



Design and Construction of Redox Flow Battery using Liquid Extract from Morinda Lucida Leaves

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ABSTRACT: The research is focused on the use of a liquid extract from morinda lucida leaves as electrolyte in the construction of a redox flow battery using fired clay pot as the separating medium. After the Soxhlet extraction, each of the liquid electrolyte was stored in a container for each half-cell, and the electrode was dipped into the electrolyte. For cell testing and charging, a wire was connected to the top of the electrodes. A 3V battery was used to charge the cell, a maximum discharge voltage of 1.31V and a maximum discharge current of 13mA were recorded from the battery testing results. Results showed that the liquid extract from morinda lucida leaves as a good conductor of electricity and a suitable electrolyte for a redox flow battery.

DOI: <https://dx.doi.org/10.4314/jasem.v26i2.2>

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Keywords: *Morinda Lucida*, Anthraquinone, Electrolyte, Redox flow battery

A redox flow battery is an electrochemical energy storage device that uses reversible oxidation and reduction of working fluids to convert chemical energy into electrical energy. Redox flow batteries are promising in stationary storage of energy from intermittent sources such as solar and wind because of their flexibility in system design and ability to scale costs. The redox flow battery (RFB), which is known for its low cost and long life, has the potential to improve renewable energy utilization (Choi *et al.*, 2017). Redox flow batteries (RFBs) are safe, inexpensive, and long-lasting when compared to current battery technologies. The use of liquid redox-active species avoids mechanical strain during cycling, which can result in a longer service life. Although vanadium RFBs have been widely used, vanadium is difficult to prepare and expensive, costing up to \$40 per kilogram, limiting their use on a large scale (Perry and Weber, 2015). Due to the obvious growing demand for static energy storage and the scarcity of available devices, redox flow batteries (RFBs) has sparked a lot of interest. The fast-growing market for intermittent solar, wind, and tidal power, as well as the promise of highly efficient power grids based on extended and versatile load-levelling capacity, are the main drivers of this trend (U.S. Department of Energy, 2013). Medium and large-scale installations are being

commercialized after decades of fundamental research and pilot-scale developments, resulting in a nascent and active RFB industry. However, the development of practical RFBs has been difficult (Dizaji and Li, 2020). Even successful systems face numerous challenges, such as the need to reduce capital costs, lower operating costs, and extend battery life. RFB research has made significant progress in the selection of electrolytes, electrodes, and membranes, as well as the proposal and reintroduction of alternative chemistries. However, unlike fields such as electrolysers and fuel cell design, the academic field has largely evolved empirically around the electrochemistry of established Redox species (Kim *et al.*, 2015). The accepted principles of electrochemical engineering have received little attention. The principles of green energy storage, such as the limitation of degradation or the minimization of critical materials, require more attention (Winsberg *et al.*, 2016). Unsuccessful scale-up, poor maintenance, expensive manufacturing, low efficiency, and a short life-cycle are all issues caused by this restricted approach. The majority of publications, in particular, have focused on short-term laboratory studies of small electrodes; only a few have looked at pilot-scale devices and the impact of cell design, electrode structure, reaction environment, and operational

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conditions on performance. As a result of their fast-Redox kinetics and chemical tenability, anthraquinone derivatives have been developed to replace vanadium as a Redox-active species in aqueous flow batteries. The implementation of an aqueous flow battery presents fewer engineering technical challenges than a non-aqueous flow battery and thus has commercial potential (Weber *et al.*, 2011). As a result, the goal of this research is to develop a long-lasting, weather-resistant, and compact redox flow battery using liquid extract derived from the leaves of *morinda lucida benth* plant species.

MATERIALS AND METHODS

Collection and processing of plant sample (*Morinda lucida*): *Morinda Lucida* leaves were collected from the Federal University of Technology Akure campus in Ondo state, Nigeria, where they were abundant. To avoid destroying the active compound, the leaves were removed and properly washed in tap water before being air-dried at room temperature until a constant weight was achieved. It was kept away from high temperatures and direct sunlight. The plant's dried leaves were pulverized with a ceramic mortar and pestle to produce a powdered form, which was then stored in an airtight container.



Figure 1: *Morinda lucida* plant

Fresh leaves of *morinda lucida* were harvested and a test sample was taken immediately after harvesting and weighed, the sample weighed 165.65g. it was then air-dried, it reduces to 55.11g after the second week, 44.07g after the fourth week which was reduced to 31.32g after it was oven-dried.

Extraction of Anthraquinone: The extraction of anthraquinone was carried out using absolute ethanol as the extracting reagent and the Soxhlet apparatus as the extraction method. The ground sample was weighed and 90g of it was placed inside the Soxhlet apparatus, 750ml of ethanol was added (600ml to the

sample and 150ml to the conical flask). The height of the constant maximum level above the liquid inside the apparatus was used to determine the amount of ethanol and the sample. The cooling chamber, re-circulator, conical flask extraction chamber, and tripod stand make up the Soxhlet apparatus. The re-circulator is in charge of recirculating cold and hot extract water from the cooling chamber via a pipe, with the pump housed inside ice-filled water. The extracting solvent is heated to evaporation in the cooling flask, followed by condensation of evaporated solvent from the conical flask back into the extracting chamber (Soxhlet apparatus). The conical flask is where the extracting solvent is heated to evaporation, condensation of vapour from the flask takes place in a cooling chamber, and then drops back into the chamber; this process continues while the extraction is taking place until the level of liquid in the chamber reaches the returning level. The liquid in the chamber returns to the flask, where it is heated and vapourized again; the cycle is repeated five times (when the solvent is clearer, as in this extraction) before the sample is discarded, and the ethanol is recovered. Only the extracting solvent evaporates when the mixture is heated; the extract is recovered after each cycle of absorption by the leaves and ethanol. The process was repeated five times, resulting in a reduction of two liters of ethanol to 0.8 liters.

Materials Selection: Materials selected for this project are locally available, anthraquinone which was extracted from *morinda lucida* obtained from university premises and can also be manufactured from carbon, hydrogen and oxygen. The choice of electrolyte counterpart and supporting electrolyte were selected based on safety, environmental concern and available model. To avoid numerous unknown parameters, the electrolyte was selected according to the alkaline quinone flow battery designed by Lin *et al.*, 2016 which comprise of 0.4M potassium ferrocyanide as electrolyte and 1.0M potassium hydroxide as supporting electrolyte and to balance this potassium ion on the other side which comprises of <0.5M anthraquinone as electrolyte and 1.0M of potassium hydroxide. The choice of supporting electrolyte can be sodium chloride, potassium hydroxide or sulphuric acid. Sulphuric acid tends to react with $K_4Fe(CN)_6$ to produce hydrogen cyanide which is toxic to the environment, also the compatible electrochemical counterpart of sulphuric acid is hydrobromic acid which may be cheaper in terms of its cost and more effective in terms of power density but bromine is not environmentally friendly and not locally available (Chen *et al.*, 2015). Most of the pumps available locally use metal (iron-rich) as a working part, the choice of acid counterpart will

corrode the metal part and tend to reduce its efficiency and cost of operation by the formation of iron (ii) sulphate and iron (ii) bromine which is soluble in water. The choice of potassium hydroxide tends to prevent iron (or steel) from moving part of the pump going into solution because potassium ion is higher than iron in electrochemical series. If iron reacts with water at this condition as in case of side reaction, iron (I) hydroxide may be formed which is not soluble in water and thereby coating the surface of the metal and preventing further corrosion. The solubility and open circuit voltage of anthraquinone decreased as pH increased.

Making of Porous Clay Pot for Battery Membrane
Sawdust was obtained from FUTA's Obakekere forestry and wood technology workshop. To achieve fineness, the sawdust was sieved with a 600microns sieve (sieve No 30). In a 50/50 ratio by volume of clay to sawdust, fine sawdust was mixed with kaolin (white clay) obtained from FUTA's industrial design workshop in Obakekere. The clay was easy to work with at 50/50 and came out of the kiln strong enough to handle. Small amounts of water were added to the mixture and mixed in until it clumped together completely and was soft and workable. Once the mixture was workable, it was kneaded again to remove any remaining bubbles. The mixture was then shaped into a pot measuring 10cm by 10cm on the pottery wheel. After removing the pot from the pottery wheel, it was allowed to dry. The pot was taken to the kiln for firing once it had gained enough strength. The pot was taken out of the kiln and placed on a cooling rack to cool to room temperature. After that, the clay was soaked in water before being tested for porosity. Water was poured into the pot after soaking, and after about 30 minutes, water was dripping out of the pot, confirming its porosity.

Battery Construction and Testing: The electrodes—which were made of carbon graphite—the electrolyte, and the ion-exchange membrane were the main components of the battery. The cell was made up of two parts: a positive half-cell and a negative half-cell separated by a porous clay ion exchange membrane that prevented the electrolytes from mixing. An electrode made of carbon graphite was placed on each side of the cell. Two external tanks were used to store the electrolyte, anolyte, and catholyte. For each half-cell, the electrolyte was stored in a container and the electrode was dipped into the electrolyte. For testing and charging the cell, a wire was connected to the top of the electrodes, and the cell was charged with a 3V battery. The battery was made up of two chambers: the inner chamber had a volume of 400 cm³ and the outer chamber had a volume of 800 cm³. The inner chamber had one electrode with a surface area of 5 cm² and the outer chamber had five electrodes with a surface area of 25 cm², resulting in an equal ratio of electrode surface area to electrolyte volume. Clay was used as the separating medium, and it was made by mixing clay with line sawdust and firing it in a kiln, where the sawdust was burned away, leaving pore spaces

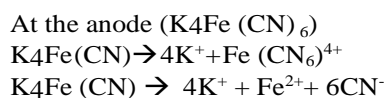
in the clay. The separating medium had a thickness of 0.35cm, and the electrodes in the outer chamber were connected in series and left for a while before being connected to the power source.



Fig 2: Operational set up

RESULTS AND DISCUSSION

The electrodes for the battery were initially made of aluminum and copper. 1.24A was measured as the open-circuit voltage. When a device (in this case a battery) is disconnected from any circuit, the open-circuit voltage (OCV) is the difference in electric potential between two terminals (in this case electrodes). There was no electric current between the terminals because there was no external load connected. The OCV and other measurements were taken by connecting a multimeter's probes to the wires connected to the electrodes and measuring the voltage that resulted. The battery produced a maximum discharge voltage of 1.25 with a charging current of 0.03A and a charging voltage of 1.5V for a few minutes, while the open circuit current was 0.12A. The battery was unplugged from the circuit and allowed to discharge for a day. The next day, a foamy reaction was observed between the electrodes and electrolyte on the anode and cathode sides, and the same reaction was observed when the electrodes were swapped. The electrolytes dissolved the copper and aluminum on both the anode and cathode sides, corroding it and making it poisonous to the environment. Despite this reaction, the battery still produced a current of 0.7A – 1.0A. For safety reasons, the copper and aluminum in the battery were replaced with carbon graphite, and the battery was charged for 30 minutes with a 3.0V power source. The charging current was 0.1 amps, and the power source was allowed to discharge for 1 minute with a maximum discharge voltage and current of 1.31 volts and 13 milliamps, respectively. The theoretical ionization in the medium is as follows



At the cathode (negative terminal) after charging
 $\text{Fe}^{2+} \rightarrow \text{Fe}^{3+} + e$



At the anode
 AQ = Anthraquinone
 $\text{AQ} + 2e \rightarrow \text{AQ}^{2-}$

Total open-circuit potential (OCP) voltage $E_{\text{cell}} = E_{\text{AQ}} - E_0 - E_{02} = 0.15 - (-0.76 - 0.33) = 1.24$

Table 1: Results from the battery test

Parameter	Value
Charging Current	0.09A
Charging Voltage	3.0V
Maximum Discharging Voltage	1.31V
Maximum Discharging Current	13MA
Testing Resistor	1Ω
Charging Time	30mins
Discharging Time	1min

Table 2: voltage vs capacity at zero-load and 330mW

Capacity	100%	90%	80%	70%	60%	50%	40%
Zero-Load	1.59V	1.44V	1.38V	1.34V	1.32V	1.30V	1.28V
330mW	1.49V	1.35V	1.27V	1.20V	1.16V	1.12V	1.10V

Voltage and Current Analysis: The transmission of power in the medium obeys beer-lambert law which states that radiant power P_0 , decreased to P_1 as it moves through the medium from 0 to concentration 'C' for portion 'b' of cell length, the power will change by the expression below;

$$P_1 = P_0 e^{-kcb} \quad (1)$$

Where k is a constant which depends on the conductivity of the medium. During the first test, the chamber had not assumed the same conductivity while during the second test, the chamber has assumed the same conductivity for anode electrolyte and membrane diffusion and cathode electrolyte. The conductivity of the medium was the same which showed that impurities had little effect on the chemistry of electro-active species since the quantity of charges generated or gain depend on contact areas between the charge and electrode areas, so the duration of dissipation of cell is large but the current is small and cannot be used to power any devices. Using a copper electrode placed at a distance of 1.0cm around the carbon electrode, it showed that a current of 0.8mA flowed through it to indicate an increase of 0.2mA after charging for 20 minutes with a current of 0.3A (0.006A/cm²). Area-specific resistance was made up of resistance of electrode, electrolyte and membrane, the reduction in current output was due to lack of saturation of the clay separating medium in the first test but the clay was left in the medium for two weeks. The current output increased 1.25mA to 43mA in the second test, it was, therefore, important to allow the clay membrane to be in contact with the medium before use. The average output at 0%,

50%, 70% SOC are 0.78V, 1.2V and 1.28V which are tallied with those obtained by (Lin *et al.*, 2016), the overall potential ranged from 60mV to 80mV depending on the level of impurity and saturation of the clay separating medium.

Conclusion: The challenge of power shortages in our communities necessitates a diversity of power sources to meet people's daily needs. After demonstrating that the extract from *morinda lucida* has a similar performance in alkaline solution to anthraquinone, the design and material chosen are based on the model described in the study and are similar to other flow battery designs. This study's alkaline medium is extremely safe, environmentally friendly, and cost-effective, demonstrating that the *morinda lucida* tree extract can be used as an electrolyte in the construction of a redox flow battery.

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