during the charging of the iron electrode. The most promising strategies rely in the modification of the iron electrode formulation, by either nano-structuring the electrode or by the addition of elements (such

as sulphur or bismuth) that are capable to increase the over-potential for hydrogen evolution [25–27]. As a consequence, different electrode formulation additives have been utilised to achieved that aim, including bismuth [28], bismuth sulphide [26], carbonaceous materials [29–31], iron oxide [32,33], etc. Undoubtedly, the development of sulphur based iron electrode formulations is one of the most promising alternatives [25]. Moreover, the performance of the NiFe cells can also be further improved by optimising the electrolyte composition; in fact, different electrolyte additives such as wetting agents [34], long chain thiols [35], organic acids [36], among others have been investigated. Our experimental results confirm the addition of soluble sulphur species will enhance the performance of the NiFe battery [37,38].

In the quest for a highly efficient NiFe battery, different materials and manufacturing strategies have been used; in fact, nickel-iron cells reaching nearly 800 mAh/g have been reported [27,39], unfortunately these batteries require costly reactants and nano-structuring techniques. These aspects would certainly influence the final price of the battery thus produced [27,39].

Without pretending to give a full introduction on experimental design or multivariate statistics, we would like to underscore the importance of using a framework that allows us to deal with multidimensional problems, especially when large variability across experiments is found or at least suspected. Basically, experimental design will provide the best evidence that factors would account for your response variables with certain degree of certainty (the so-called α level).

Under the light of our previous arguments, we have decided to investigate and improve aqueous nickel-iron cells as cost effective solutions to store grid-scale amounts of energy. Broadly speaking, we have been using experimental design and multivariate analysis to facilitate our research [26,28,40]. In this manuscript, a 3×4 full factorial design has been used to investigate the incidence of both lithium hydroxide and potassium sulphide in the electrolyte as a means to increase the hydrogen energy barrier (thus preventing hydrogen evolution from the decomposition of the electrolyte). In addition, we have used FeS/Fe based electrodes as anodes, commercial nickel electrodes as cathodes, and concentrated solutions of potassium hydroxide as the electrolyte system. The production details of the electrodes can be found elsewhere [26,37].

Experimental

Iron based electrodes were produced by coating strips of nickel foam with an Fe/FeS active paste which consists of varying amounts of electroactive material (with this term we mean iron) with a mixture of iron sulphide and PTFE (acting as a binder). The chemicals and materials used to develop our electrode formulations were of the following specifications.

Iron powder (purity 99.5%, $<10\ \mu\text{m}$) from Alfa Aesar
 Iron sulphide (purity 99.5%) from Sigma Aldrich
 PTFE (Teflon 30-N, 59.95% solids) from Alfa Aesar
 Nickel foam (purity 99.0%, density $350\ \text{g/m}^2$) from Sigma Aldrich

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