

Design of an experimental reactor for the selective and efficient recovery of lithium from waste battery cathode leaching processes

Diseño de un reactor experimental para la recuperación selectiva y eficiente del litio a partir de procesos de lixiviación de cátodos de baterías de desecho

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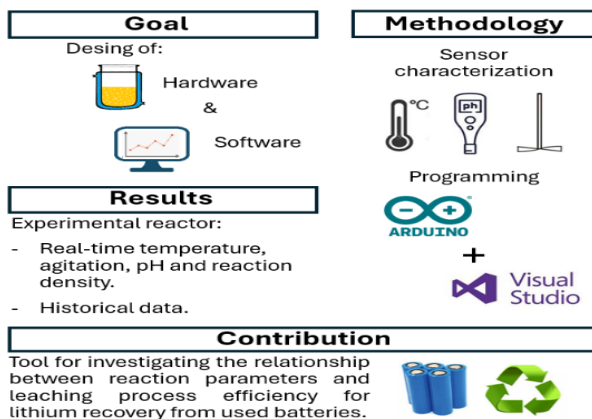


Abstract

This paper presents the design of an experimental reactor based on hardware and software, which monitors the reaction parameters of leaching processes for the recovery of lithium from waste battery cathodes. The hardware consists of a 250 ml double-layer reaction bottle, a magnetic stirring controller, and an Arduino-based system for measuring the temperature and pH of the reaction. The measured data is fed into the software, where it is processed to determine reaction parameter profiles. The experimental reactor will fulfill its purpose by correlating the selective lithium recovery efficiency with the different reaction profiles carried out, which will allow the identification of key variables, the establishment of mathematical models that describe the behavior of the reaction, and the determination of the conditions that improve the efficiency of the process.

Resumen

En este trabajo se expone el diseño de un reactor experimental basado en hardware y software, el cual monitorea los parámetros de reacción de procesos de lixiviación para la recuperación del litio a partir de cátodos de baterías de desecho. El hardware consiste en una botella de reacción de 250 ml de doble capa, un controlador de agitación magnética y un sistema basado en Arduino para la medición de temperatura y pH del medio en reacción. Los datos medidos son llevados al software, donde son procesados para determinar perfiles de parámetros de reacción. El reactor experimental cumplirá su propósito al relacionar la eficiencia de recuperación selectiva del litio con los distintos perfiles de reacción llevados a cabo, lo cual permitirá identificar las variables clave, establecer modelos matemáticos que describan el comportamiento de la reacción y determinar las condiciones que mejoran la eficiencia del proceso.



Reactor, Leaching, Lithium, Efficiency, Hardware, Software



Reactor, Lixiviación, Litio, Eficiencia, Hardware, Softwar

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Introduction

One of the greatest challenges facing the scientific community today in its various fields is approaching research from a sustainable development perspective [ONU, 2025], where essentially, the solution to a specific need does not compromise the ability to meet the potential needs of future generations [Sostenibilidad, 2025]. Globally, halting or even slowing climate change is one of the greatest needs of our time, and the energy transition is emerging as the most promising strategy for achieving this. However, in this context, significant global demand for lithium is expected [John D. Graham et al., 2021] as a key element in energy storage devices [P.E. Marín et al., 2021], with the environmental impact of meeting this demand solely through mining being evident.

A sustainable solution to the anticipated high demand for lithium is to recover this strategic mineral by recycling batteries that have reached the end of their useful life [Chunwei Liu et al., 2019; Min Yu et al., 2019; Jingbo Yang et al., 2021; Urias, P. M. et al., 2020; Fei Meng et al., 2020].

Recent research methods, such as leaching processes, have demonstrated high percentages of selective lithium recovery [Weiguang Lv et al., 2020]; however, testing has been limited mainly to modifying the initial experimental conditions and maintaining them throughout the test period [Qian Cheng et al., 2019], which excludes the possibility of evaluating the effects of variable conditions during the process on recovery efficiency.

This work promises to meet that expectation by monitoring leaching processes for selective lithium recovery in real time, evaluating efficiency under different experimental conditions.

Methodology

The hardware is the central element where the leaching process for selective lithium recovery takes place, and is designed on the basis of a 250 ml double-layer reaction bottle. As can be seen in Figure 1, the double layer is useful for circulating a fluid at a controlled temperature without coming into direct contact with the reaction solution, transferring heat to it, while the opening necks allow the entry of temperature and pH sensors, as well as reagents.

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Box 1

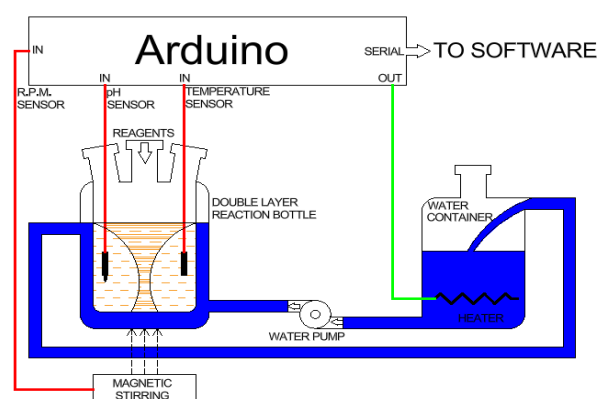


Figure 1

Schematic diagram of the hardware.

Source: Own elaboration.

A variable-speed magnetic stirring module has been designed to be placed under the reaction bottle. Temperature control and monitoring, as well as pH and stirring speed monitoring, are performed using an Arduino board.

The desired temperature is programmed into the Arduino code and measured using a sensor inserted through one of the opening necks of the reaction bottle, in direct contact with the solution. When the measured temperature is lower than the temperature programmed into the Arduino code, one of its outputs turns on the heater and turns off when the measured temperature is equal to the programmed temperature.

During the characterization of the temperature sensor, a commercial laser thermometer was used to perform the necessary calibration, as shown in Figure 2. The temperature is sent to the Arduino serial port within a character string, in the variable defined as “temp.”

Box 2



Figure 2

Calibration of the temperature sensor.

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The pH monitoring is carried out using a sensor inserted through another open neck of the reaction bottle, which was previously characterized using pH standards and test strips for proper signal processing within the Arduino code, as shown in Figure 3.

This parameter will be determined by the amount and concentration of the acidic and oxidizing agents introduced into the process, which must be done manually in this first version of the reactor. Once the corresponding pH value has been calculated, this signal is also sent to the serial port character string, in the variable defined as “pH.”

As for monitoring the agitation, the magnetic stirrer controller was previously characterized by measuring the speed as a function of the position of the control potentiometer, and the Arduino code calculates the revolutions per minute, which is also sent to the serial port character string, in the variable defined as “rpm.”

In the Arduino code, the variable “status” has also been defined to be sent within the character string; this variable indicates the status of the heater and is used in the software to turn the corresponding indicator on and off.

Box 3

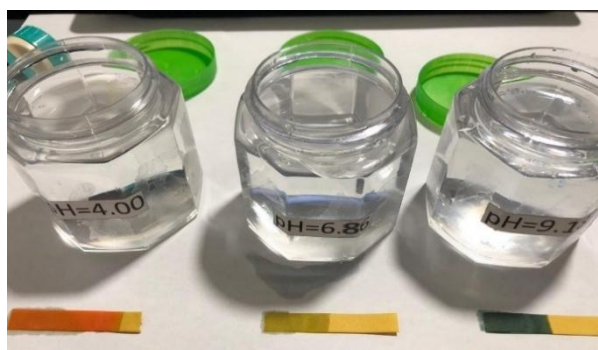


Figure 3

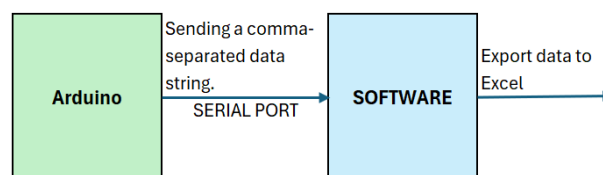
Standard solutions and reagent strips used during the characterization of the pH sensor.

Source: Own elaboration.

The software was developed in the Visual Studio C# programming environment, using the Windows Forms interface framework.

The objective of this development is to receive, graphically represent, and tabulate the information sent by Arduino through the serial port at a sampling frequency of 1 data string per second, as shown in Figure 4.

Box 4



Arduino code used:

```
Serial.println(String(temp)+","+)+(pH)+(",")++(rpm)+(",")++(status));
```

Figure 4

Serial communication between hardware and software.

The software separates the data from the received information string and processes it within the code for graphical representation and tabulation. The reaction time parameters and solution density are calculated within the software code.

Results

Figure 5 shows the hardware assembly, where the reaction bottle has been secured to an aluminum support using a clamp. A black container with an internal heater adapted to it can also be seen. From this container, and through a pump, the fluid circulates through the outer layer of the reaction bottle at a programmed temperature.

The temperature and pH sensors are placed inside the inner layer of the bottle, and at the rear, a metal cabinet can be seen housing the Arduino board along with all the circuits necessary to power each hardware component.

Box 5



Figure 5

Hardware assembly

Figure 6 shows the section of the software developed where the quantity and type of reagents added to the reaction bottle are entered, the main data being the quantity in grams of NMC cathode material to be leached.

The software allows for the registration of any addition of reagent that is desired to be made after the start of the test, indicating the corresponding amount and pressing the "Add" button for each action. In Figure 7, a graph of the data exported to Excel from a simulation is shown, where 15 grams of NMC Cathode material are added to 50 ml of deionized water 10 seconds after the process begins, showing how the calculated density changes from 0 g/l to 300 g/l; subsequently, when 5 grams of a solid reagent are added 10 seconds after the first action, the density is recalculated to 400 g/l; on the contrary, if liquid reagents are added, the density decreases, as observed in the graph in Figure 8, where the addition of 10 ml of a liquid reagent is simulated 31 seconds after the process starts.

Box 6

Real Time Parameters

MASS PARAMETERS:

WNCM: 15 g Add 15 g

Reagent 1 5 g Add 10 g

Total added mass = 25 g

VOLUME PARAMETERS:

Water: 50 ml Add 50 ml

Reagent 2 10 ml Add 20 ml

Reagent 3 10 ml Add 10 ml

Total added liquid = 80 ml

Slurry density = 312.5 g/l

Figure 6

Reagent data section of the software.

Box 7

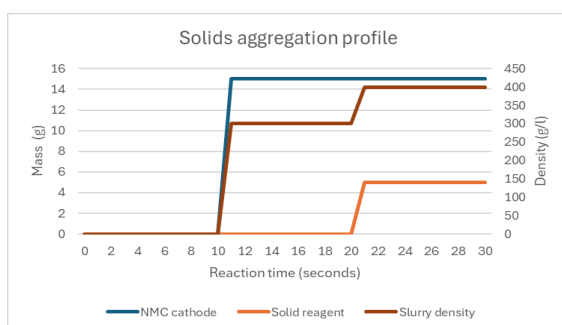


Figure 7

Graph of solid reactants aggregation profile.

Box 8

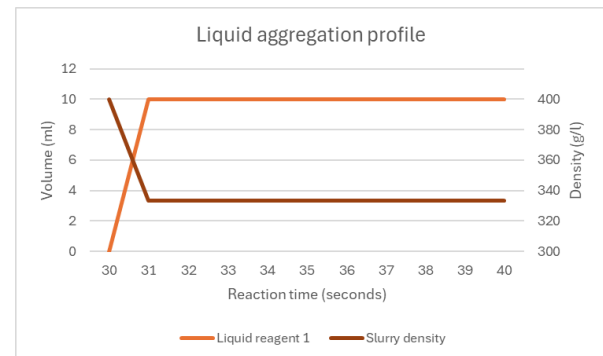


Figure 8

Profile graph of liquid reagent aggregation.

Finally, Figure 9 shows the complete software window, displaying the temperature and pH graphs throughout the reaction time, as well as the table with historical data for all parameters involved in the selective lithium leaching processes described in the literature

Box 9



Figure 9

Software window of the experimental reactor

Conclusions

This article presents the design, construction, and performance testing of an experimental reactor based on hardware and software, which promises to be a valuable tool for studying the efficiency of methods for recovering lithium from cathode materials in used batteries through leaching processes.

It was confirmed that the experimental reactor developed can monitor in real time each and every one of the parameters involved in the leaching processes described in the literature, allowing a future version to modify the experimental conditions with actuators that dose the reagents to control the pH and density of the solution, as well as vary the temperature and agitation with specific reaction profiles, etc.

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This can be done after using the experimental reactor in its first phase of development to investigate the relationship between changes in experimental parameters during the process and lithium recovery efficiency, moving towards the development of a reactor that is intelligent in every sense of the word.

Declaracions

Conflict of interest

The authors declare no interest conflict. They have no known competing financial interests or personal relationships that could have appeared to influence the article reported in this article.

Author contribution

Herrera-Gutiérrez, Hugo: Development of hardware and software, writing [review and editing].

Cisneros-Villalobos, Luis: Conceptualization, methodology, and supervision.

Torres-Islas, Álvaro: Formal analysis and research.

Saldarriaga-Noreña, Hugo Albeiro: Formal analysis and research.

Availability of data and materials

The original contributions presented in this study are included in the article. For more information, please contact the corresponding author.

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Abbreviations

ml	Milliliter
NMC	Nickel-Manganese-Cobalt
Ph	Hydrogen Potential
g	Gram
g/l	Gram per liter

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Basics

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