



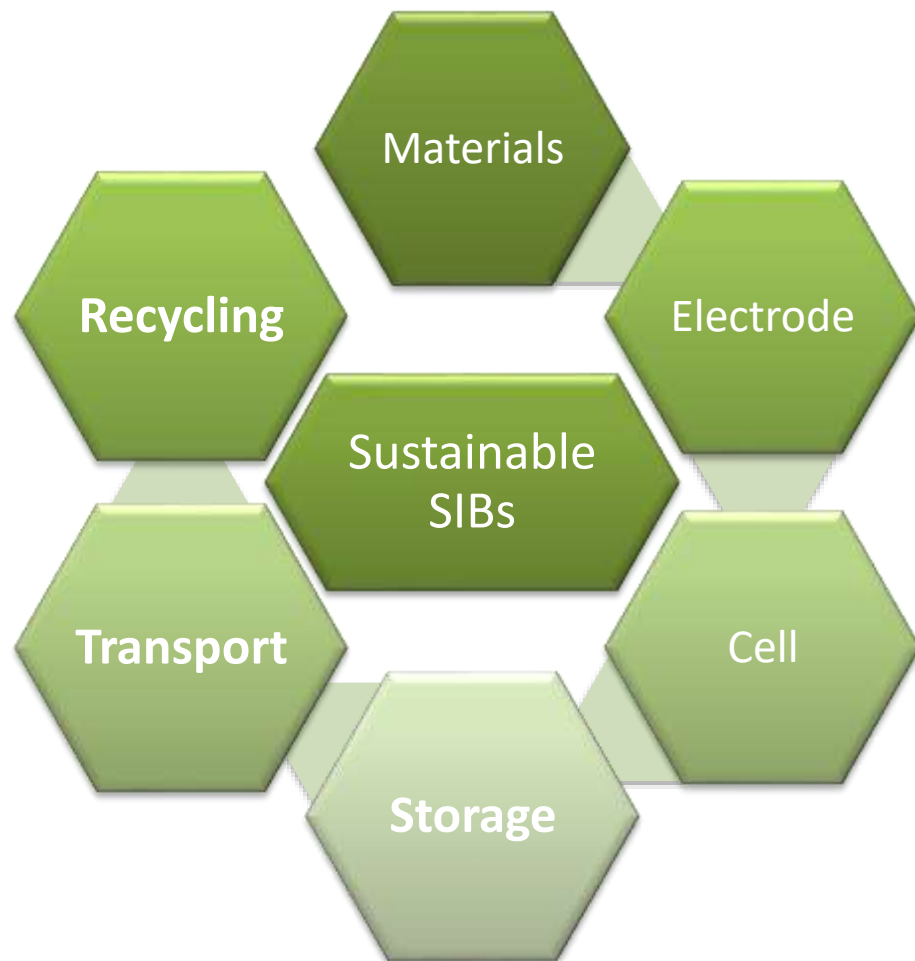
Safety and Transport Standards and Recycling Steps

SIMBA AB Workshop
20th of July 2022

University of Birmingham
Yazid Lakhdar



Introduction



WP1: Specification of KPIs and use cases including reuse and recycling

- + **T1.2** Regulations and Standards
(Lead: FHG, partners: Altris, SAFT, UBham) [M6-M42]
- + **T1.4** Recycling and Reuse
(Lead: TES-Recupyl, partners UBham) [M24-M36]

WP6: Circular economy Cell and Material lifecycle design

- + **T6.1** Design for disassembly, separation and recycling
(Lead: Recupyl, partners UBham) [M3-M36]
- + **T6.2** Reclamation and Reuse
(Lead: UBham, partners Recupyl, Altris) [M18-M42]
- + **T6.3** Life cycle assessment (LCA) analysis
(Lead: KIT, partners UBham, Recupyl) M36-M42

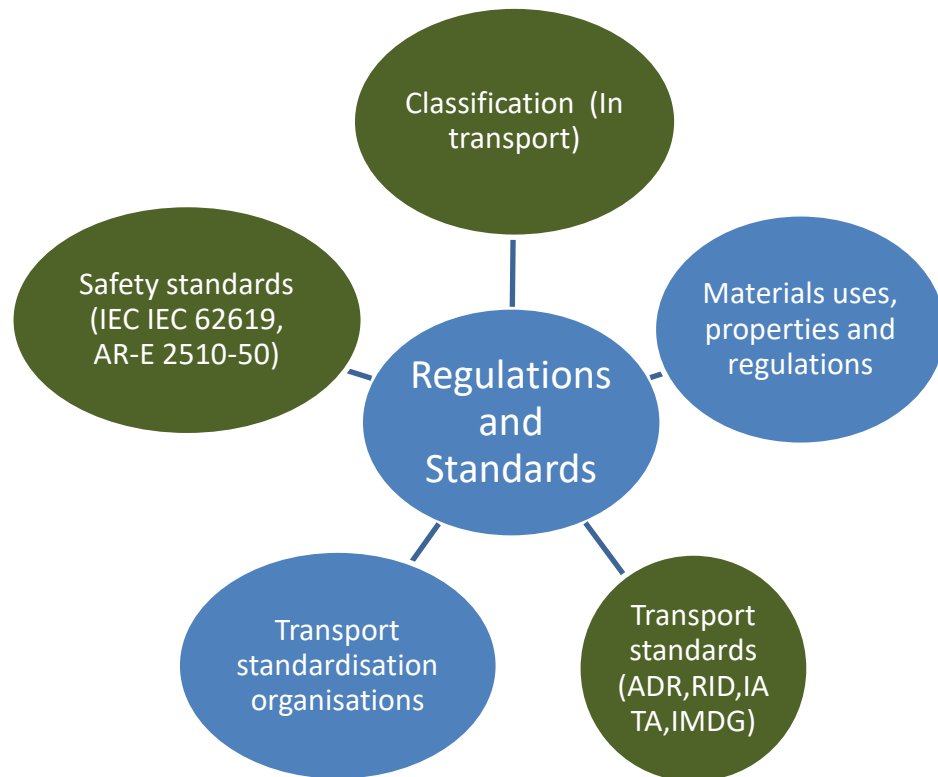


SAFETY AND TRANSPORT STANDARDS

- 1) Transport standards and regulations - *WP1*
- 2) Battery configuration, from liquid to solid electrolyte - *WP2*
- 3) Safe state-of-charge transport - *WP5*

T1.2 Regulations and Standards

- + **T1.2 Regulations and Standards (Lead: FHG partners: Altris, SAFT, UBham) [M6-M42]**
- + Objective: Collate and disseminate in a report EU, UK and Global regulations and standards for materials, safety and recycling for solid state batteries. Handling, manufacturing and application standards. (M18)



- + **D1.2 M18 – Report on T1.2**
- + **Rail and road** : sodium-ion batteries with liquid electrolytes are treated the same as lithium-ion batteries ([ADR M340](#), [RID 6/2021](#))
- + **Materials**: [PAS 7062:2021](#) electrode and cells code of practice health and safety, environmental and quality management consideration in cell manufacturing and finished cell.
- + **Battery Level Relevant Standards**
- + **High temperature sodium batteries** - [IEC 62984-3:2020](#), Labelling and marking - [IEC 62902](#), ADR – batteries containing sodium - [UN 3292 Class 4.3](#), Overview of battery safety testing (LER and ESS) - [UL 1973](#)

Batteries and ADR



+ Task 6.1 Design for disassembly, separation and recycling (M3-M36)

+ 6.1.1 Storage, Transport Safety Base Line Cell (BLC) recycling Cell



International organizations relevant to shipment of batteries	International Organization Europe connection	International Organization out of Europe
United Nation Recommendation of the Transport of Dangerous Good , 13th revised Ed. 2003		ASEAN countries: based on ADR but completed with a Framework for Facilitation of Goods transit
European Agreement International Carriage of Dangerous Goods by Road (ADR) ECE/TRANS/175, 2005	For ADR: UNECE Inland Committee WP15, Geneva	MERCOSUR Countries: Agreement based on ADR-RID
European Agreement International Carriage of Dangerous Goods by Inland Waterway (ADN) ECE/TRANS/172, 2005	For ADN: UNECE Joint Meeting of Expert Regulation, Geneva	ANDEAN countries : legislation in preparation based on ADR
Regulation concerning International Transport of Dangerous Goods by Rail (RID) OTIF	For RID: OTIF Safety Committee, Bern	

+ Batteries are under ADR regime:

- + Identification of the risk
- + Fitting with the existing list and classification
- + By the electrolytes (corrosives class 8)
- + By the reactive metals like Li (class 9)
- + By the solvent or organic electrolytes (class 4?)

+ What needed if under ADR ?

After Classification and Identification of the risk

- + Adapted and approved packaging,
- + Adapted procedures,
- + Transport operation requirements, documents, signals, training, company management...

Identification	Class 1	Class 2	Class 3	Class 4	Class 5	Class 6	Class 7	Class 8	Class 9
	Explosive materials	Gases	Flammable liquids	Flammable solids	Peroxides activating fire	Toxic materials	Radio elements	Corrosives products	Other dangerous products
Labels									
				Class 4 = Battery emit flammable gas in contact with water. High energy storage batteries Containing sodium					Class 9 = Miscellaneous dangerous substances: UN 3090 Lithium batteries UN3091 Lithium batteries contained in equipment or batteries packed with equipment Lithium, lithium ions, lithium alloys based batteries
				UN 3292 Batteries containing Sodium (Actually only for Na batteries { Zebra })					

Updates to standards relevant to the shipment of batteries

- + United Nation Recommendation of the Transport of Dangerous Goods ~~13th revised Ed. 2003~~ → ST/SG/AC.10/1/Rev.21
- + European Agreement International Carriage of Dangerous Goods by Road ([ADR](#)) ~~ECE/TRANS/175, 2005~~ → ECE/TRANS/300, 2020
- + European Agreement International Carriage of Dangerous Goods by Inland Waterway ([ADN](#)) ~~ECE/TRANS/172, 2005~~ → ECE/TRANS/301, 2021
- + Regulation concerning International Transport of Dangerous Goods by Rail ([RID](#)) OTIF

Very few mentions of SIBs in ADR

# of mentions of ↓ in →	ADR vol1 &2	ST/SG/AC.10/1/Rev.21
Battery / batteries	900+	245
Lithium batteries	90	26
Lithium ion	63	31
Lithium metal	101	29
Sodium batteries	10	4
<i>Sodium ion</i>	0	0
<i>Sodium metal</i>	0	0

Vehicles powered by a fuel cell engine shall be assigned to the entries UN 3166 VEHICLE, FUEL CELL, FLAMMABLE GAS POWERED or UN 3166 VEHICLE, FUEL CELL, FLAMMABLE LIQUID POWERED, as appropriate. These entries include hybrid electric vehicles powered by both a fuel cell and an internal combustion engine with wet batteries, sodium batteries, lithium metal batteries or lithium ion batteries, carried with the battery(ies) installed.

Other vehicles which contain an internal combustion engine shall be assigned to the entries UN 3166 VEHICLE, FLAMMABLE GAS POWERED or UN 3166 VEHICLE, FLAMMABLE LIQUID POWERED, as appropriate. These entries include hybrid electric vehicles powered by both an internal combustion engine and wet batteries, sodium batteries, lithium metal batteries or lithium ion batteries, carried with the battery(ies) installed.

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377 Lithium ion and lithium metal cells and batteries and equipment containing such cells and batteries carried for disposal or recycling, either packed together with or packed without non-lithium batteries, may be packaged in accordance with packing instruction P909 of 4.1.4.1.

These cells and batteries are not subject to the provisions of 2.2.9.1.7 (a) to (g).

Packages shall be marked "LITHIUM BATTERIES FOR DISPOSAL" or "LITHIUM BATTERIES FOR RECYCLING".

Identified damaged or defective batteries shall be carried in accordance with special provision 376.

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If a vehicle is powered by a flammable liquid and a flammable gas internal combustion engine, it shall be assigned to UN 3166 VEHICLE, FLAMMABLE GAS POWERED.

Entry UN 3171 only applies to vehicles powered by wet batteries, sodium batteries, lithium metal batteries or lithium ion batteries and equipment powered by wet batteries or sodium batteries carried with these batteries installed.

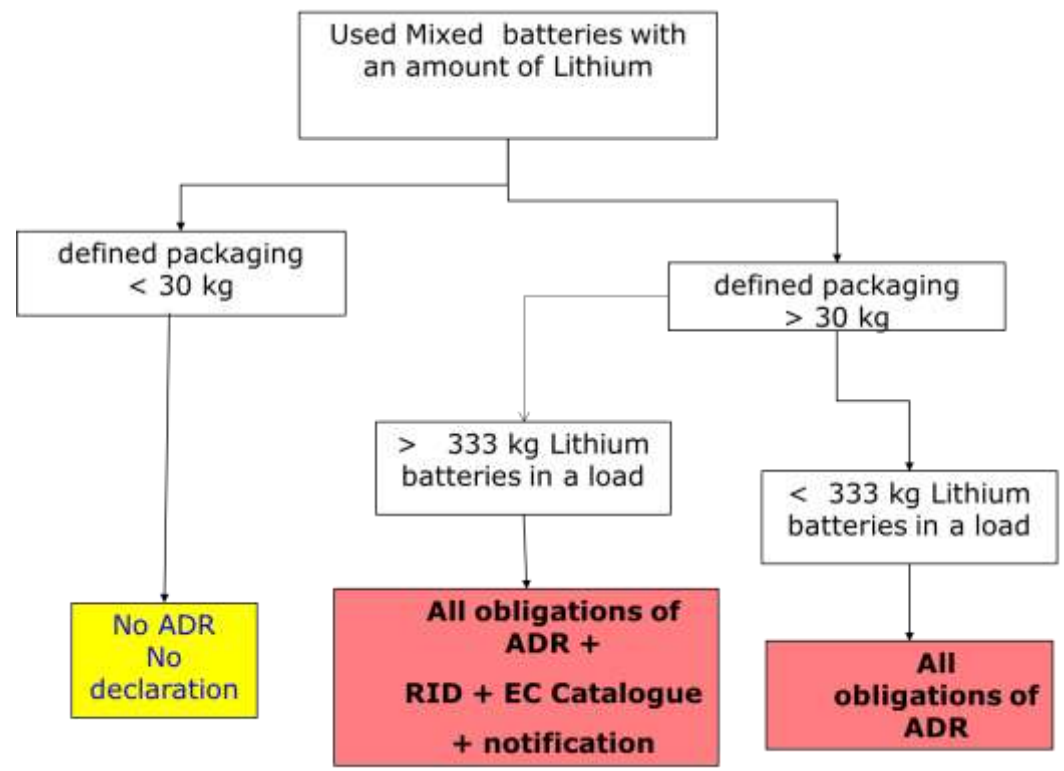
Packaging of used batteries

+ Task 6.1 Design for disassembly, separation and recycling (M3-M36)



+ 6.1.1 Storage, Transport Safety Base Line Cell (BLC) recycling Cell

ADR Regulation and mixed batteries from primary collection
(FINAL TEXT : ECE-TRANS-WP15-AC21)



P903b	PACKING INSTRUCTION	P903b
This instruction applies to used cells and batteries of UN Nos. 3090 and 3091.		
Used lithium cells and batteries, with a gross mass of not more than 500 g collected for disposal, together with other used non-lithium batteries or alone, may be carried, without being individually protected, under the following conditions:		
(1) In 1H2 drums 4H2 boxes conforming to the packing group II performance level for solids or		
(2) In 1A2 drums 4A boxes fitted with a PE bag and conforming to the packing group II performance level for solids. The PE bag shall :		
have an impact resistance of at least 480 grams in both parallel and perpendicular planes with respect to the length of the bag,		
have a minimum of 500 microns of thickness with electrical resistivity of more than 10 M ohms and low water adsorption rate over 24 hours at 25°C lower than 0.01% the PE bag may be used once only.		
(3) In collecting trays with a gross mass of less than 30 kg made from non-conducting material meeting the general conditions of 4.1.1.1, 4.1.1.2 and 4.1.1.5 to 4.1.1.8.		
Additional requirement:		
The empty space in the packaging shall be filled with cushioning material. The cushioning may be dispensed when the package is entirely fitted with a plastic bag and the bag is closed.] Hermetically sealed packaging shall be fitted with a venting device according to 4.1.1.8. The venting device shall be so designed that an overpressure caused by gases does not exceed 10 kPa.		

The SIMBA electrolyte

+ Base-line Cell: 1M NaPF₆ in EC:DEC

- + Excellent ionic conductivity and satisfactory electrochemical stability window.
- + Safety concerns arise due to the high volatility and flammability of the organic liquid solvents and the instability of the NaPF₆ especially toward H₂O trace impurities.
- + Undesired dendrite formation, side reactions leading to thermal runaways and explosion events.

+ SIMBA Cell: **Solid-State Electrolytes (SSEs) - Single-Ion conductor Polymer Electrolytes (SIPES).**

- + Good Conductivities and expanded stability window.
- + Reduction in Dendritic growth?
- + Does not spill.

Potential implications of using SIPLEs?

238 (a) Batteries can be considered as non-spillable provided that they are capable of withstanding the vibration and pressure differential tests given below, without leakage of battery fluid.

Vibration test: The battery is rigidly clamped to the platform of a vibration machine and a simple harmonic motion having an amplitude of 0.8 mm (1.6 mm maximum total excursion) is applied. The frequency is varied at the rate of 1 Hz/min between the limits of 10 Hz and 55 Hz. The entire range of frequencies and return is traversed in 95 ± 5 minutes for each mounting position (direction of vibration) of the battery. The battery is tested in three mutually perpendicular positions (to include testing with fill openings and vents, if any, in an inverted position) for equal time periods.

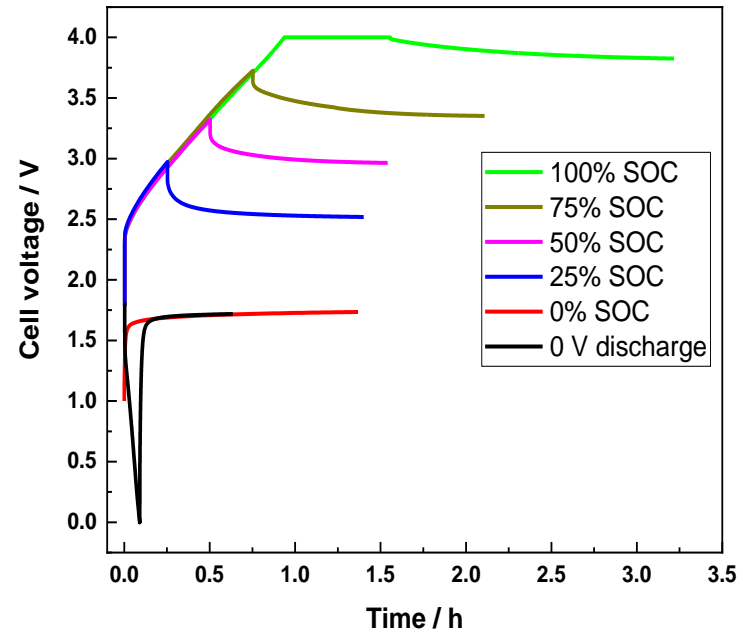
Pressure differential test: Following the vibration test, the battery is stored for six hours at 24 °C ± 4 °C while subjected to a pressure differential of at least 88 kPa. The battery is tested in three mutually perpendicular positions (to include testing with fill openings and vents, if any, in an inverted position) for at least six hours in each position.

(b) Non-spillable batteries are not subject to the requirements of ADR if, at a temperature of 55 °C, the electrolyte will not flow from a ruptured or cracked case and there is no free liquid to flow and if, as packaged for carriage, the terminals are protected from short circuit.

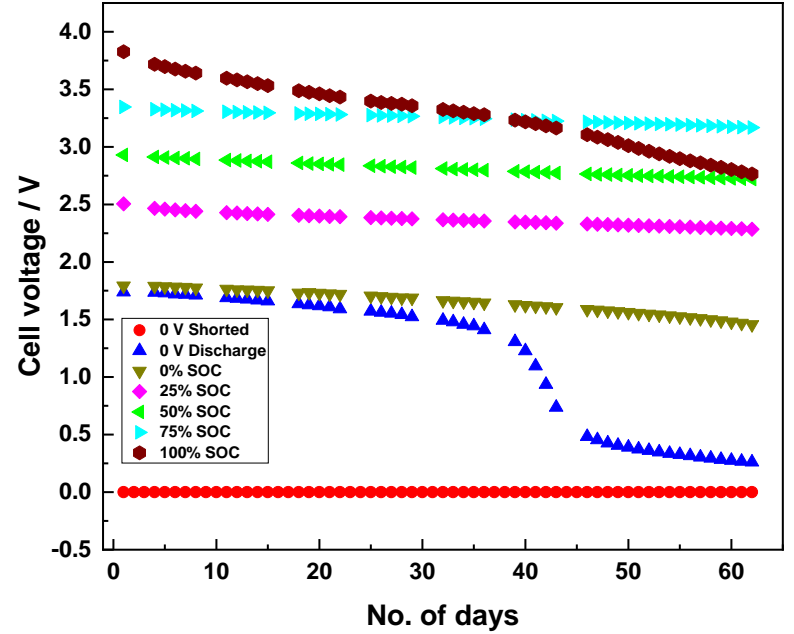
239 Batteries or cells shall not contain dangerous substances other than sodium, sulphur or sodium compounds (e.g. sodium polysulphides and sodium tetrachloroaluminate). Batteries or cells shall not be offered for carriage at a temperature such that liquid elemental sodium is present in the battery or cell unless approved and under the conditions established by the competent authority of the country of origin. If the country of origin is not a Contracting Party to ADR, the approval and conditions of carriage shall be recognized by the competent authority of the first country Contracting Party to ADR reached by the consignment.

P801	PACKING INSTRUCTION	P801
This instruction applies to UN Nos. 2794, 2795 and 3028 and used batteries of UN No. 2800.		
The following packagings are authorized, provided that the provisions of 4.1.1.1, 4.1.1.2, 4.1.1.6, and 4.1.3 are met:		
(1) Rigid outer packagings, wooden slatted crates or pallets.		
Additionally, the following conditions shall be met:		
(a) Battery stacks shall be in tiers separated by a layer of electrically non-conductive material;		
(b) Battery terminals shall not support the weight of other superimposed elements;		
(c) Batteries shall be packaged or secured to prevent inadvertent movement;		
(d) Batteries shall not leak under normal conditions of carriage or appropriate measures shall be taken to prevent the release of electrolyte from the package (e.g. individually packaging batteries or other equally effective methods); and		
(e) Batteries shall be protected against short circuits.		
(2) Stainless steel or plastics bins may also be used to carry used batteries.		
Additionally, the following conditions shall be met:		
(a) The bins shall be resistant to the electrolyte that was contained in the batteries;		
(b) The bins shall not be filled to a height greater than the height of their sides;		
(c) The outside of the bins shall be free of residues of electrolyte contained in the batteries;		
(d) Under normal conditions of carriage, no electrolyte shall leak from the bins;		
(e) Measures shall be taken to ensure that filled bins cannot lose their content;		
(f) Measures shall be taken to prevent short circuits (e.g. batteries are discharged, individual protection of the battery terminals, etc.); and		

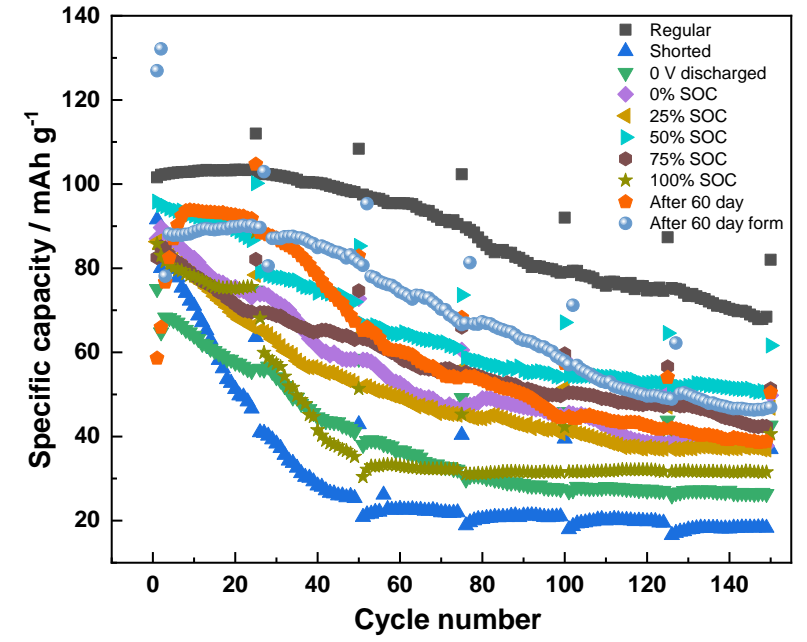
Storage of SIMBA cells – O3 cathode



Different charge / voltage for storage conditions

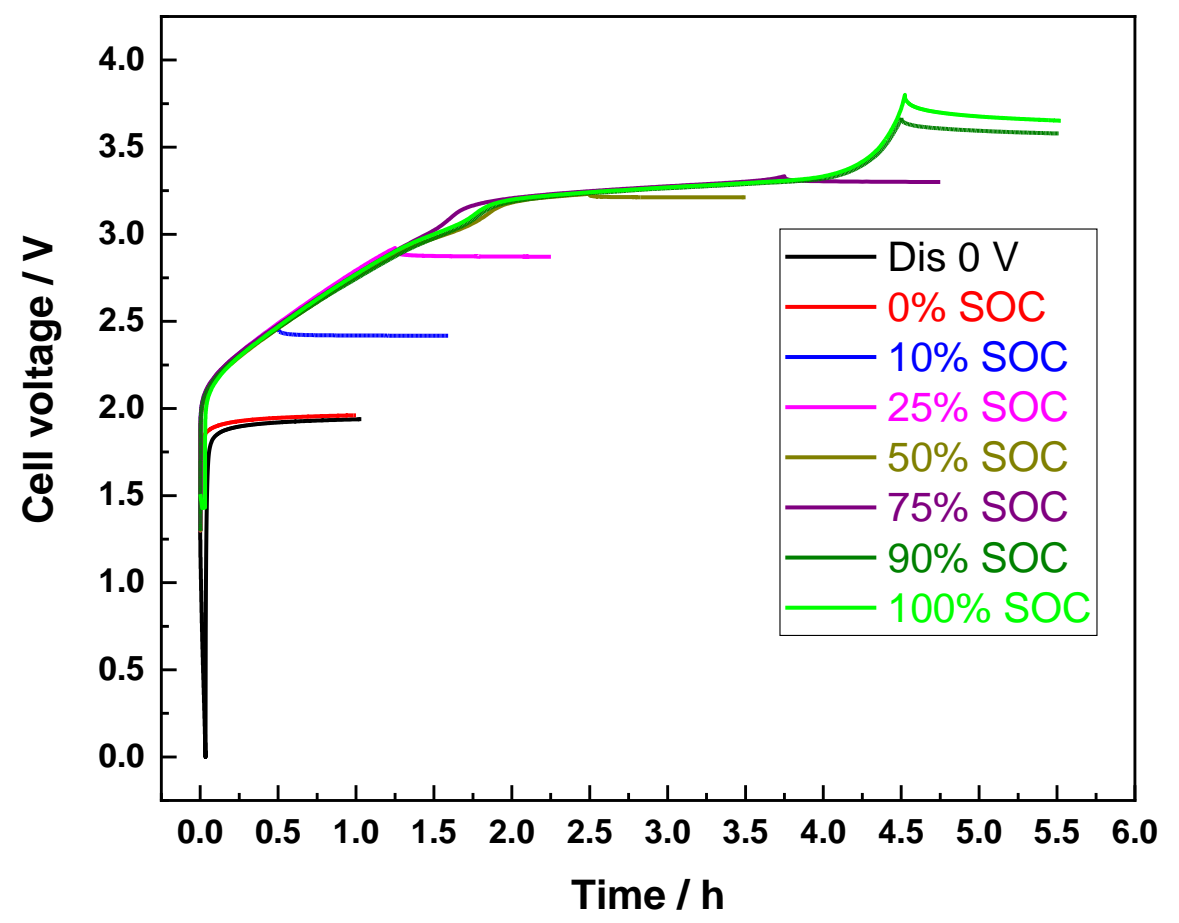


Open circuit Voltage Monitoring during storage

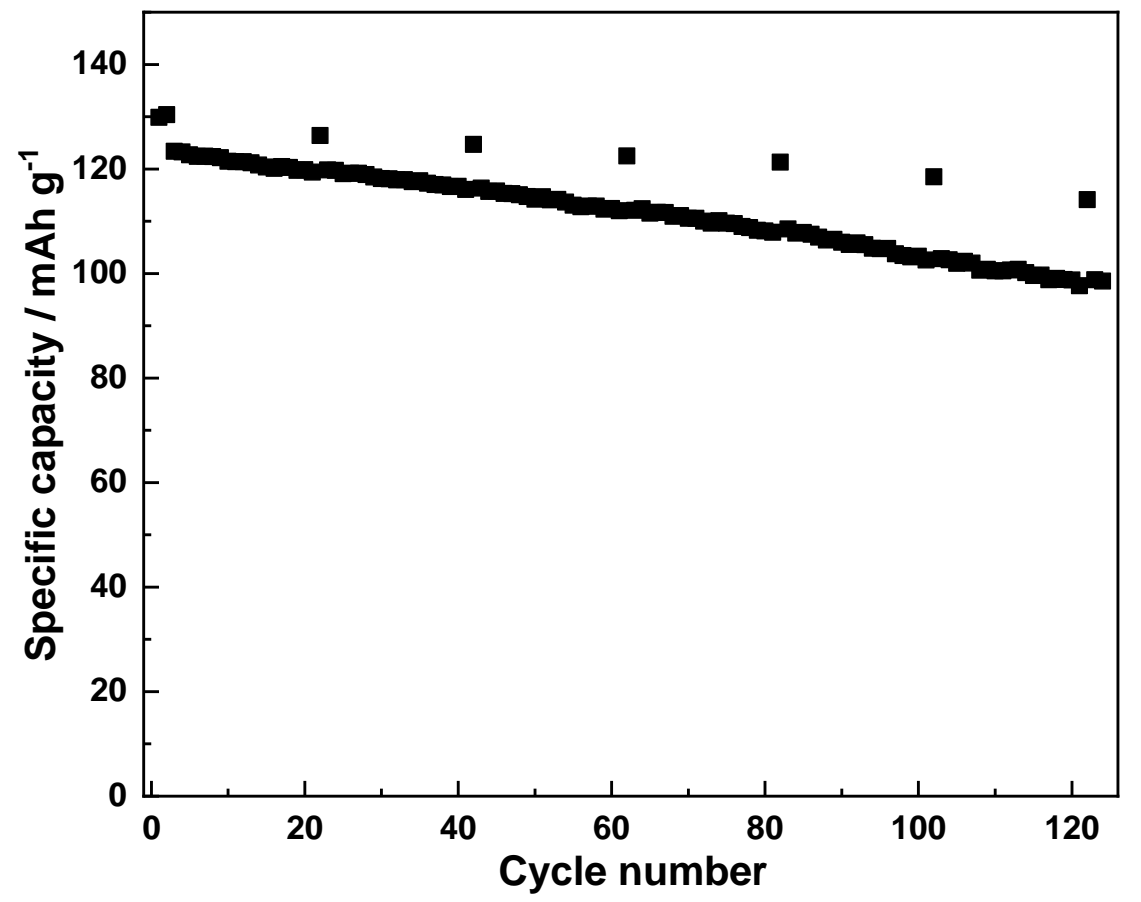


Cycle Life (Accelerated ageing) 1V to 4.2V and 100 mA g⁻¹

Storage of SIMBA cells – PW cathode



Voltage profiles at various SOC levels



Cycle life data for the reference cell

Storage of SIMBA cells – PW cathode

+-Zero Volts Discharge – gassing issues

BLCs Tear down and opening in the fume hood



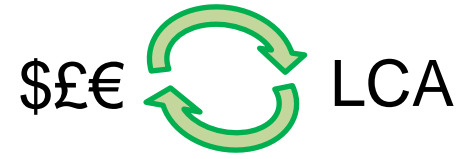


RECYCLING STEPS

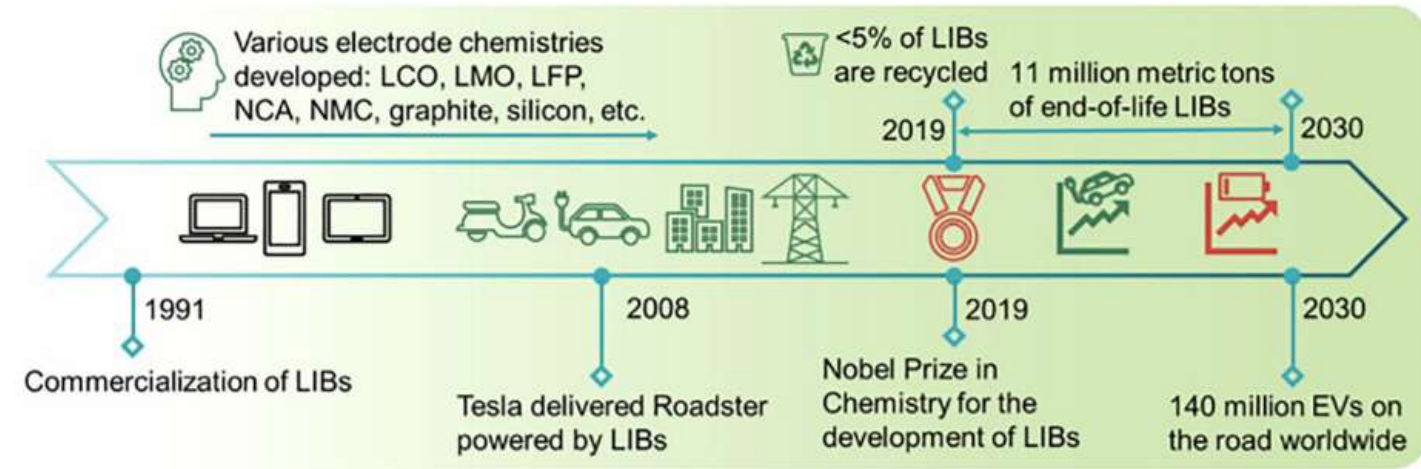
- 1) Why is recycling batteries important?
- 2) State-of-the-art recycling procedures for LIBs
- 3) SIMBA recycling of SIBs - *WP6*
- 4) Challenges

Recycling of batteries: why is it important?

- + E-waste has been the fastest growing segment of the solid waste stream + rise of EVs.
- + In 2019 still only 5% of LIBs were recycled in the EU.
- + Cu, Ni, Co
- + Graphite, Mn, Fe

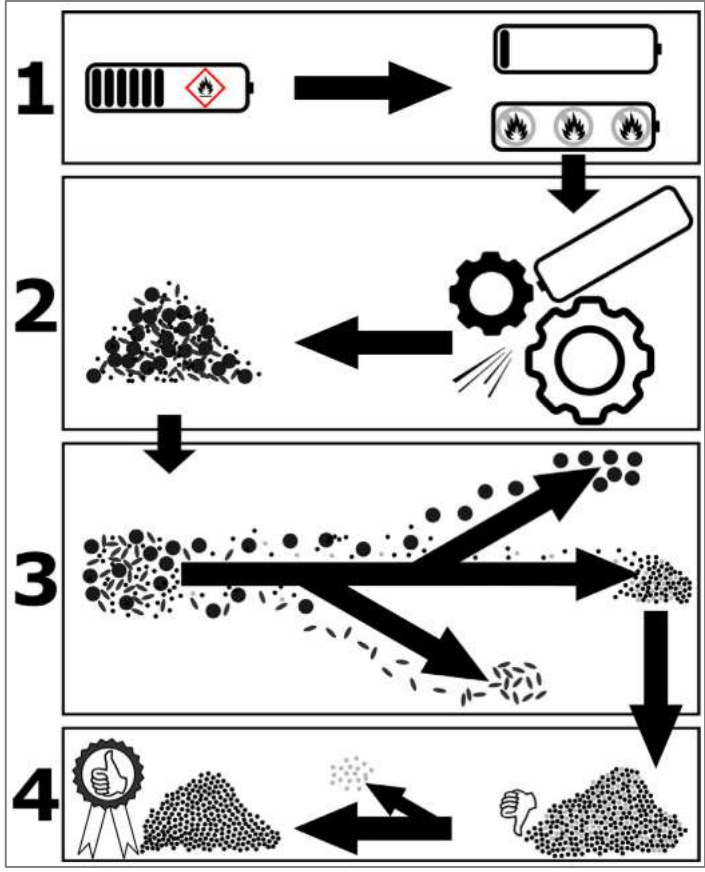


+ Li vs Na

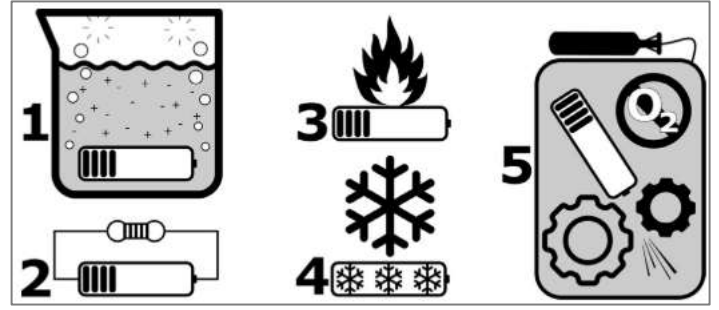


Recycling of LIBs: physical processes

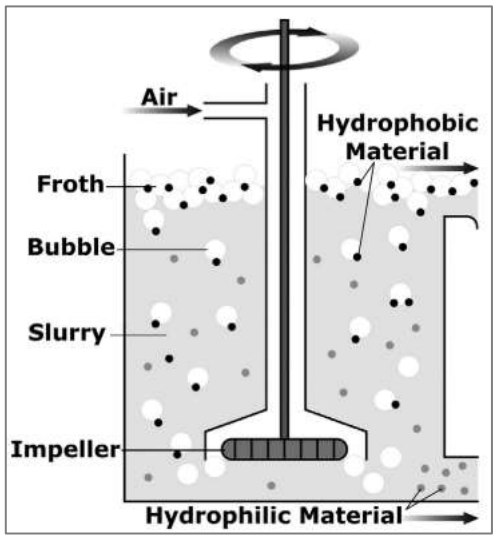
process flow for LIB recycling and disposal



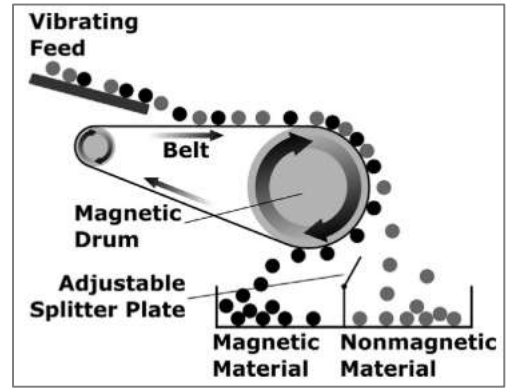
stabilization processes



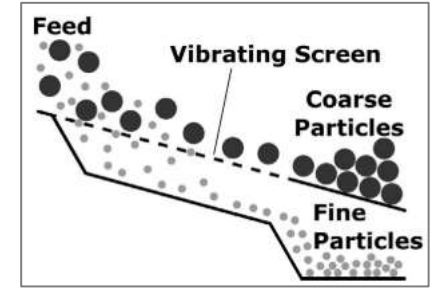
froth flotation



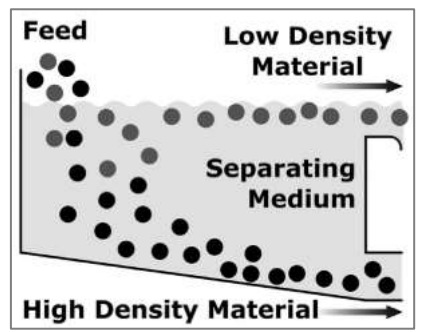
magnetic separation



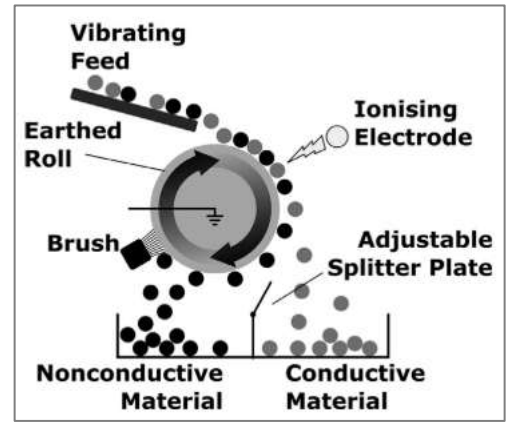
vibrating screen



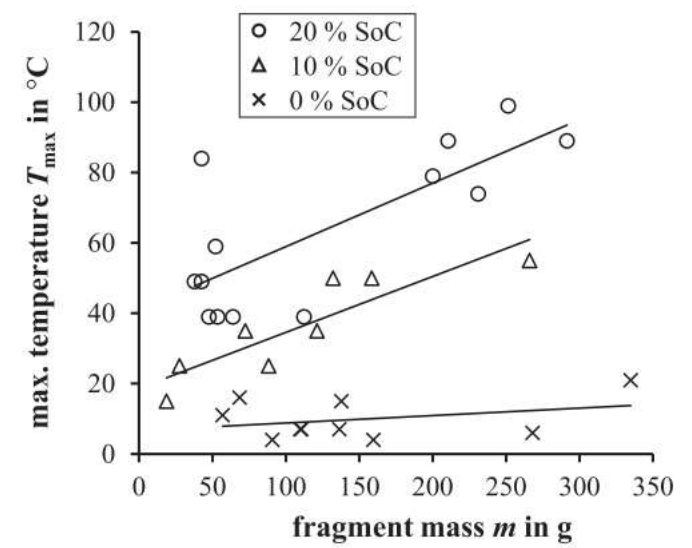
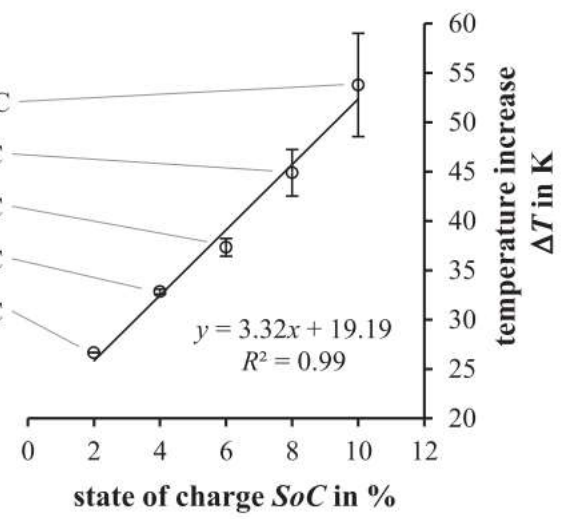
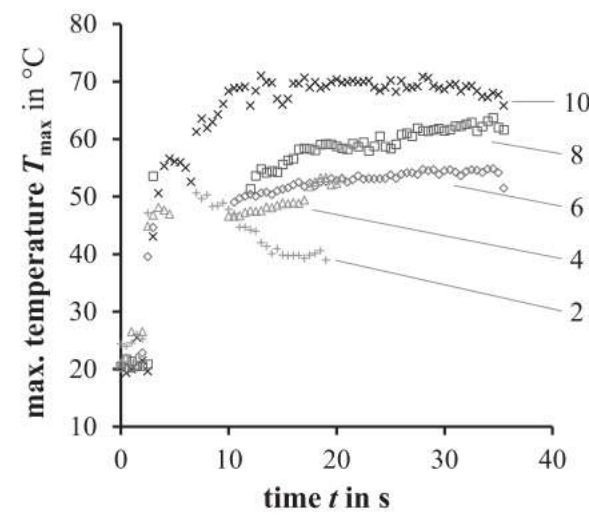
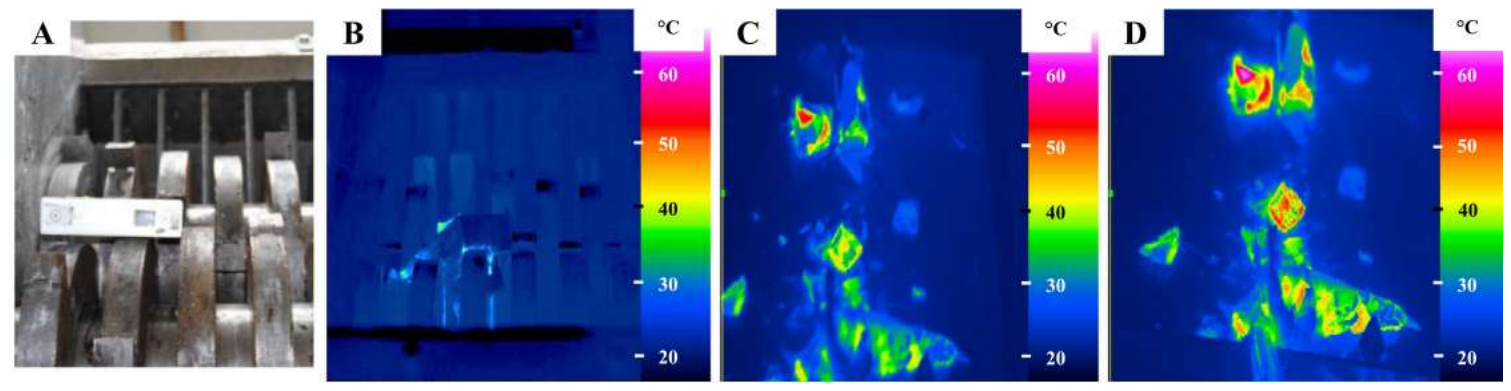
density separation



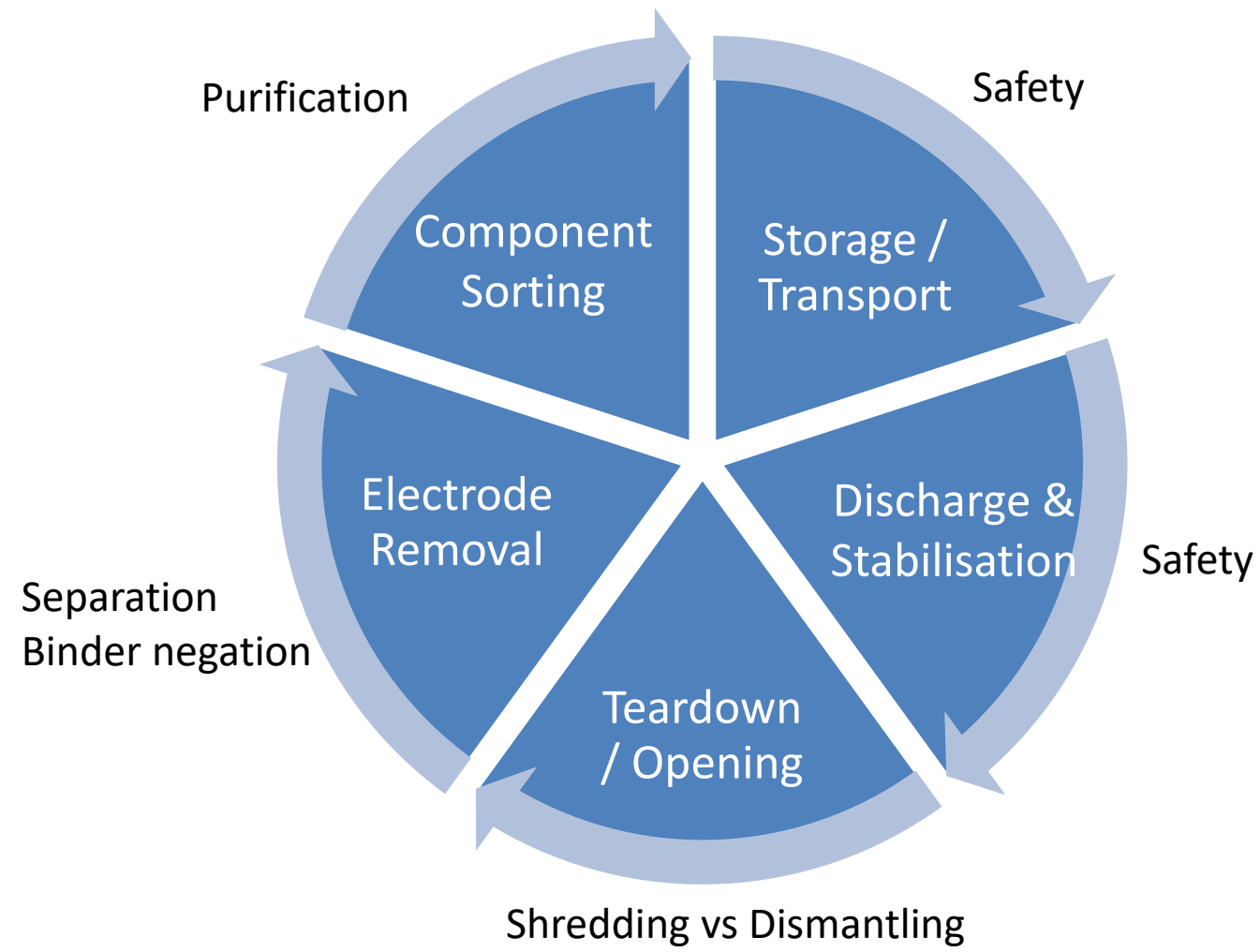
electrostatic separation



Shredding of batteries: SOC and T° rise

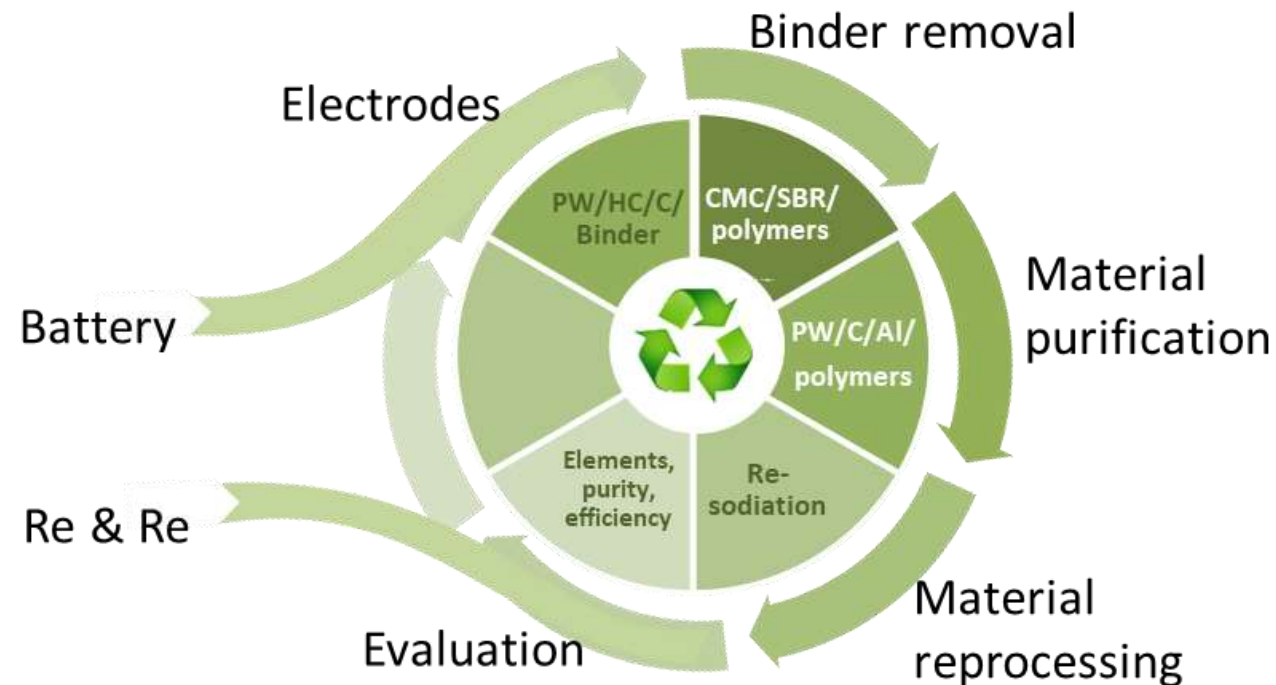


T6.1 Design for disassembly, separation and recycling

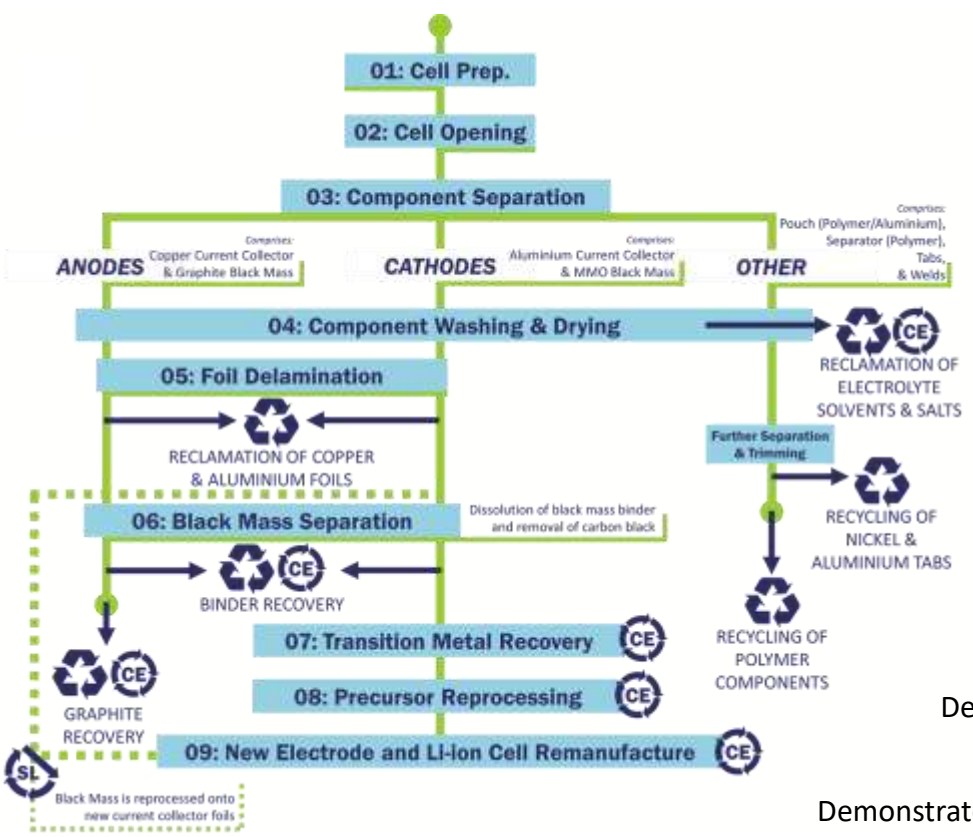


T6.2 Reclamation and Reuse

- + Objective: Report detailing cell re-use and material reclamation processes and waste stream analysis for materials. Detail reuse possibilities, and other material streams for reclaimed materials.



WP6 | deliverables and Milestones



Year 1												Year 2												Year 3												Year 4											
Jan/21	Feb/21	Mar/21	Apr/21	May/21	Jun/21	Jul/21	Aug/21	Sep/21	Oct/21	Nov/21	Dec/21	Jan/22	Feb/22	Mar/22	Apr/22	May/22	Jun/22	Jul/22	Aug/22	Sep/22	Oct/22	Nov/22	Dec/22	Jan/23	Feb/23	Mar/23	Apr/23	May/23	Jun/23	Jul/23	Aug/23	Sep/23	Oct/23	Nov/23	Dec/23	Jan/24	Feb/24	Mar/24	Apr/24	May/24	Jun/24	Jul/24					
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42						

Deliverables

- Deliver process for **80% of materials to be reclaimed** from the base-line cell
Recupyl, D6.1, **M36**
- Deliver process for 80% of materials to be reclaimed from the solid-state cell
Recupyl, D6.2, **M36**
- Develop a **safe storage, transport and opening protocol** for the solid-state batteries at end of life
UBham, D6.3, **M38**
- Demonstrate the **reclamation** of the anode and cathode from a solid-state cell, and the **re-use** in a battery,
Ubham, D6.4, **M40**
- Techno-economics and **Life Cycle Analysis** including environmental impact of full-size industrial capacity unit
Recupyl, D6.5, **M42**

Recycling Steps

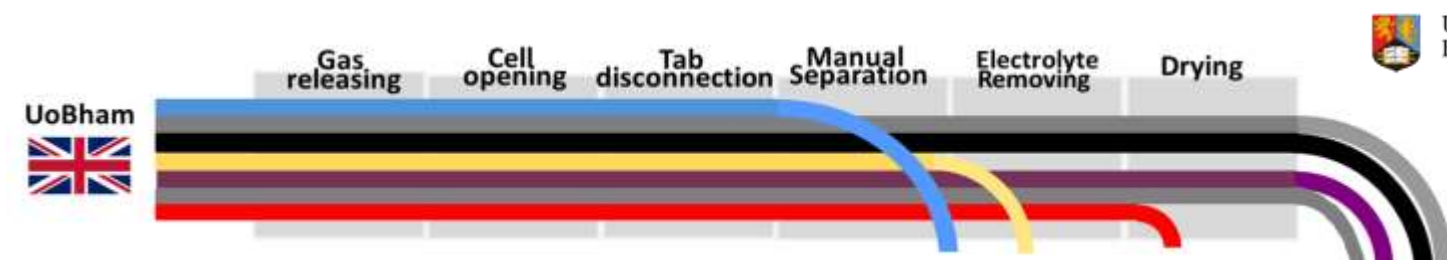
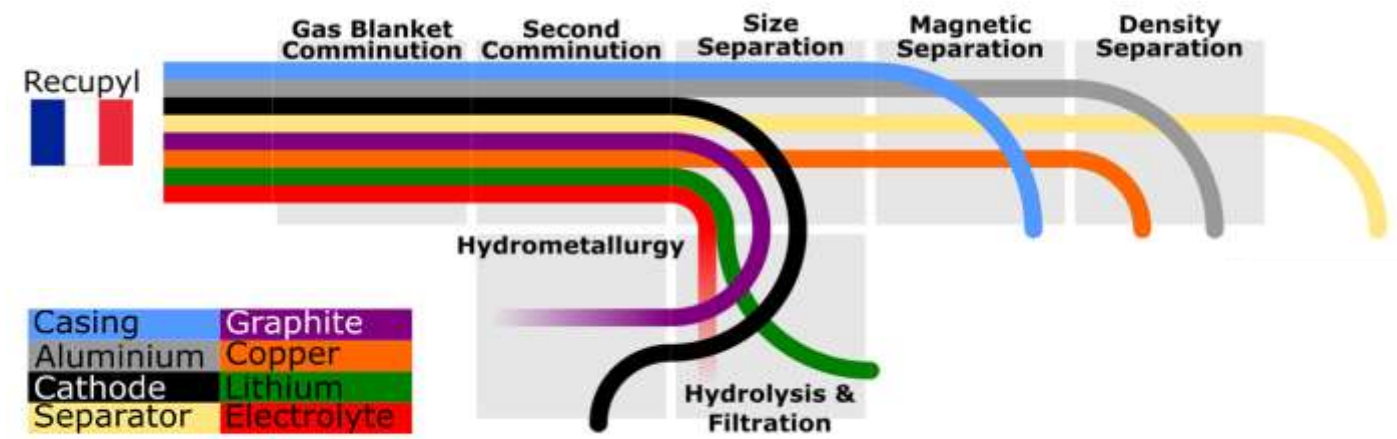
+ Task 6.1 Design for disassembly, separation and recycling.

Step 1: Discharge and stabilization



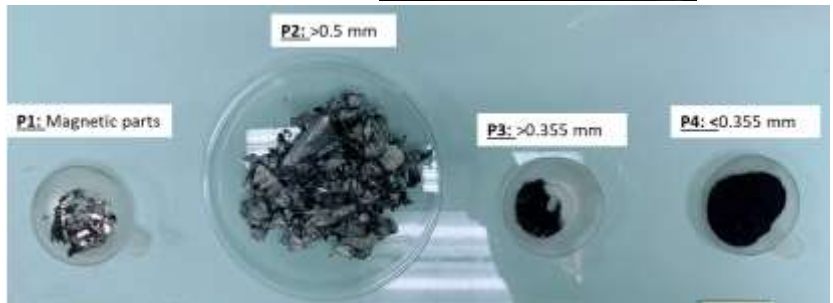
Discharge the cell under fix resistance until V=0.

Step 2: Disassemble and separation



WP6 | Results and outcomes_T6.1

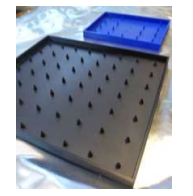
I



II



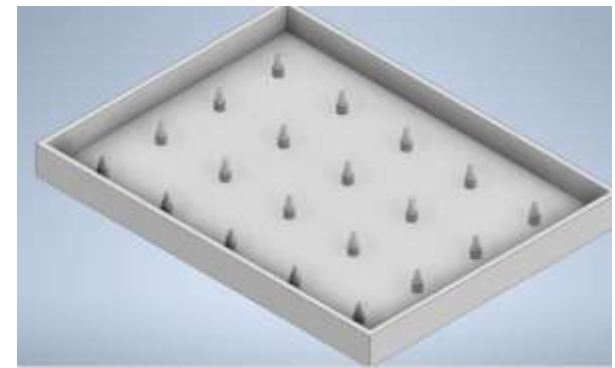
III



WP6 | Results and outcomes_T6.1

+ New recycling technique 1: Ice-stripping

Designed and 3D printed tailored tray for delamination



#1-1 anode delamination



#1-2 cathode delamination



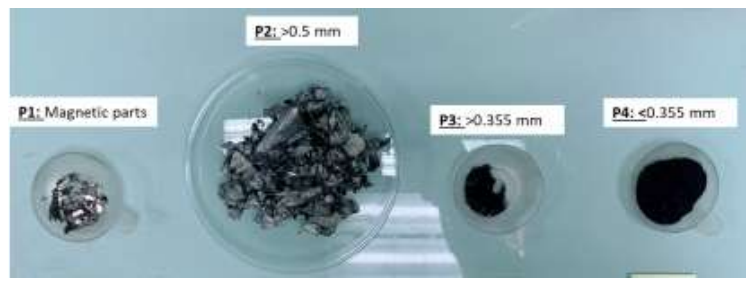
_Material recovery from BLCs



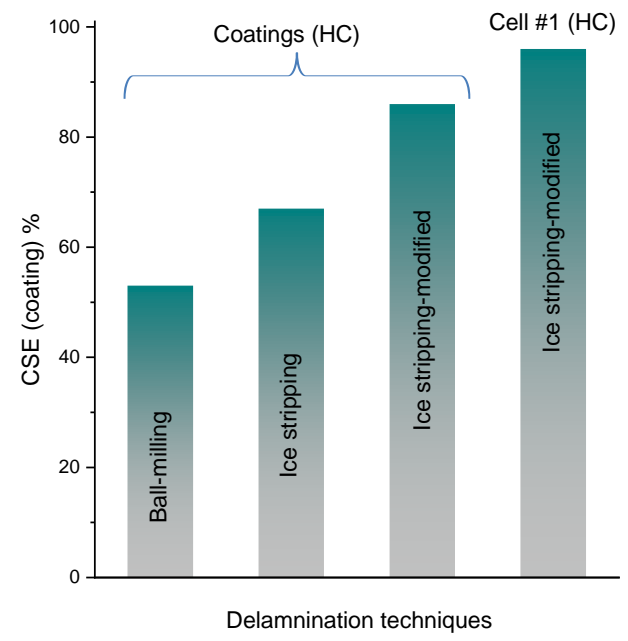
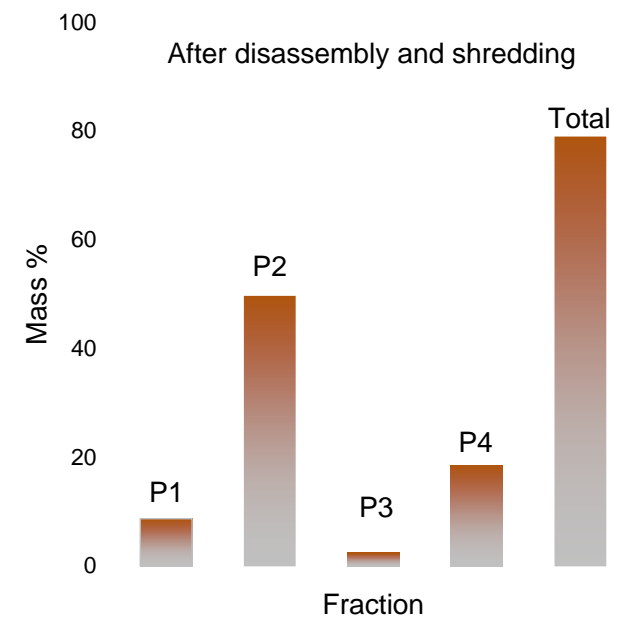
- Notes:
- + Coatings become brittle after freezing
 - + Back side delamination is challenging without damaging the current collector

WP6 | Results and outcomes_T6.1

Task 6.1 Design for disassembly, separation and recycling.

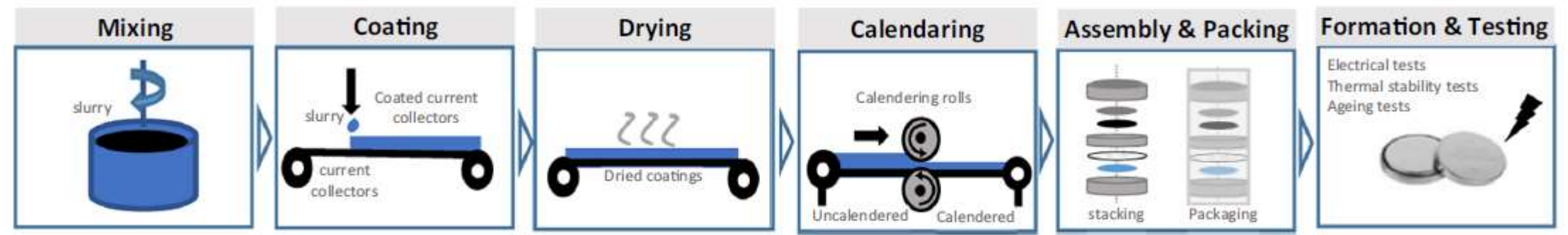
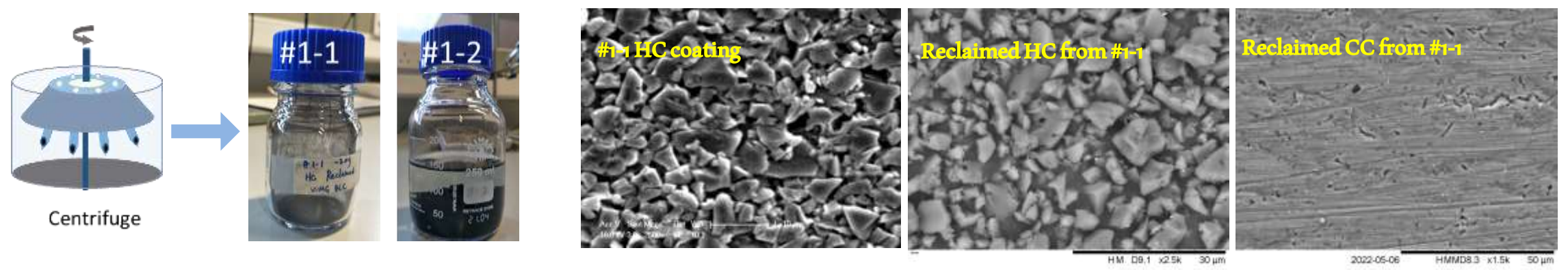


$$CSE_{A/coating} = \frac{\text{mass of separated A/coating}}{\text{initial mass of A/coating}}$$



WP6 | Results and outcomes_T6.2

+ Task 6.2 Reclamation, Remanufacturing and Reuse.



Electrode and cell manufacturing main processes.

Conclusions

Storage and Transport
Regulations

Safety during storage,
transport, and recycling

Recycling **Efficiency**

Recycling **Scalability**

Baseline SIB cell

vs

SIMBA SSB cell

Conclusions

	SIMBA <u>safety and transport standards</u> and <u>recycling steps</u>
SoA stationary storage batteries	How do the SIMBA materials and cell development compare with the current State-of-the-Art for stationary storage batteries?
Challenges for Na technology	What steps are still needed to make the SIMBA Na-ion technology even more promising?
Future pathways - Impact	What do end user and policymakers request from SIB technology for stationary storage applications?