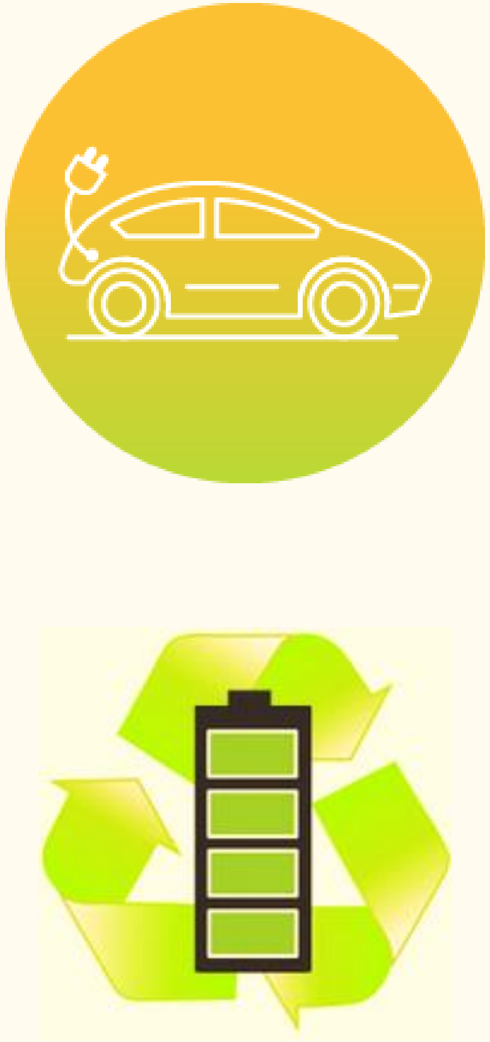
Recycling & Reuse of EV Battery Materials



Dr. Manjusha Shelke,

Principal Scientist,

CSIR-National Chemical Laboratory, Pune

Email: mv.shelke@ncl.res.in

http://academic.ncl.res.in/mv.shelke

Project by:

National Chemical Laboratory

ARAI
Progress through Research

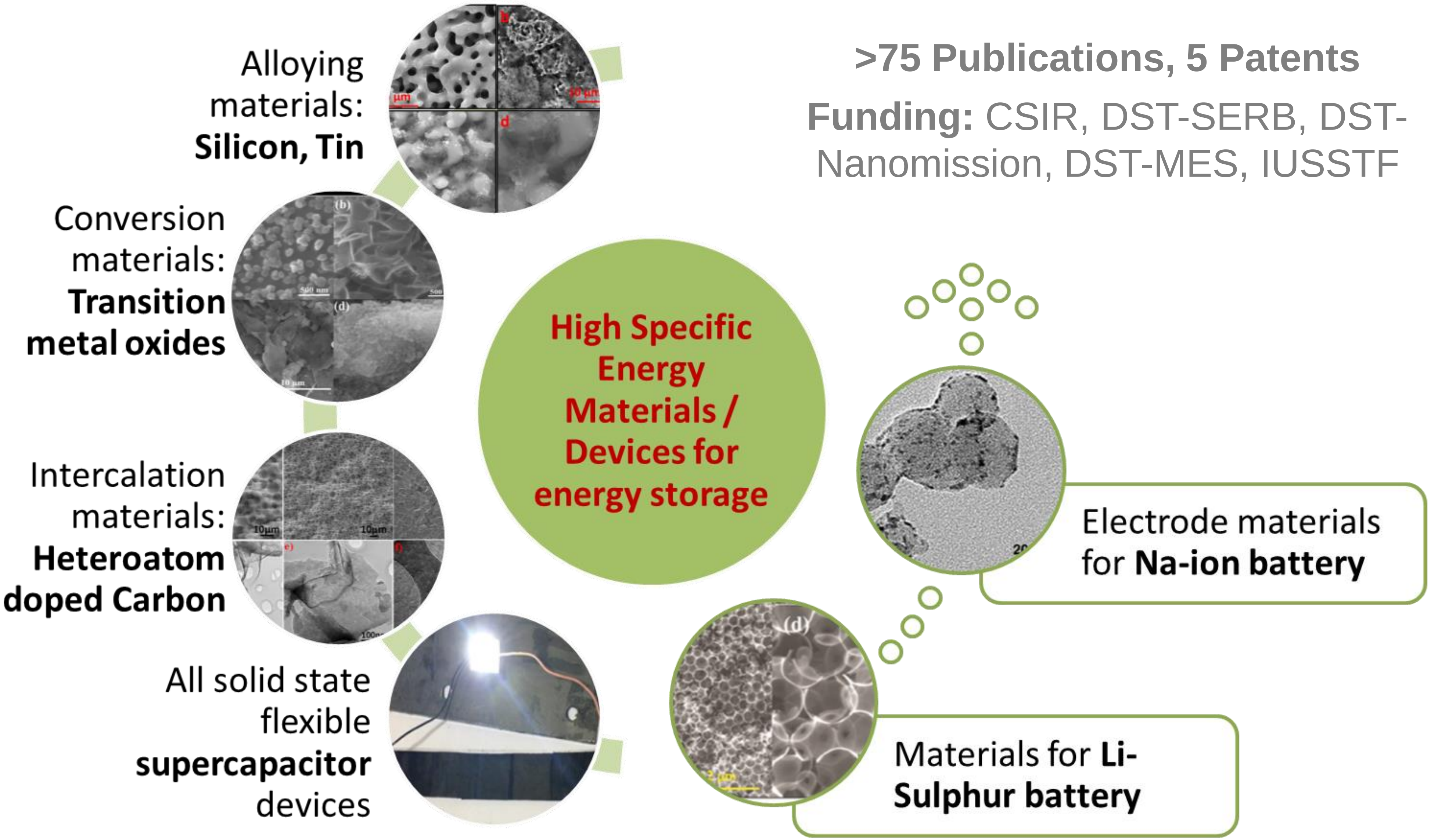
ELECTRONIC WASTE RECYCLING
E - Parisaraa Private limited

Energy Storage Research at CSIR-NCL

National Chemical Laboratory

>75 Publications, 5 Patents

Funding: CSIR, DST-SERB, DST-Nanomission, DST-MES, IUSSTF



Alloying materials: **Silicon, Tin**

Conversion materials: **Transition metal oxides**

Intercalation materials: **Heteroatom doped Carbon**

All solid state flexible **supercapacitor** devices

High Specific Energy Materials / Devices for energy storage

Electrode materials for **Na-ion battery**

Materials for **Li-Sulphur battery**

Rechargion Energy Pvt. Ltd.

Redefining clean energy storage world map….

Rechargion

Baner, Pune 411045
INDIA
https://rechargion.com

Supported/Mentored by:-

National Chemical Laboratory

NCL INNOVATIONS
Solutions from CSIR India

VENTURE CENTER

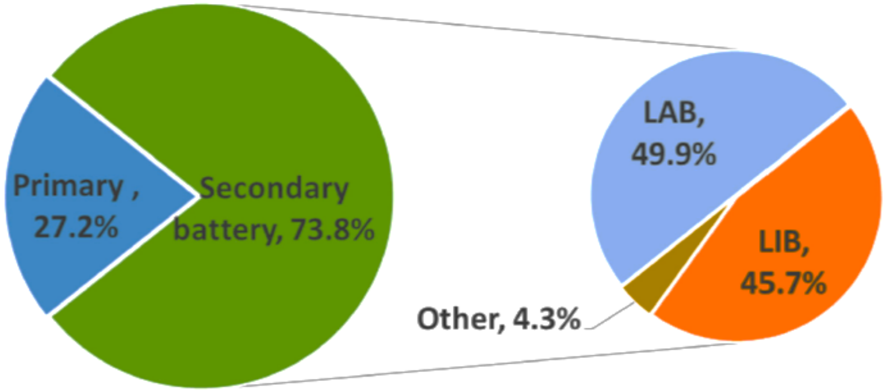
SOCIAL alpha

AIM prime
ATAL INNOVATION MISSION

#startupindia

An Indo-UK joint venture developing next generation Na-ion battery for mobility and stationary clean energy storage

EV Batteries (Li-ion)

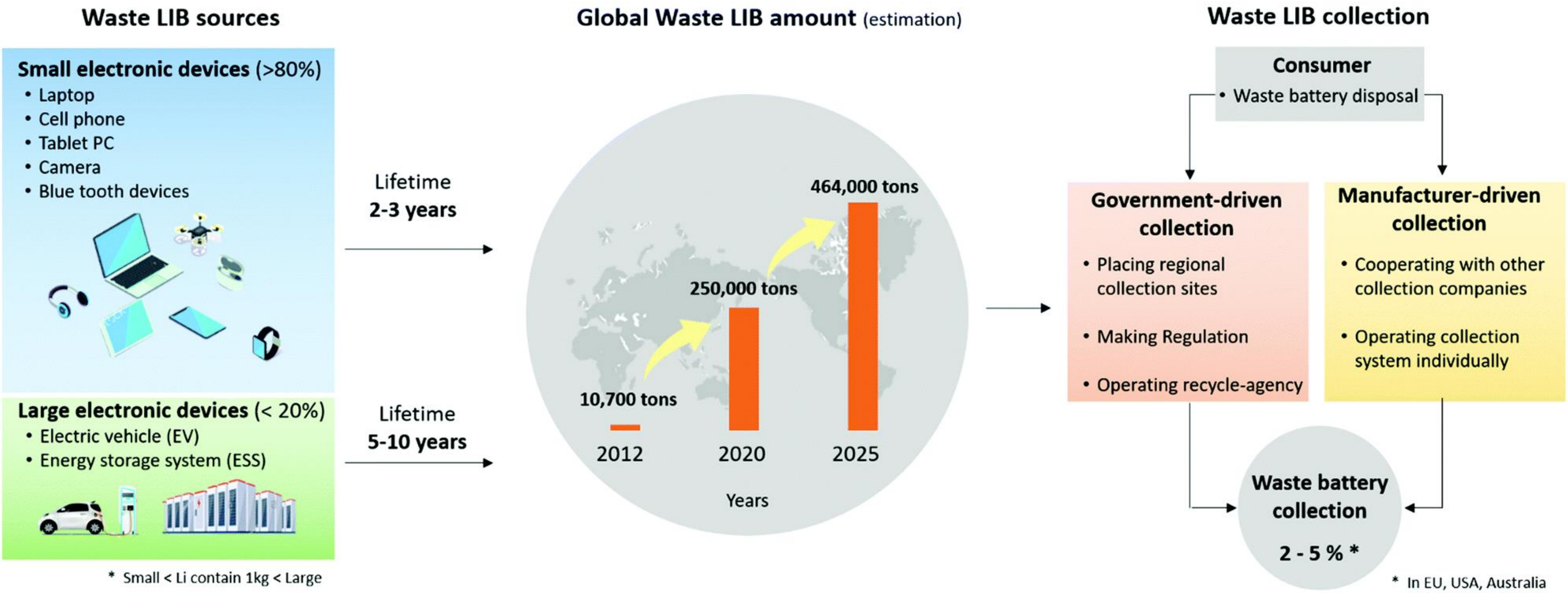


Source: Technavio, UK, 2020.

The global battery market share by different chemistries, 2019

- India's electric vehicle sales to grow at 26% annually
- Lithium-ion battery market in India is expected to increase from 2.9 GWh in 2018 to about 132 GWh by 2030 at a CAGR of 35.5%
- Lithium required for EV battery is 1000 times more than that of a smartphone

Current state of waste Lithium batteries



Waste LIB sources

Small electronic devices (>80%)
- Laptop
- Cell phone
- Tablet PC
- Camera
- Blue tooth devices

Large electronic devices (< 20%)
- Electric vehicle (EV)
- Energy storage system (ESS)

* Small < Li contain 1kg < Large

Lifetime **2-3 years**

Lifetime **5-10 years**

Global Waste LIB amount (estimation)

2012	10,700 tons
2020	250,000 tons
2025	464,000 tons

Waste LIB collection

Consumer
- Waste battery disposal

Government-driven collection
- Placing regional collection sites
- Making Regulation
- Operating recycle-agency

Manufacturer-driven collection
- Cooperating with other collection companies
- Operating collection system individually

Waste battery collection

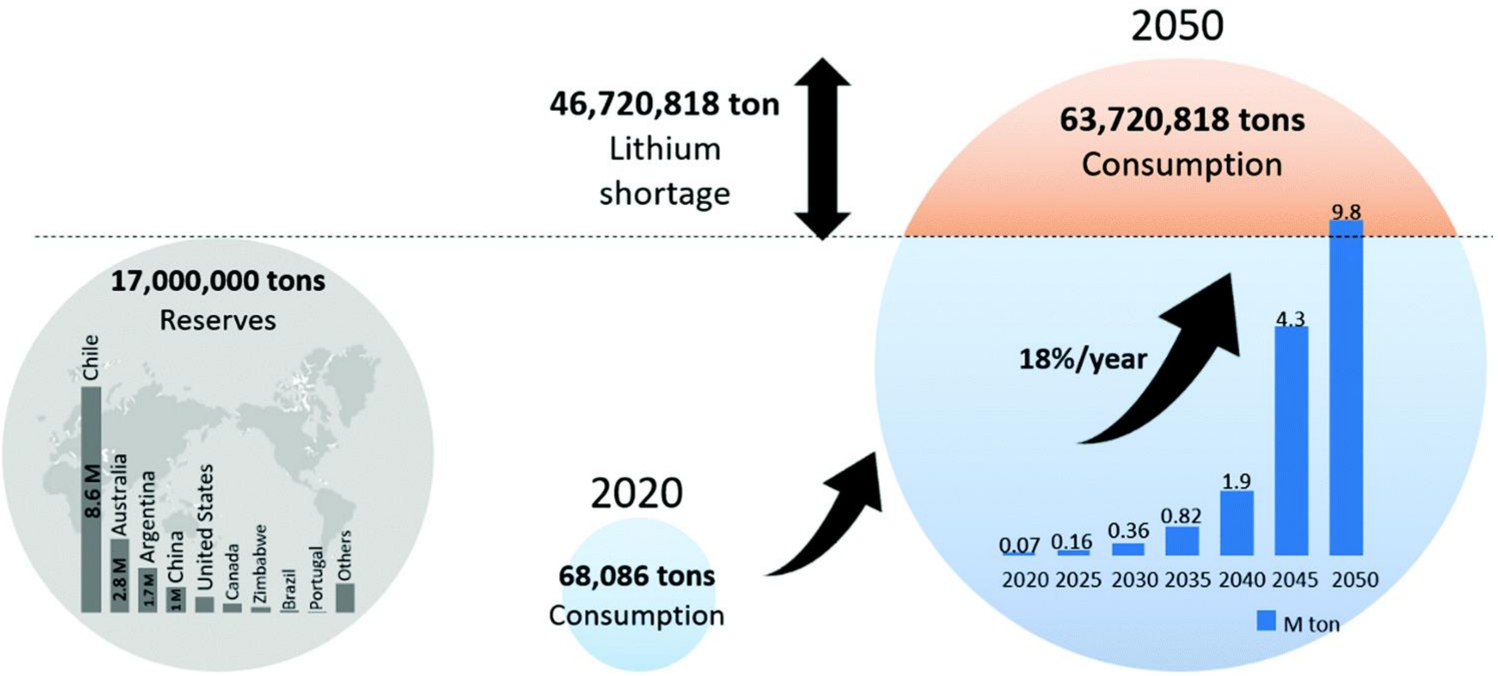
2 - 5 % *

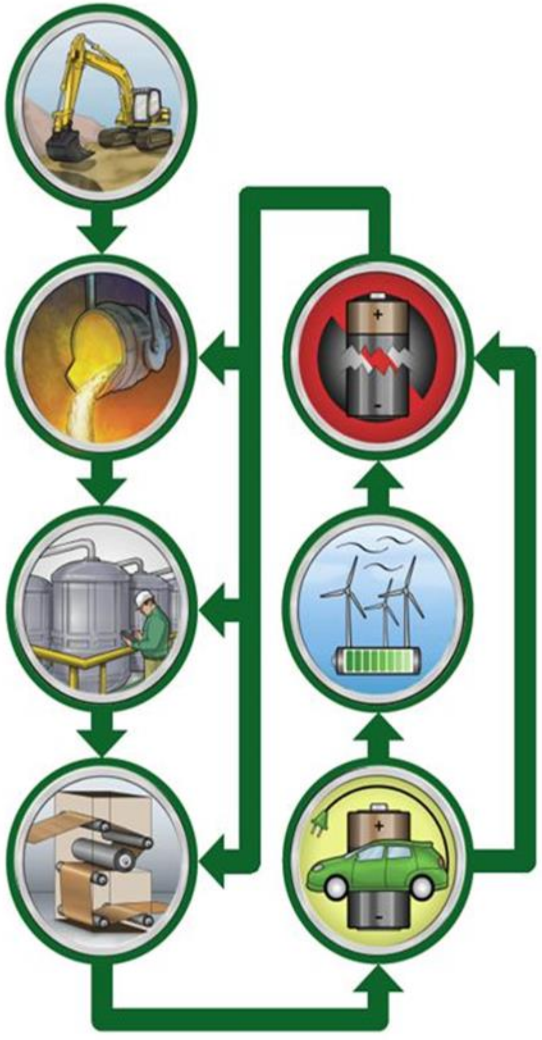
* In EU, USA, Australia

Source: Mater. Adv., 2021, 2, 3234-3250

Why Recycling

National Chemical
Laboratory





Reduce

Recycle

Reuse

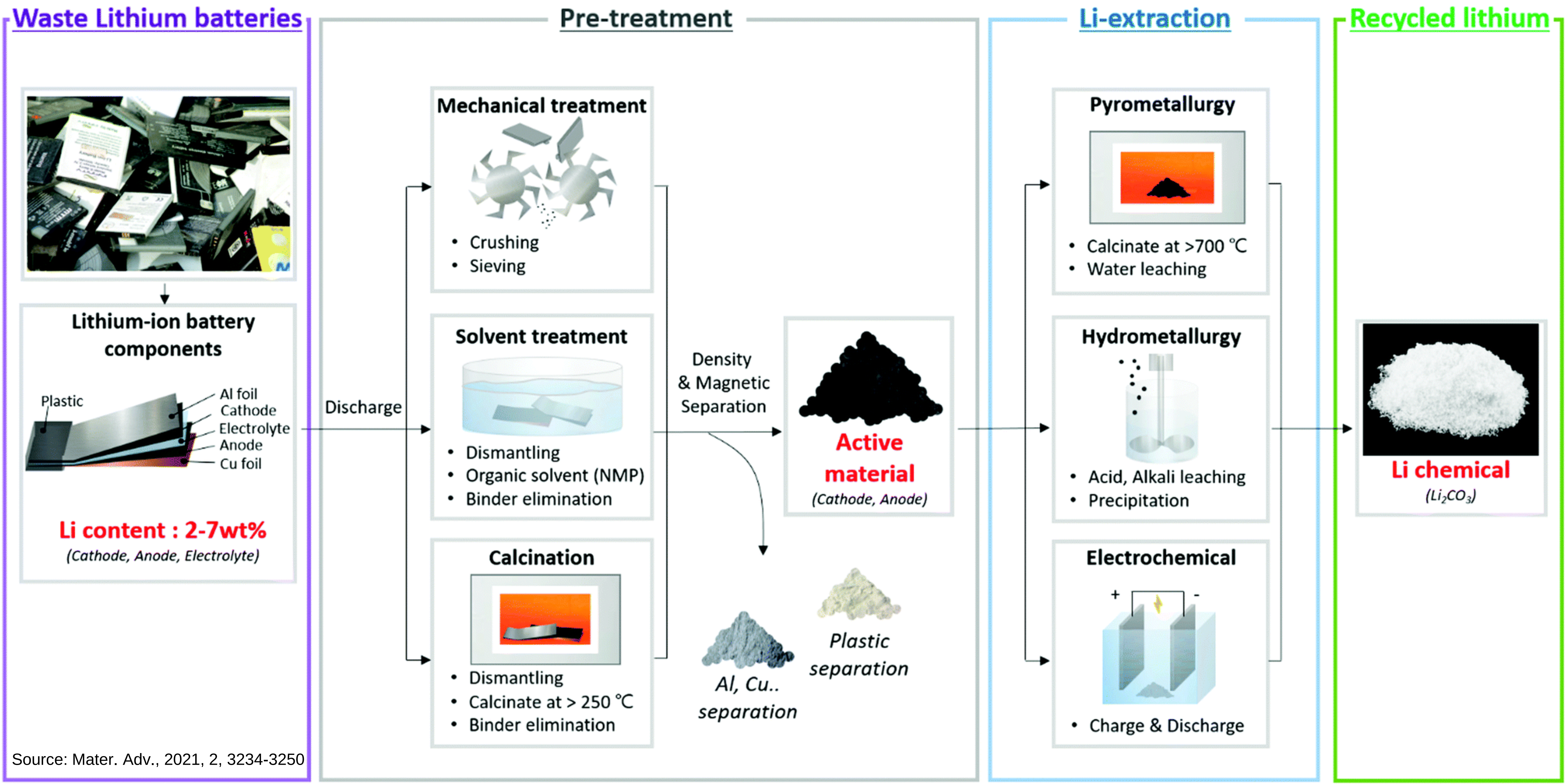
Sustainability/ supply risk

- Disproportionately distributed lithium reserves and ever-fluctuating costs.
- **Huge cost associated** with mining and extraction of pure Li
- Co deposits **in Democratic Republic of Congo**
- Ni will require 170 fold of the current extraction capacity.
- The demand of Li will be greater than the mining supply, unless LIBs are not recycled with a 90% efficiency.

Source: Mater. Adv., 2021, 2, 3234-3250

Typical recycling processes

National Chemical



Waste Lithium batteries

Lithium-ion battery components

Li content : 2-7wt%

(Cathode, Anode, Electrolyte)

Discharge

Pre-treatment

Mechanical treatment

- Crushing
- Sieving

Solvent treatment

- Dismantling
- Organic solvent (NMP)
- Binder elimination

Calcination

- Dismantling
- Calcinate at > 250 ℃
- Binder elimination

Density & Magnetic Separation

Active material

(Cathode, Anode)

Al, Cu.. separation

Plastic separation

Li-extraction

Pyrometallurgy

- Calcinate at >700 ℃
- Water leaching

Hydrometallurgy

- Acid, Alkali leaching
- Precipitation

Electrochemical

- Charge & Discharge

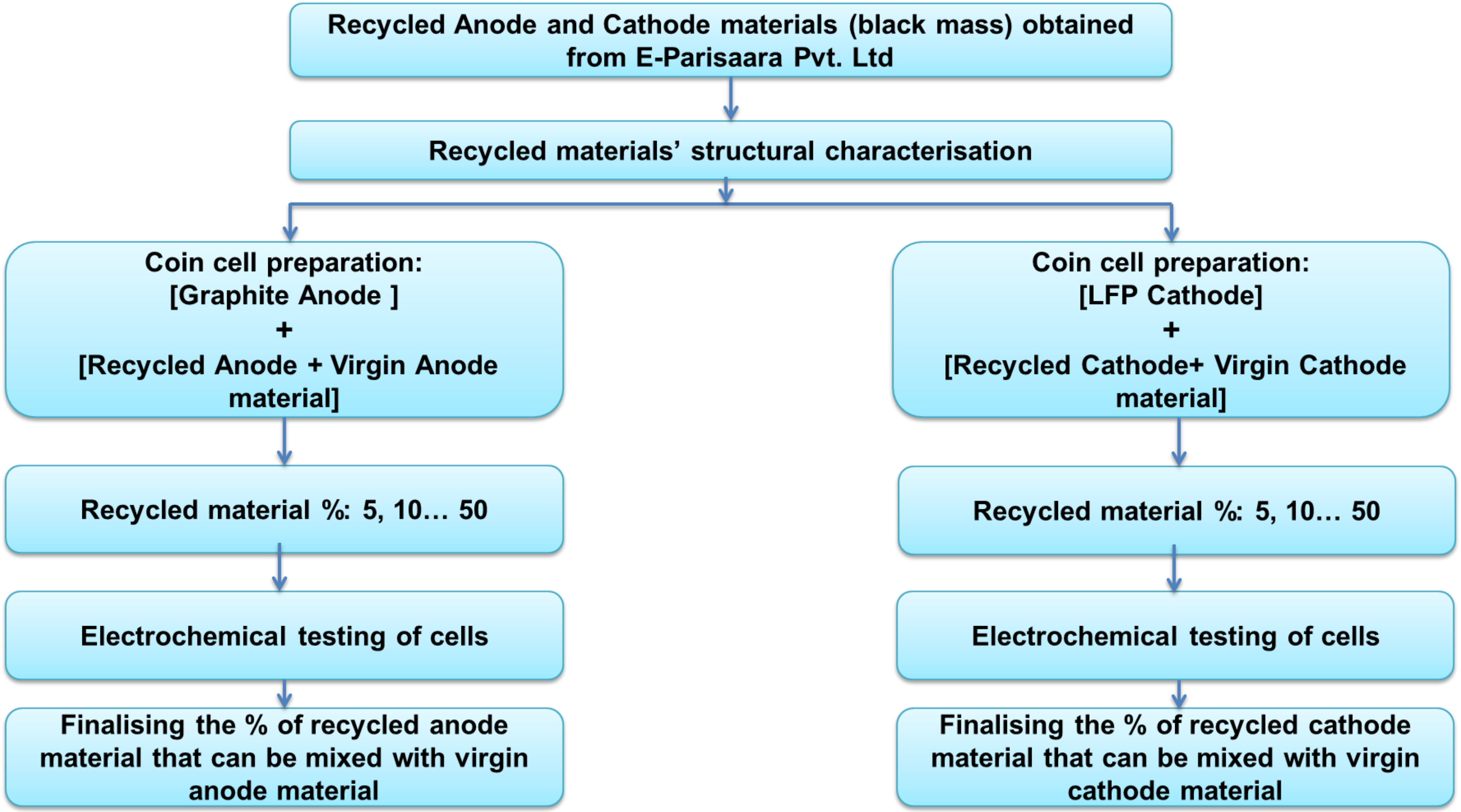
Recycled lithium

Li chemical

(Li_2CO_3)

Source: Mater. Adv., 2021, 2, 3234-3250

Recycled electrode materials testing at NCL



Recycled Anode and Cathode materials (black mass) obtained from E-Parisaara Pvt. Ltd

Recycled materials' structural characterisation

Coin cell preparation:
[Graphite Anode]
+
[Recycled Anode + Virgin Anode material]

Recycled material %: 5, 10… 50

Electrochemical testing of cells

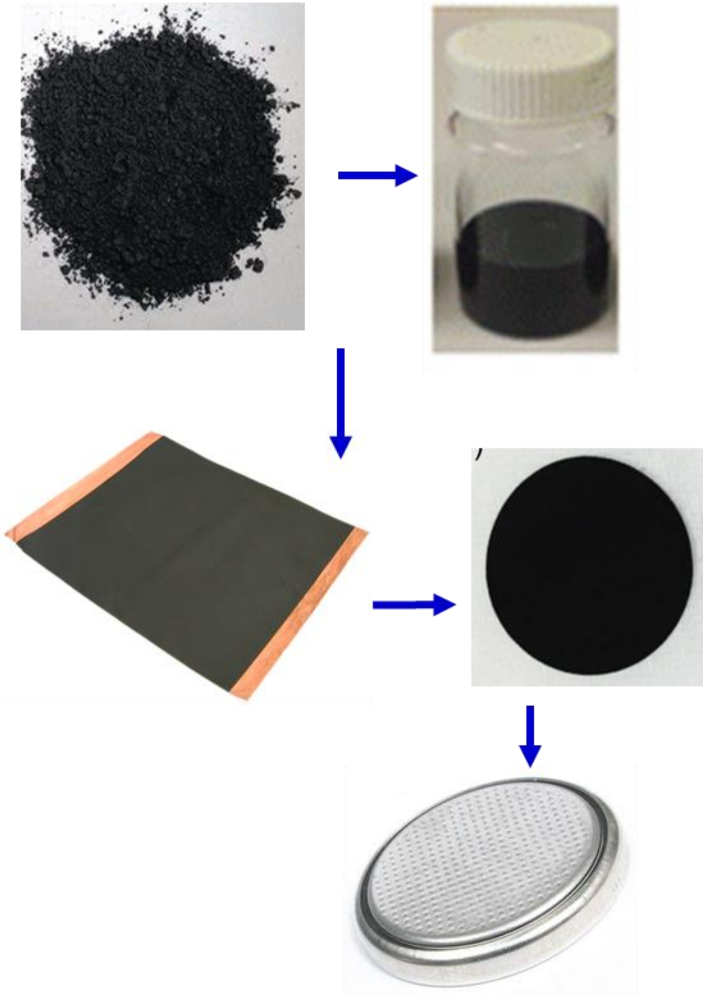
Finalising the % of recycled anode material that can be mixed with virgin anode material

Coin cell preparation:
[LFP Cathode]
+
[Recycled Cathode+ Virgin Cathode material]

Recycled material %: 5, 10… 50

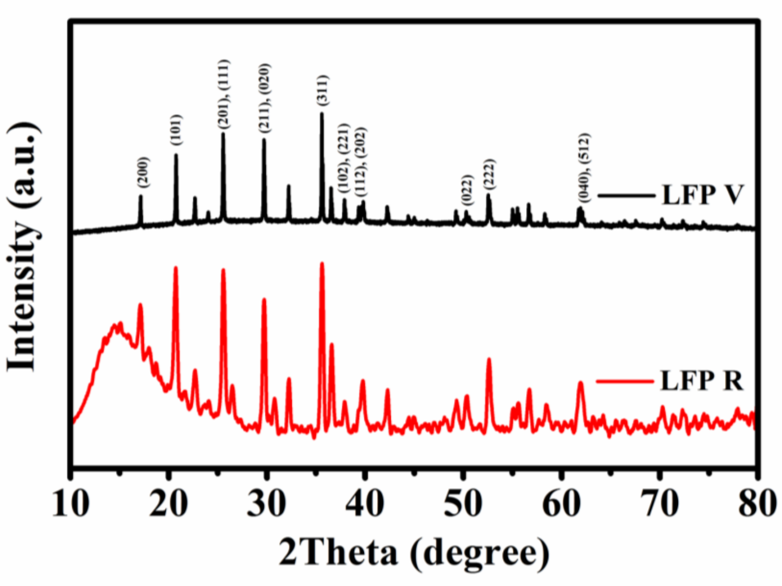
Electrochemical testing of cells

Finalising the % of recycled cathode material that can be mixed with virgin cathode material

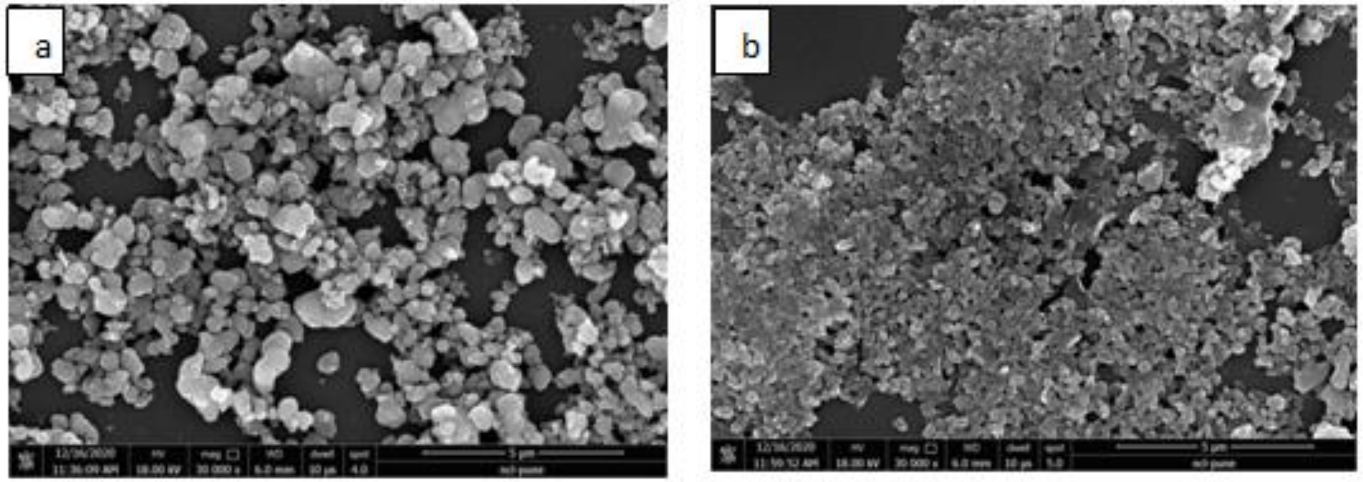


Cathode: Structural Characterization

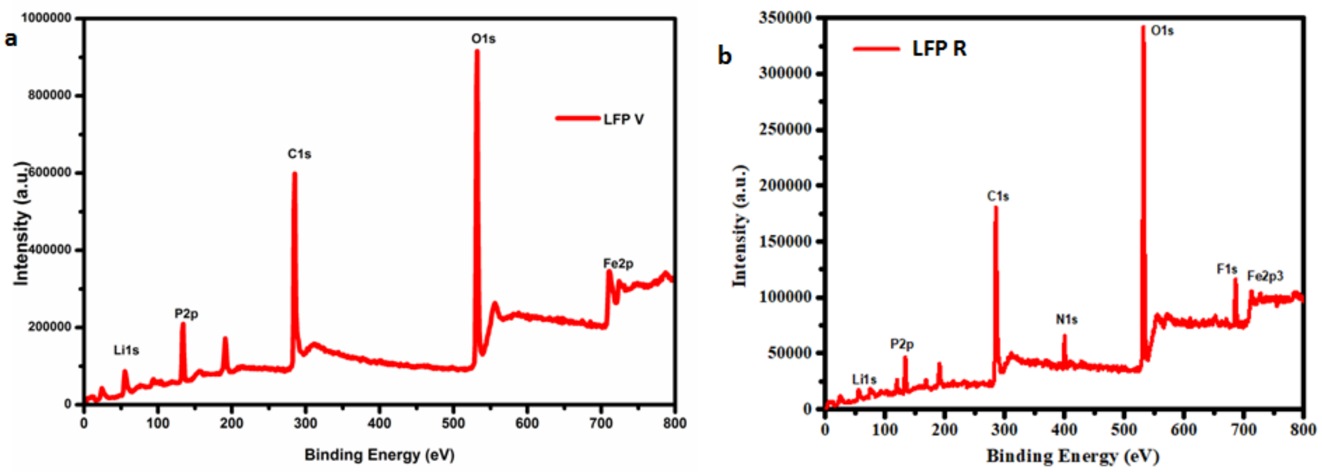
National Chemical Laboratory



XRD patterns of LFP V and LFP R



FESEM of (a) LFP V (b) LFP R

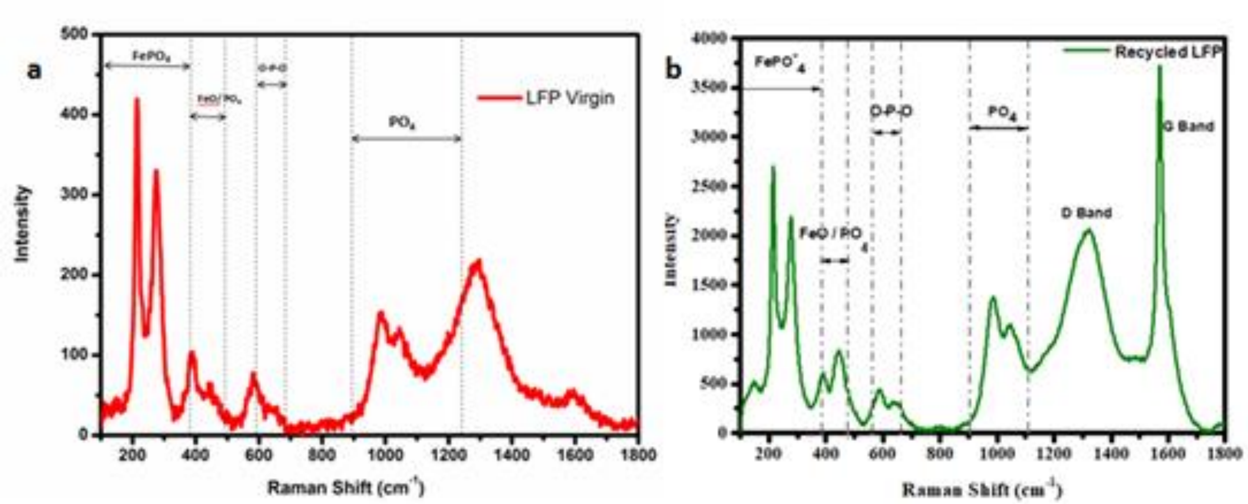


XPS survey scan for (a) LFP V) (b) LFP R

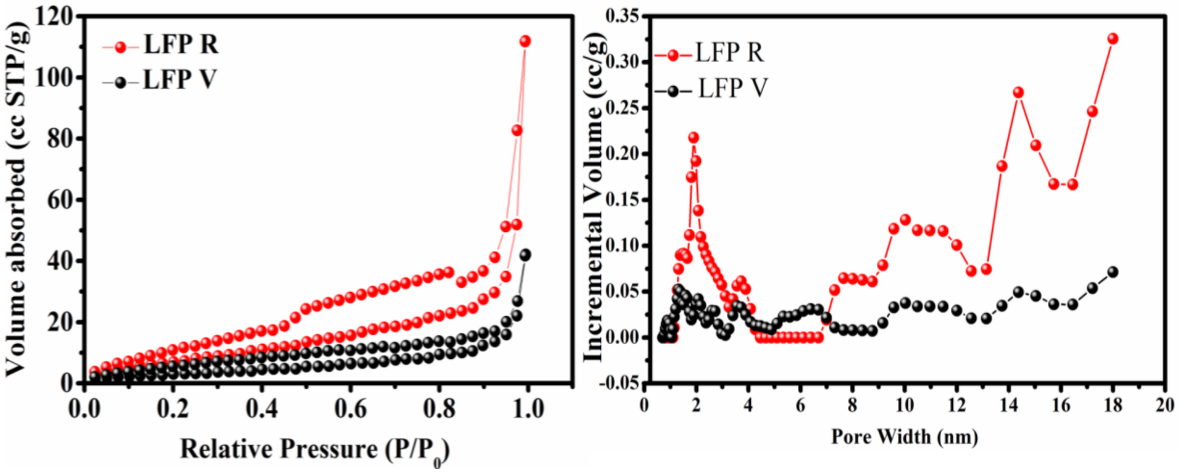
LFP V	
Name	Atomic %
O1s	18.76
C1s	29.17
P2p	5.4
Fe2p3	1.8
Li1s	44.86

LFP R	
Name	Atomic %
O1s	22.19
C1s	34.78
P2p	3.63
F1s	2.89
N1s	2.93
Cu2p	0.93
Al2p	5.12
Fe2p	1.23
S2p	1.06
Li1s	25.24

Cathode: Structural Characterization



Raman spectra of (a) LFP V and (b) LFP R materials



(a) N_2 adsorption –desorption isotherm curve of LFP materials (b) DFT method pore size distribution of LFP materials

A dominant G- band along with D- band obtained for recycled **LFP R** cathode

During cycling- **formation of CEI**

Graphitic carbon uniformly disperses with LFP particles act as conducting buffer

Surface area: **29.88 m^2g^{-1} and 11.481 m^2g^{-1}** for LFP R and LFP V.

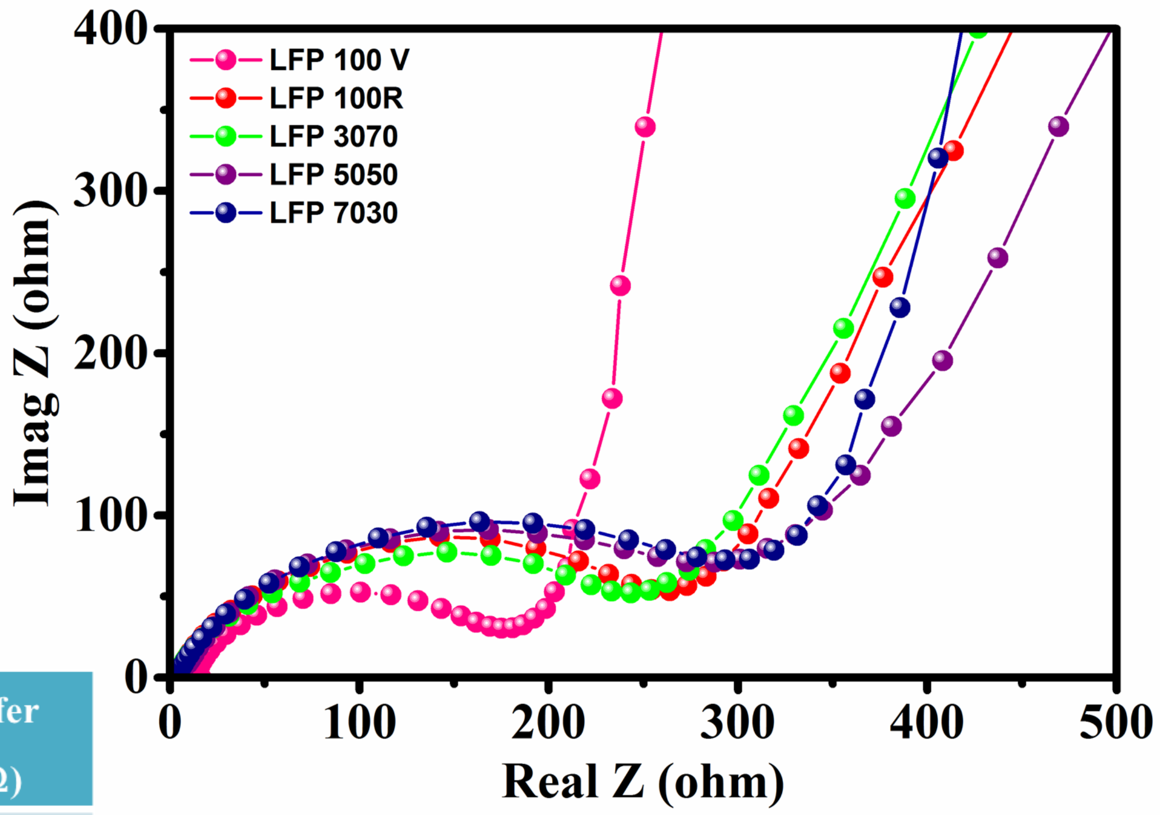
The pore volume: **0.099 and 0.034 ccg^{-1}** for LFP R and LFP V

Cathode: Electrochemical Characterization

Sample Name	Virgin Material(%)	Recycled Material (%)
New LFP	100	0
Recycled LFP	0	100
LFP 7030	70	30
LFP 5050	50	50
LFP 3070	30	70

Testing of Different combination (Physical Mixing)

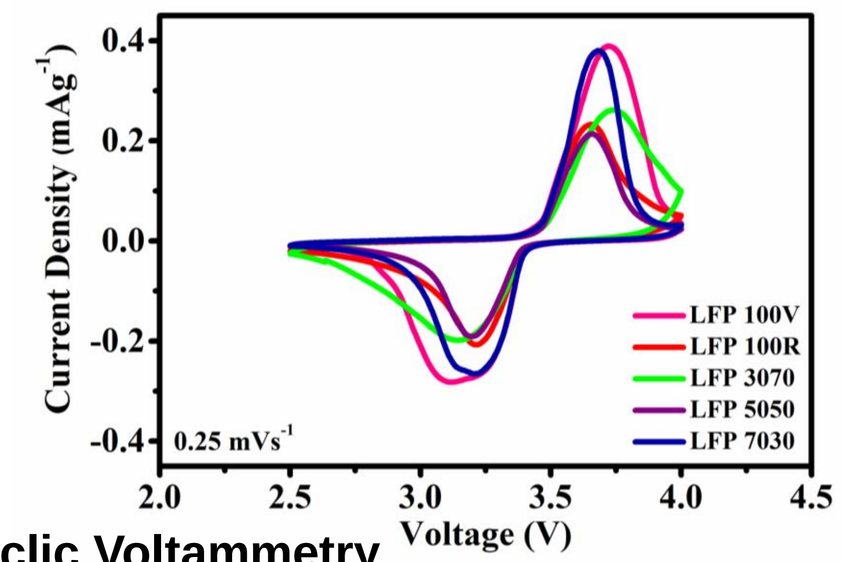
Coin cell composition	ESR (Ω)	Charge transfer resistance (Ω)
LFP 100 V	5.1	190
LFP 100R	2.6	260
LFP 3070	3.9	250
LFP 5050	3.8	290
LFP 7030	4	310



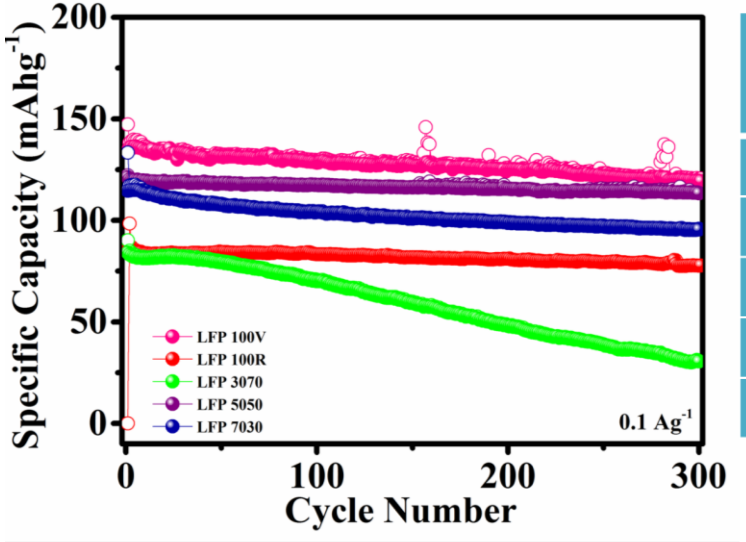
Nyquist impedance for LFP 100V, LFP 100R, LFP 3070, LFP 5050, LFP 7030

Cathode: Electrochemical Characterization

National Chemical Laboratory

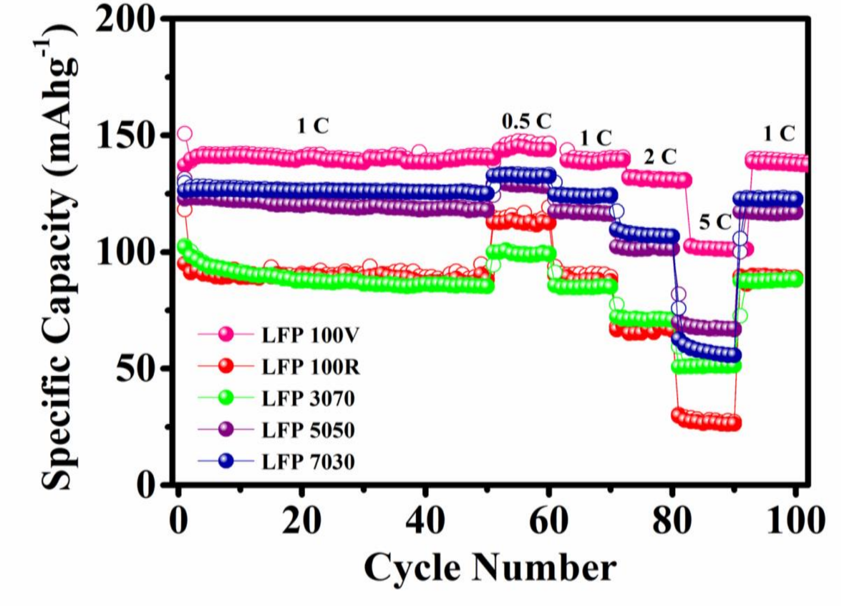


Cyclic Voltammetry



Coin Cell	Specific Capacity (mAg^{-1})	
	2nd Cycle	300th Cycle
LFP 100V	135	120
LFP 100R	88	78
LFP3070	84	31
LFP 5050	120	113
LFP 7030	114	96

Cyclic Stability at 1C rate



Cells	C-rate					
	1C	0.5 C	1C	2C	5C	1C
LFP 100V	140	145	139	130	101	137
LFP 100R	88	112	89	67	26	88
LFP 3070	90	99	85	71	51	88
LFP5050	122	128	117	101	67	117
LFP 7030	126	132	124	107	57	123

Rate capability

Conclusion

As received low cast black mass **recycled LFP-R** tested as cathode material **in composition with LFP-V**, for **second life usage** in coin cell LIB batteries.

We can conclude that the **LFP 5050 composition** showing **better performance** than the other two compositions in the terms of **rate capacity and cyclic stability**. (*80% capacity at 50% cost up to 300 cycles*)

Findings suggest that LFP-R on mixing with new virgin LPF-V are **better suitable for secondary storage application** (Low current application such as stationary storage and powering electronics devices, EVs are considered as primary application)

Thank you

National Chemical Laboratory

CSIR-National Chemical Laboratory, Pune

www.ncl-india.org