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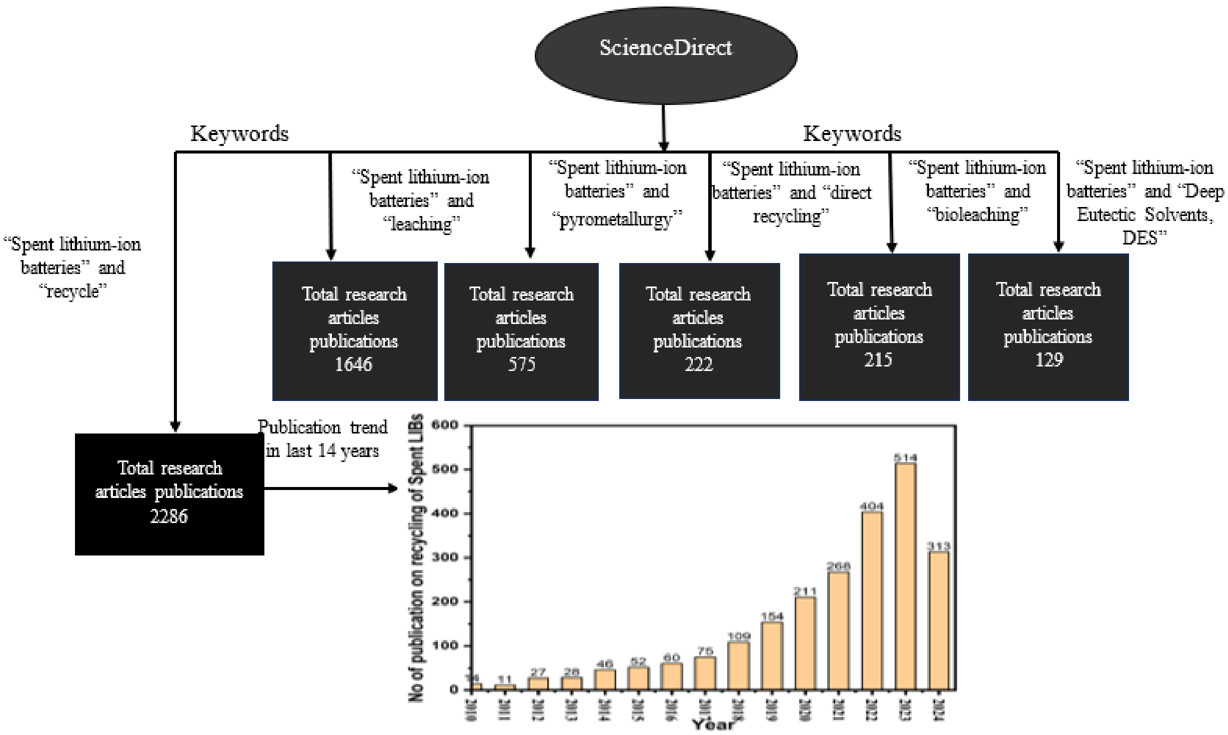
Supplementary material

Sustainable Lithium and Cobalt Recovery from spent lithium-ion batteries: Best Practices for the Future. A Review

1 Supplementary Figures and Tables

1.1 Supplementary Figures



Supplementary Figure 1: Flowchart for the literature review method.

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1.2 Supplementary Tables

Table S1: Residual parameters and conditions of hydrometallurgy used for S-LIBs from literature.

Spent materials	Method	Residue	Efficiency	Purity	Condition	Ref.
$LiCoO_2$	Precipitation	Li_2SO_4	90%	—	4M, H_2SO_4, H_2O_2 [80°C/4 h]	[1]
LIB scraps	Precipitation	LiF	50%	>99wt%	Ethanol, LiOH 500°C/5h, $KHSO_4$ 9 M H_2SO_4, 30 wt%H_2O_2 (3 mLg^{-1}) [90–100 °C] 6 M NaOH	[2]
Cathode materials	Precipitation	Li_2CO_3	80 ± 1%	96.97%	4MHCl, [80 °C] (20 g L^{-1}) Na_2CO_3 [100°C]	[3]
Cathode materials	Precipitation	Li_2CO_3	71%	—	2MH_2SO_4,2% H_2O_2 (33gL^{-1}) [60 °C] Na_2CO_3 [50 °C]	[4]
Mixed cathode materials	Precipitation	Li_2CO_3	80%	—	4M, H_2SO_4, 30wt%H_2O_2 (50gL^{-1}) [70–80 °C] NaOH, Na_2CO_3 [40 °]	[5]
$LiNi_{0.3}Mn_{0.3}Co_{0.3}O_2$	Precipitation	Li_2CO_3	Leaching 99.7%	—	3.0 M trichloroacetic acid, 4.0 vol% H_2O (50 g L^{-1}) [70–80 °C] Saturated Na_2CO_3 solution	[6]
$LiNi_{0.3}Mn_{0.3}Co_{0.3}O_2$	Precipitation	Li_2CO_3	Leaching 99%	—	2 M H_2SO_4 + 4 vol% H_20_2 (50g L^{-1}) [50 °C/2 h] $KMnO_4$, $C_4H_8N_2O_2$ Na_2CO_3 [90 °C]	[7]
$LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$	Precipitation	Li_2CO_3	76%	>99.5%	1Mof oxalic acid (10gL$^{-1)}$ $^{[}$95 °C/12 h] 5MK_2CO_3 [80°C/4 h]	[8]
$LiFePO_4$	Precipitation	Li_2CO_3	99.35%	—	Ball mill with citric acid, H_2O_2 (20 g g^{-1}) SaturatedNa_2CO_3[95 °C]	[8]
Cathode	Precipitation	Li_2CO_3	38%	99.48%	1MH_2SO_4,5vol% H_2O_2 [60°C/1h], sonication 2M NaOH	[9]
$LiFePO_4$	Precipitation	Li_2CO_3	80%	—	4M MSA acid (80g L^{-1}) 18%, H_2O_2 5%NaOH solution, 30%Na_2CO_3 [96°C/30 min]	[10]
$LiNi_{0.5}Co_{0.2}Mn_{0.3}O_2$	Precipitation	Li_2CO_3	91.23%	99%	Sodium persulfate (400 g L^{-1}) [85 °C] Na_2CO_3	[11]
$LiNi_xMn_yCo_{1-x-y}O_2$	Precipitation	Li_3PO_4	Leaching 99.1%	—	750°C/3h,2.75MH_3PO_4 [40°C/10 min]	[12]
$LiNi_{0.3}Mn_{0.3}Co_{0.3}O_2$	Precipitation	Li_2CO_3	Leaching 99%	—	2 M H_2SO_4 + 4 vol% H_20_2 (50g L^{-1}) [50 °C/2 h] $KMnO_4$, $C_4H_8N_2O_2$	[7]

$LiNi_{0.5}Mn_{0.3}Co_{0.2}O_2$	Precipitation	Li_2CO_3	76%	>99.5%	Na_2CO_3 [90 °C] 1Mof oxalic acid (10gL$^{-1)}$ [95 °C/12 h]	[8]
$LiFePO_4$	Precipitation	Li_2CO_3	99.35%	—	5MK_2CO_3 [80°C/4 h] Ball mill with citric acid, H_2O_2 (20 g g^{-1})	[8]
Cathode	Precipitation	Li_2CO_3	38%	99.48%	SaturatedNa_2CO_3[95 °C] 1MH_2SO_4,5vol% H_2O_2 [60°C/1h], sonication	[9]
$LiFePO_4$	Precipitation	Li_2CO_3	80%	—	2M NaOH 4M MSA acid (80g L^{-1}) 18%, H_2O_2	[10]
$LiNi_{0.5}Co_{0.2}Mn_{0.3}O_2$	Precipitation	Li_2CO_3	91.23%	99%	5%NaOH solution, 30%Na_2CO_3 [96°C/30 min] Sodium persulfate (400 g L^{-1}) [85 °C]	[11]
$LiNi_xMn_yCo_{1-x-y}O_2$	Precipitation	Li_3PO_4	Leaching 99.1%	—	Na_2CO_3 750°C/3h,2.75MH_3PO_4 [40°C/10 min] 10 M NaOH	[12]
Cathode materials	Precipitation	Li_2CO_3	>90%	99.93%	3.5Macetic acid (40 g L^{-1}), H_2O_2 4vol% [60°C]	[13]
Cathode materials	Precipitation	Li_3PO_4	85.56%	—	Saturated Na_2CO_3 [20\60 °C] Oxidation at 600 °C, 0.28 M H_2SO_4 + H_2O_2 (16 g L^{-1}) [85°C/2 h]	[14]
Anode	Precipitation	Li_2CO_3	Leaching ≈ 100%	>99%	NaOH, Na_3PO_4 1.5M HCl (100 g L^{-1}) CO_2	[15]

Table S2: Bacterial-based bioleaching for recovery of valuable metals from spent Li-ion batteries (LIBs)[16].

		Recovery efficiency			
Bacteria	**Key leaching condition**	**Co**	**Li**	**Additional information**	**References**
A. thiooxidans (80191)	Pulp density: 0.25% (w/v), pH: 2.4	23%	60%	Co and Li dissolution were higher in two-step bioleaching	[17]
A. ferrooxidans (ATCC 19859)	Solid-to-liquid ratio: 5 g/L, pH: 2.5	65%	9.5%	Higher solid/liquid ratios reduced leaching efficiency	[18]
A. ferrooxidans (DSMZ, 1927)	Pulp density: 100 g/L	94%	60.30%	For optimum metal extraction, replenishment of microbial culture	[19]

				was done every 24 h for 3 cycles	
A. ferrooxidans (DSMZ 1927)	Pulp density: 100 g/L	82%	89%	Leaching efficiency was increased with increase of sulphuric and ferric ion in the leaching medium as well as by replenishing the culture for three cycles	[19]
A. ferrooxidans (DSMZ 1927)	Pulp density: 100 g/L	90.4%	89.9%	NMC (NMC_{111} and NMC_{622}) were regenerated from the oxalate-based coprecipitated product. The electrochemical stability of the regenerated NMC was similar to the commercial NMC.	[20]
A. ferrooxidans (isolated)	Pulp density: 1% (s/v), bacteria inoculation: 5% (v/v), pH: 1.5	47.60%	NA	Enhancement of cobalt dissolution was observed at higher redox potential	[21]
A. thiooxidans (PTCC 1717)	Pulp density: 30 g/L, pH: 2.0	60%	99%	Bioleached spent LIB residue was safe to disposal as meets the TCLP limit	[22]
A. thiooxidans (PTCC 1647)	Pulp density: 40 g/L, pH: 2.0	88%	100%	The shrinking core model predicted that the	[23]

				diffusion of ferric ions plays a key role in metal leaching.	
A. ferrooxidans (isolated)	Pulp density 1% (s/v)	99.90%	NA	Enhancement of cobalt dissolution was noticed with addition of copper ions (0.75 g/L).	[24]
A. ferrooxidans (isolated)	Pulp density 1% (s/v)	98.40%	NA	Enhancement of cobalt dissolution was noticed with addition of silver ions (0.02 g/L).	[25]
A. ferrooxidans (PTCC 1647)	Pulp density: 10 g/L	19.0%	67%	Ultrasonic treatment (203.5 W for 30 min) enhanced metal leaching efficiency.	[26]
A. ferrooxidans (isolated)	pH: 2.5, inoculum concentration: 20% (v/v)	57.8%	NA	Highest Co recovery was found at an inoculum concentration of 20% (v/v) in 14 days of incubation time.	[27]
A. ferrooxidans (isolated)	Pulp density: 10 g/L, pH: 2-4, inoculum concentration: 20% (v/v)	73.95%	NA	Bacterial strain isolated from the acid mine drainage has the potential as oxidizing agent for recovery of metals (Co and Li) from spent LIBs.	[28]
L. ferriphilum (isolated)	Pulp density: 1% (w/v), pH:	NA	49%	Leaching tests were done using pyrite (FeS_2, 16	[29]

	1.0			g/L) as the energy source and LFP as the cathode material.	
A. thiooxidans (isolated)	Pulp density: 1% (w/v), pH: 1.0	NA	98%	Leaching tests were done using S^0 (16 g/L) as the energy source and LFP as the cathode material.	[29]
A. thiooxidans (isolated)	Pulp density: 1% (w/v), pH: 1.0,	NA	97%	Leaching tests were done using S^0 (16 g/L) as the energy source and LMO as the cathode material.	[29]
Mixed bacterial culture (isolated)	Pulp density: 2 g/L, pH: 7.0	NA	63.8%	Adaptation of bacteria with LiCl solution (576 µM) enhanced leaching efficiency of bacteria to Li.	[30]
Mixed culture of IOB and SOB (isolated)	Pulp density 10 g/L, pH: 1.5 (2.0 g/L sulfur + 2.0 g/L FeS_2)	90.00%	80.0%	Acidolysis was the main mechanism for Li dissolution, whereas both acidolysis and redoxolysis contributed for Co dissolution.	[31]
Mixed culture 1 *A. ferrooxidans* and *A. thiooxidans*	Iron sulfate: 36.7 g/L; sulfur: 5.0 g/L, pH: 1.5	50.40%	99.20%	Metal contents in spent LIB residue reduced to below the regulatory standard (USEPA), thus the bioleached LIBs can be reused or	[32]

				disposed safely	
Mixed culture 1 *A. ferrooxidans* and *A. thiooxidans*	Pulp density: 1% (w/v), pH: 2.0	99.95%	NA	High metal extraction yield observed in short time (3 days) in two-step leaching with addition of silver ions (0.02 g/L)	[33]
Mixed culture 1 *A. ferrooxidans* and *A. thiooxidans*	Pulp density: 10% (w/v), pH: 1.8	53.20%	60.00%	Biogenic ferric ion-based critical metal leaching yield was further improved with addition 100 mM H_2SO_4.	[34]
Alicyclobacillus sp. (sulfur-oxidizing) and Sulfobacillus sp. (iron-oxidizing) bacteria		72%	89%	Thermodynamics analysis shows bioleaching has much greater potential to happen compared to chemical leaching.	[35]

Table S3: Fungal-based bioleaching for recovery of valuable metals from spent Li-ion batteries (LIBs) [16].

Fungal	**Key leaching condition**	**Recovery efficiency**		**Additional information**	**References**
		Co	**Li**		

Aspergillus niger (PTCC 5210)	Pulp density: 1% (w/v); pH: 6.0	45%	95%	Spent medium exhibited highest metal extraction yield. Also, bioacids yielded higher metal extraction than synthetic chemical acids.	[36]
A. niger MM1/SG1 (isolated)	Pulp density: 0.25% (w/v); carbon source: sucrose; pH: 3.5	80–82%	100%	Leaching efficiency was higher in cell-free spent medium. Also, bioacids yielded higher metal extraction than the synthetic chemical acid (citric acid).	[37]
A. niger (PTCC 5210)	Pulp density: 1–2% (w/v), carbon source: sucrose	64%	100%	Co and Ni recovery were higher at 1% pulp density, while Li, Cu, Al and Mn recovery was higher at 2% pulp density.	[38]
A. niger (PTCC 5210)	Pulp density: 1 % (w/v), carbon source: sucrose	38%	100%	Adapted fungi showed higher metal leaching performance compared to unadopted fungi.	[39]
A.niger (PTCC 5010)	Pulp density: 10% (w/v), carbon source: glucose; pH: 4.5,	NA	73.3%	*A. niger* showed higher metal leaching performance than *Penicillium chrysogenum*.	[40]
P. chrysogenum (PTCC 5037)	Pulp density: 10% (w/v), carbon source: glucose; pH: 4.5,	NA	54.6%	*A. niger* showed higher metal leaching performance than *P. chrysogenum*.	[40]
A.niger (isolated)	Carbon sources: glucose,	57%	72%	Highest valuable metal recovery obtained in the one step process	[27]

	incubation time: 21 days				
Mixed culture 1 *A. niger* and *Aspergillus tubingensis*	Pulp density: 1% (w/v), carbon source: sucrose, impure sucrose or vinasse	~60%	~95%	Spent medium leaching showed higher metal recovery efficiency with vanasse as the carbon source	[41]

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