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ORIGINAL ARTICLE

Safety considerations for hydrometallurgical metal recovery from lithium-ion batteries

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Abstract

This paper presents a comprehensive overview of the critical process safety considerations inherent in hydrometallurgical metal recovery within the lithium-ion battery (LiB) recycling process. As hydrometallurgy application in LiB recycling is still in the early stages of development, it is crucial to identify the hazards and provide safety recommendations. Hazards related to hydrometallurgy are identified and categorized in process, toxic, fire, explosion, corrosion, environment, storage, and transport hazards. Risk reduction measures are suggested using the hierarchy of control methodology to eliminate and reduce risks to as low as reasonably practicable (ALARP), based on UK regulatory framework.

KEYWORDS

hazard, hydrometallurgical, lithium-ion battery, metal recovery, process safety, recycling

1 | INTRODUCTION

Lithium-ion batteries (LiBs) can be used in various applications ranging from portable electronic devices to energy storage.^{1,2} The demand for LiBs is seen to be rising rapidly to meet the global net zero targets.³ By 2050, an increase of 80%–90% is predicted in the automobile industry⁴ and a significant rise is expected in the use of LiBs in storage of renewable energy across the globe.⁵

With an increase in demand for LiBs, the demand for constituent metals, that is, Li, Ni, Mn, and Co, would also increase. These metals are not abundant in many regions of the world, especially in the UK and USA, and mining for these metals is not sustainable.^{6,7} Therefore, resulting in increasing focus on recycling and closed loop economy solutions.⁸ Recycling of LiB also helps reduce waste batteries from

being disposed unsafely or being accumulated as waste on battery collection and landfill sites.^{9,10} Improper storage or handling of waste batteries can result in thermal runaway or ignition.^{11–13}

Recycling of LiB is completed in three main stages, namely, dismantling, separation, and metal recovery, as shown in Figure 1. Dismantling and separation of batteries are widely undertaken across the globe; however, metal recovery is still in the early stages of development.

One of the techniques being researched for metal recovery is hydrometallurgy.^{15,16} As this is mainly being researched in small-scale laboratories, the severity of the hazards in research facilities is small. As these techniques start to be commercialized and larger scale facilities are installed and operated, the severity of the hazards would increase exponentially.^{17,18}

Past incidents show that hazards, such as thermal runaway, ignition of battery, explosion, and so forth, exist in the storage, pretreatment, and separation processes.¹¹ No incidents were identified relating to use of hydrometallurgy in LiB recycling, with only limited information available on the process safety issues.^{15,19}

Therefore, there is an opportunity for proactive approach to integrate inherent safety in design, operation, and maintenance of these

Abbreviations: ALARP, as low as reasonably practicable; BAT, best available technique; BREF, BAT reference; CCPS, center for chemical process safety; CRW, chemical reactivity worksheet; EBRA, European Battery Recycling Association; HSE, health and safety executive; LEL, lower explosion limit; LiB, lithium-ion battery; MIE, minimum ignition energy; MSDS, material safety datasheet; RRM, risk reduction measure; SMS, safety management system; UEL, upper explosion limit; UK, United Kingdom.

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recycling facilities.^{17,18} This may be achieved by collaboration between greenfield sites, researchers, and regulatory bodies.

This paper summarizes the process safety issues associated with the use of hydrometallurgy in recycling of LiBs. All background information, such as methodology, material data, and regulatory data, related to this paper is provided in an online supplement.

2 | PROCESS OVERVIEW

LiB recycling consists of three main steps, as shown in Figure 1. Hydrometallurgy is a metal recovery technique used for the recovery of cathode material. It is a pH and temperature-sensitive process, where chemicals and solvents are used to recover metals from cathode metal slag (also known as black powder). The metal slag is formed in the preceding material separation step, through pyrometallurgy or thermal processing.

Leaching is the first step in hydrometallurgy, during which, acid, bases, or specialist chemicals are used for reaction with the black powder.^{14,20} This separates out the powder into its constituent metals and creates metal salts in the leaching solution. Individual metal is extracted and precipitated out from the leaching solution to obtain metal salts or pure metals. Additional acids, bases, or specialist chemicals are used in the extraction and precipitation stages to adjust pH of the leaching solution, based on their selectivity to certain metals.^{15,21} The constituent metals may be recovered in multiple stages, therefore, making hydrometallurgy a series of batch reactions. The reaction temperature may be varied to increase the recovery efficiency of metals of interest.

Figure 2 gives a basic block flow diagram for the hydrometallurgy process studied in this project.

3 | HAZARD IDENTIFICATION

The hazard study technique recommended by Tony Ennis⁶ has been used together with detailed research on hydrometallurgy process and associated materials and their hazards. An in-depth review of the best available techniques, policies, and regulations was completed to recommend risk reduction measures (RRMs) for future commercialization of the use of hydrometallurgy in LiB recycling.

3.1 | Chemical hazards

Table 1 summarizes the common leaching agents, extractants, precipitants, and recovered metals associated with hydrometallurgy.^{14,21–24} However, hydrochloric acid, sulfuric acid, sodium hydroxide, sodium carbonate, hydrogen peroxide, D2EPHA, Cyanex 272, and PC 88-A are noted to be widely used chemicals.

The hazards associated with these chemicals can be categorized into toxic, fire, explosion, corrosivity, environmental, storage, and transport hazards.

3.1.1 | Toxic hazards

Reaction between the metal slag and leaching agent, extractants, or precipitants results in the formation of a metal salt and the generation of gases. Depending on the chemical agents used, toxic gases, such as sulfur dioxide or chlorine, can be released from the reaction.²⁵ For example, use of HCl leads to the generation of Cl₂ gas or the use of H₂SO₄ generates SO₂ gas.

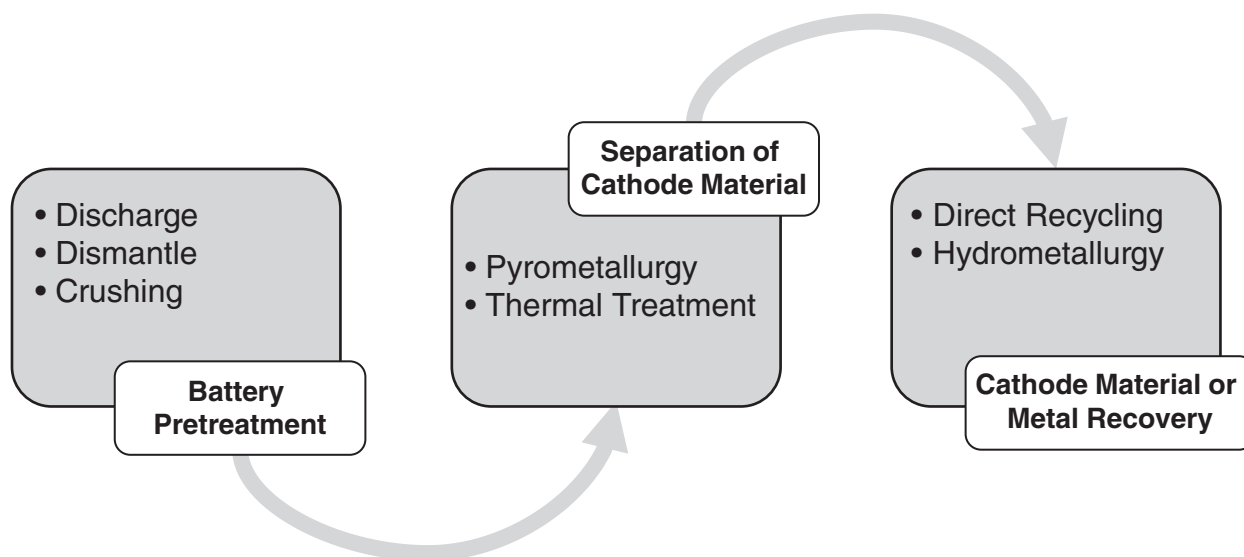


FIGURE 1 Generic stages of LiB recycling.¹⁴

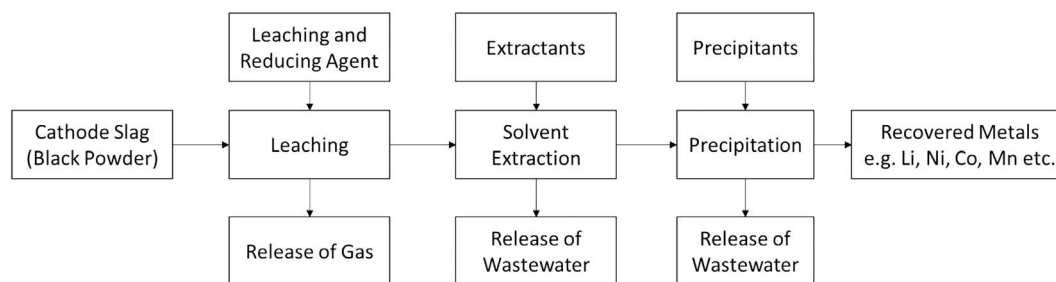


FIGURE 2 Basic block flow diagram without risk reduction measures.

Some of the raw materials and products have been noted to have toxic properties and have associated workplace exposure limits, which must be adhered to during plant operations (e.g., nickel, manganese, lithium, and hydrogen peroxide).

3.1.2 | Fire and explosion hazards

From study of the fire and explosion parameters, that is, MIE, flash points, autoignition or decomposition temperature, flammability or explosion limits, and allowable surface temperatures for common chemicals, it is noted that the fire and explosion hazards are inherent to many materials involved in hydrometallurgy. Explosion hazards may also exist during storage and handling of raw materials and final products in solid or dust form. Some flammable or explosive materials include acetic acid, formic acid, Cyanex, D2EPHA, cellulose, maleic acid, and manganese.

3.1.3 | Corrosivity hazards

The corrosion hazard is inherent to acids and bases, such as H_2SO_4 or NaOH , which are commonly used in hydrometallurgy. Corrosion over time can damage the pipework and equipment, leading to loss of containment over time. Prolonged leaks of corrosive materials can also lead to damage to the surrounding areas, such as damage to concrete in bunds, flooring (concrete or metal), vessel supports, and so forth, which can escalate into a significant incident. Risk to operators due to corrosion may exist during handling of these materials, for example, coming into contact with surfaces with leaks or during manual handling of containers etc.

3.1.4 | Environmental hazards

The toxicity and corrosiveness of the chemicals also impact the environment. Release of substances such as lithium, H_2SO_4 , HCl , and so forth, into the water drains or nearby water bodies may lead to significant harm to aquatic life. Fire at vent stacks may also result in harm to nearby personnel or arial environment. The hydrometallurgy process also generates a significant amount of wastewater, which may be contaminated with hazardous leaching agents, extractants, precipitates,

and trace amounts of unrecovered products. This harmful wastewater may enter surface water drains, which if not managed can be drained into local water bodies. Uncontained wastewater may also run off onto the vulnerable offsite facilities and population, for example, nearby farmland, schools, offices, and so forth.

3.1.5 | Storage and transport hazards

Many of the hazardous chemicals used in hydrometallurgy require specific storage conditions, such as dry places, lower ambient temperatures, or segregation with incompatible materials. The common requirement between all substances is tightly closed containers with authorized access.

Risk may exist during transport of raw materials and product to and from site.²⁶ During transport, the substances may be carried through off-site areas, such as public motorways, railway lines, and so forth. In addition, there is a limited level of control to prevent uncontrolled releases into the environment. Another aspect of transport is the transfer of products into storage vessels at consumer sites. Transfer of products from transport vessels and storage vessels may include manual steps such as connection/disconnection, purging/draining of hoses, or starting/stopping the transfer, which requires specialist training. Any errors during these tasks and activities can lead to a process safety incident.

3.2 | Compatibility hazards

Hydrometallurgy process is highly dependent on reaction kinetics and the concentration of the solutions. Therefore, there is inherent incompatibility between the raw materials or products in use. Any loss of control of concentration and therefore, the pH, may lead to production of harmful by-products. Using the CRW software, compatibility charts were created for the common chemicals used in the hydrometallurgy process.

From the compatibility study, it is noted that all substances have varying levels of incompatibility between them. It is, however, noted that phosphoric acid and DES leaching solvents, such as urea and thio-urea, are the most compatible with other solvents and agents. Figure 3 shows that the components used in the LiB at their elemental levels may react with each other releasing heat.

TABLE 1 Common leaching agents, extractants, precipitants, and products from hydrometallurgy of LiB.

Materials	Key hazards	Type
Hydrochloric acid, HCl	Corrosive	Leaching agent
Sulfuric acid, H ₂ SO ₄	Corrosive	Leaching agent Reducing agent Extractant
Phosphoric acid, H ₃ PO ₄	Corrosive	Leaching agent
Sodium hydroxide, NaOH	Corrosive	Leaching agent Extractant Precipitant
Ammonia, NH ₃	Toxic	Leaching agent
Oxalic acid	Toxic	Leaching agent
Citric acid	Irritant	Leaching agent
Maleic acid	Toxic	Leaching agent
Choline chloride	Not hazardous	Leaching agent
PEG200 (polyethylene glycol)	Not hazardous	Leaching agent
Urea	Not hazardous	Leaching agent
Thiourea	Toxic	Leaching agent
Grape seed	Irritant	Leaching agent
Hydrogen peroxide, H ₂ O ₂	Flammable/toxic	Reducing agent
Ethylene glycol	Toxic	Reducing agent
Sodium carbonate, Na ₂ CO ₃	Irritant	Extractant Precipitant
Dimethylglyoxime (DMG)	Flammable	Extractant
(4,4-trimethylpentyl) phosphinic acid (Cyanex 272)	Irritant	Extractant
(2-ethylhexyl) phosphonic acid mono-2-ethylhexyl ester (PC-88A)	Corrosive/toxic	Extractant
D2EHPA	Toxic/corrosive	Extractant
Versatic 10 acid	Not hazardous	Extractant
Potassium permanganate, KMnO ₃	Corrosive/toxic	Precipitant
Lithium	Explosive in dust Form/corrosive	Product
Lithium carbonate	Toxic	Product
Lithium sulphate	Toxic	Product
Cobalt	Flammable	Product
Cobalt sulphate	Carcinogenic	Product
Cobalt oxide	Carcinogenic	Product
Nickel	Carcinogenic	Product
Nickel sulphate	Toxic	Product
Nickel hydroxide	Toxic	Product
Manganese	Not hazardous	Product
Manganese hydroxide	Toxic	Product

Another aspect is the compatibility between the substances and material of construction as not all materials are suitable for all substances, pH, or temperature conditions. In addition, in hydrometallurgy, the pH and temperature are continuously varied, to change product selectivity and efficiency. Use of unsuitable material for construction may lead to loss of containment from equipment or an unwanted reaction between the construction material and process fluid.

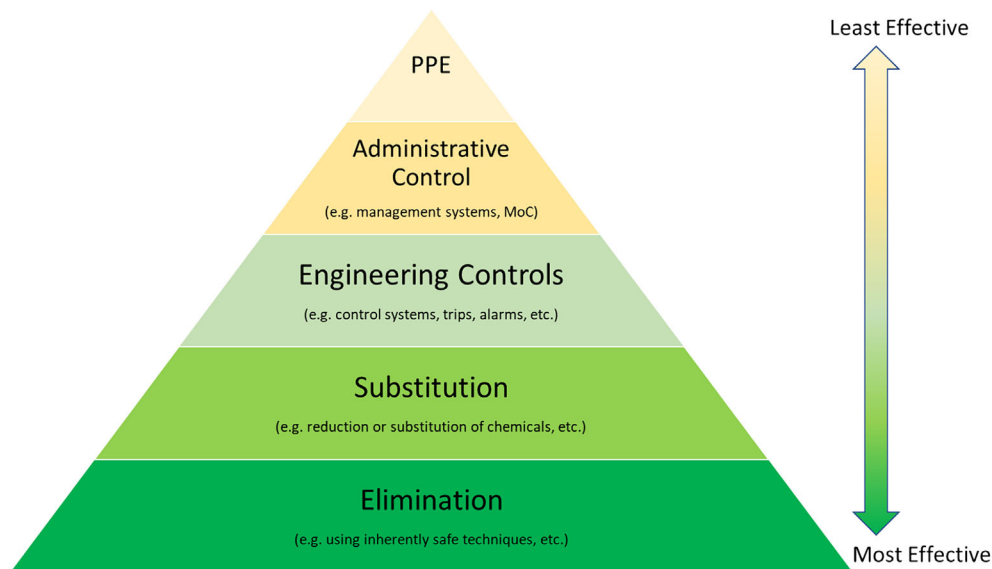
3.3 | Process hazards

Loss of pH and/or temperature control is identified to be the two main processing hazards.²⁷ Loss of pH or temperature control may result from control system failures and may lead to unwanted reactions or thermal runaway. Changes in pH may escalate the compatibility hazards, as discussed in Section 3.2. Increase in temperature may

FIGURE 3 CRW software compatibility matrix output pure elements used in LiB (compatibility matrix legend: Y—compatible, C—caution).

Mixture Manager				Mixture Report		Compatibility Chart			Reactive Groups		Cust Chem	
Print Chart												
Export Chart Data												
NFPA		Chemical Pairs										
Health	Flammability	Instability	Special	Mix 2 Compatibility Chart								
				ALUMINUM POWDER, UNCOATED	COBALT	COPPER	LITHIUM ATOM	MANGANESE	NICKEL			
0	3	1		ALUMINUM POWDER, UNCOATED	Y							
				COBALT								
				COPPER	C	C						
3	2	2	No	LITHIUM ATOM	C	C	C					
				MANGANESE	C	C	Y	C				
2	4	1		NICKEL	Y	Y	C	C	C			

FIGURE 4 Hierarchy of control adapted from NIOSH.³⁸



result in flammable or explosion hazards, as discussed in Section 3.1.2, if the temperature reaches the auto-ignition temperatures.

3.4 | Risk reduction measures

BREF documents issued by the European Commissions have been used to identify best practices.²⁸⁻³⁷ Hierarchy of control (Figure 4) may be used to select appropriate RRM for a facility.

3.4.1 | Elimination

Use of another inherently safer metal recovery technique, which is not pH or temperature dependent, may be considered to eliminate the use of hydrometallurgy. If not possible, consideration should be given to choosing inherently safer non-hazardous chemicals, where possible for leaching, extraction, and precipitation. For example, use of ammonium sulfate, aluminum hydroxide, ascorbic acid, cellulose, choline chloride, glucose, sodium thiosulfate, sodium sulfate, PEG200, sucrose, urea, or Versatic 10 Acid.

3.4.2 | Substitution

Substitution measures should include the use of substances with high ignition temperatures, with flash point above the surface temperatures and worst-case temperatures expected on loss of control. Recovery of metals in their stable phase or form, for example, recovering lithium as lithium carbonate, which can be directly reused as a cathode material, can also be considered. Another substitution technique is minimization, for example, reducing the number of flanges or joints will reduce the leak points or reducing the storage inventory stored or transported to/from site to reduce the severity of a loss of containment event. Minimization can also be achieved through continuous operations instead of batch processes to reduce the capacity of reactors or vessels, hence, reducing the severity of the event. Optimization of water and energy use and maximizing reuse and recycle of wastewater will minimize the use of freshwater.

3.4.3 | Engineering controls

Engineering controls, such as pH, temperature, pressure, speed, and level control systems as per IEC 61508 guidance, active safety systems (relief devices, quenching systems, etc.) or passive safety systems (blast walls, bunds, dikes, etc.) should be considered. Suitably designed interceptors on sites effluent and surface water drainage system, scrubber systems or incinerators on vent or relief device discharge, dust prevention systems, anti-towaway or breakaway connections, and barriers in the import/export area are other forms of engineering controls.

Other design requirements include control of ignition or heating sources, safe location of vent and relief outlets, site layout, material separation and segregation, and selection of suitable material of

construction for equipment with consideration given to fatigue due to pH–temperature cycles and worst-case ambient conditions.

3.4.4 | Administrative controls

Recyclers should ensure compliance with industry good practices, regulations, and policies, including guidance on workplace safety measures. This can be achieved through robust and well-defined safety management systems (SMS), including process safety, environment and energy efficiency, and completion of a suitable and sufficient risk assessment.

SMS should be supported by well-written procedures for routine and non-routine operations, operator competency and training programs, periodic monitoring and trending of process conditions, and any emissions and development of emergency plans with onsite and offsite requirements.

3.4.5 | Personal protective equipment

Personal protective equipment (PPE), such as fire-resistant overalls, Hi-Viz jackets, hard hats, safety boots, safety glasses, hearing protections, and safety gloves, should be used during routine or non-routine operations. Specialized PPE, such as personal gas monitors, safety showers, eye washing stations, gas masks, and breathing apparatus, should also be considered using an appropriate risk review of the facility.

3.4.6 | Summary of RRM

Figure 5 provides a recommended process block flow for hydrometallurgical metal recovery from LiBs.

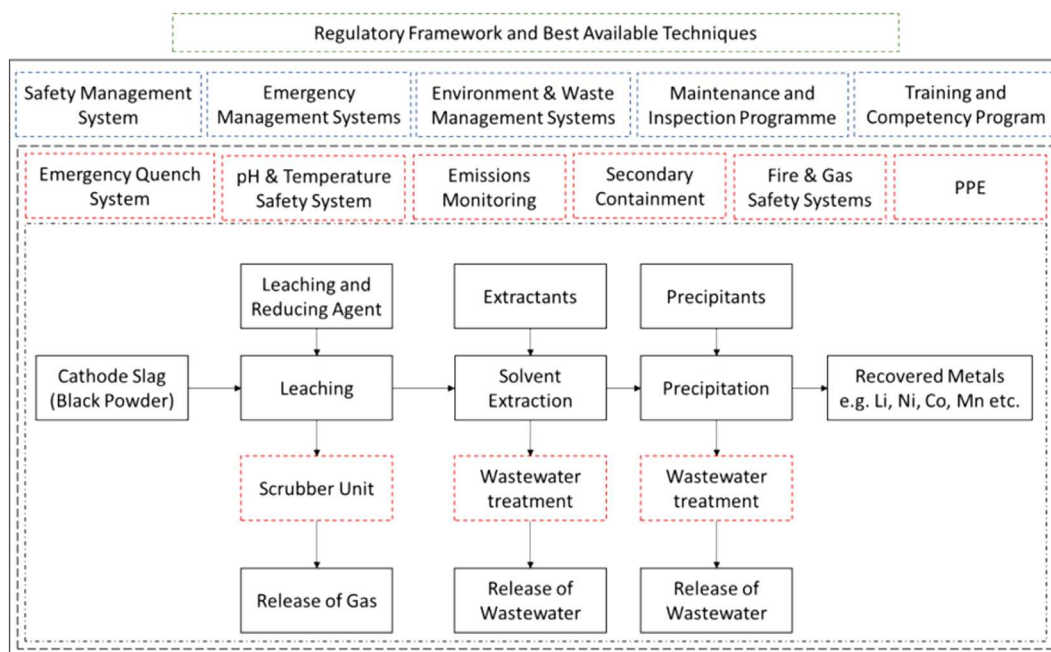


FIGURE 5 Recommended process block flow diagram with suggested risk reduction measures.

4 | CONCLUSION

Recycling of LiB is of utmost importance to meet the increasing demand. This is especially important in countries like the UK, where battery materials, such as Li, Ni, Co, and Cu, are not naturally occurring. The overall recycling process, including hydrometallurgy, can result in potential accidents if the recyclers do not manage the risk suitably and sufficiently. Therefore, the most critical finding of this work to enhance process safety is the crucial importance of establishing a robust process safety management system right from the outset when risks are more manageable. This paper identifies the common hazards and recommends RRM, based on the hierarchy of control, which can be used to mitigate and reduce the risks to ALARP. Future work should consider collaborating with greenfield sites and regulatory bodies to identify best practices for hydrometallurgical recovery of metals from LiBs.

AUTHOR CONTRIBUTIONS

Sakshi Jain: Conceptualization (equal); data curation (equal); formal analysis (equal); investigation (equal); methodology (equal); resources (equal); software (equal); validation (equal); visualization (equal); writing – original draft (equal). **Seyed Mojtaba Hoseyni:** Conceptualization (equal); data curation (equal); formal analysis (equal); methodology (equal); supervision (equal); validation (equal); visualization (equal); writing – original draft (equal); writing – review and editing (equal). **Joan Cordiner:** Conceptualization (equal); methodology (equal); project administration (equal); supervision (equal); validation (equal); visualization (equal); writing – review and editing (equal).

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DATA AVAILABILITY STATEMENT

The data that supports the findings of this study are available in the supplementary material of this article.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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