Supplementary Information

Low-Voltage Suspension Electrolysis Enables Redox Coupled Synergistic Leaching for Closed Loop Recycling of Mixed NCM-LFP Cathodes

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Table S1 Elemental content analysis of NCM and LFP black powder.

Materials	element					
	Li	Ni	Co	Mn	Fe	C
LFP	3.12	\	\	\	34.85	3.69
NCM	6.23	38.24	13.28	20.64	\	0.05

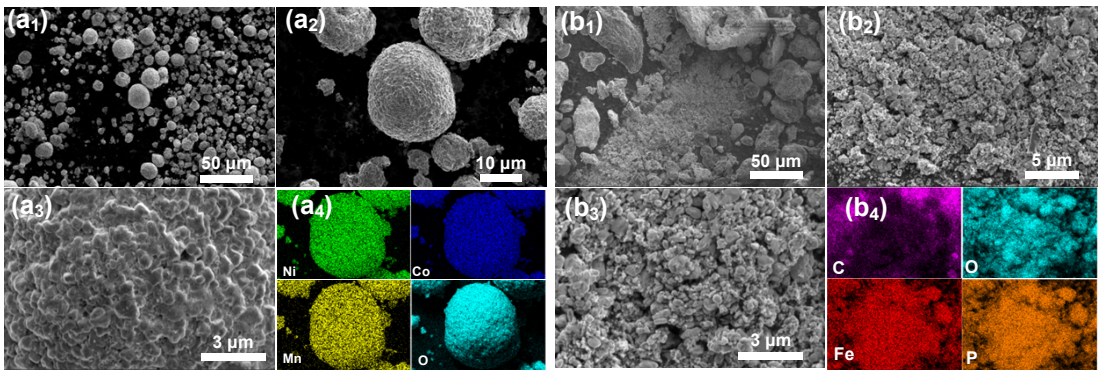


Figure S1 (a) SEM and EDS spectra of NCM and (b) LFP black powder.

Table S2 Calculation Results of EDS elements for NCM and LFP black powder.

material	element	*W*t %
NCM	O	27.54
	Mn	18.91
	Co	14.53
	Ni	39.01
LFP	C	31.83
	O	35.27
	P	10.72
	Fe	22.19

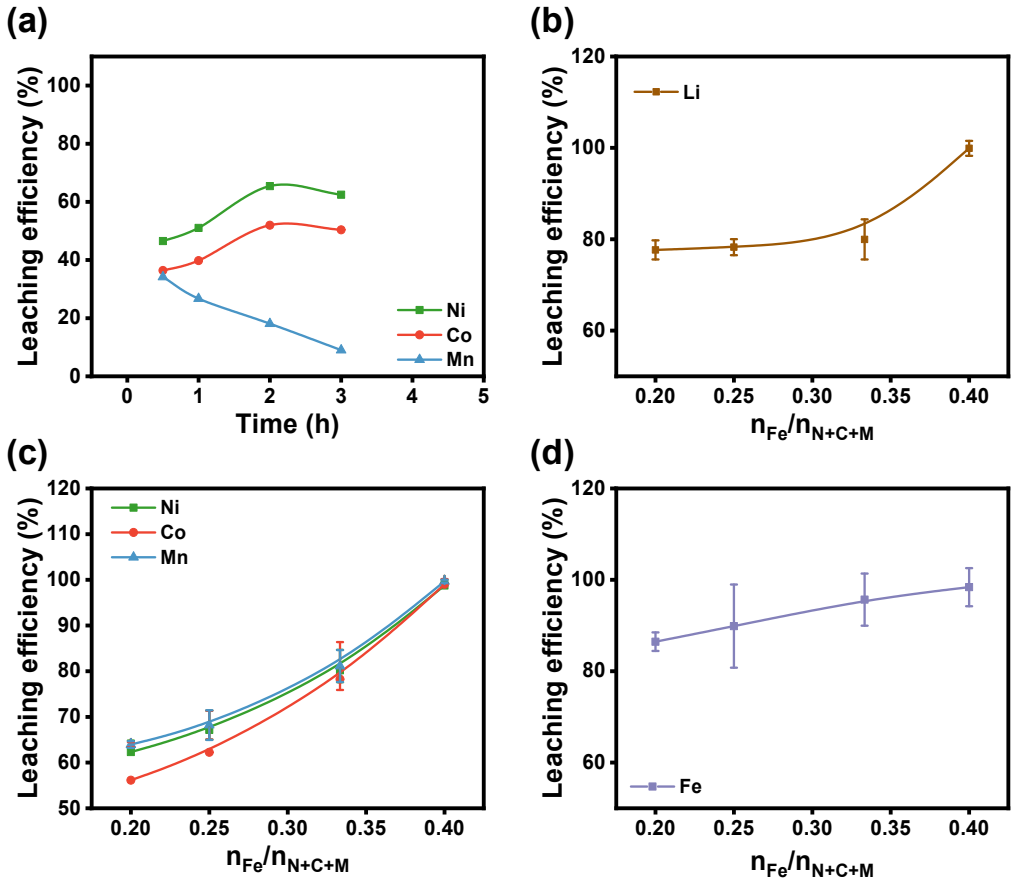


Figure S2 (a) The variation of the leaching efficiency of Ni, Co and Mn with time when $n_{Fe}/n_{(Ni+Co+Mn)}$ =0, (b-d) The effect of different black powder ratios on the leaching efficiency of Li, Ni, Co, Mn and Fe under the conditions of a solid-liquid ratio of 40g L^{-1}, an acid concentration of 1 M H_2SO_4, a working voltage of 1.2 V, a reaction temperature of 40 ℃, and a leaching time of 1 h.

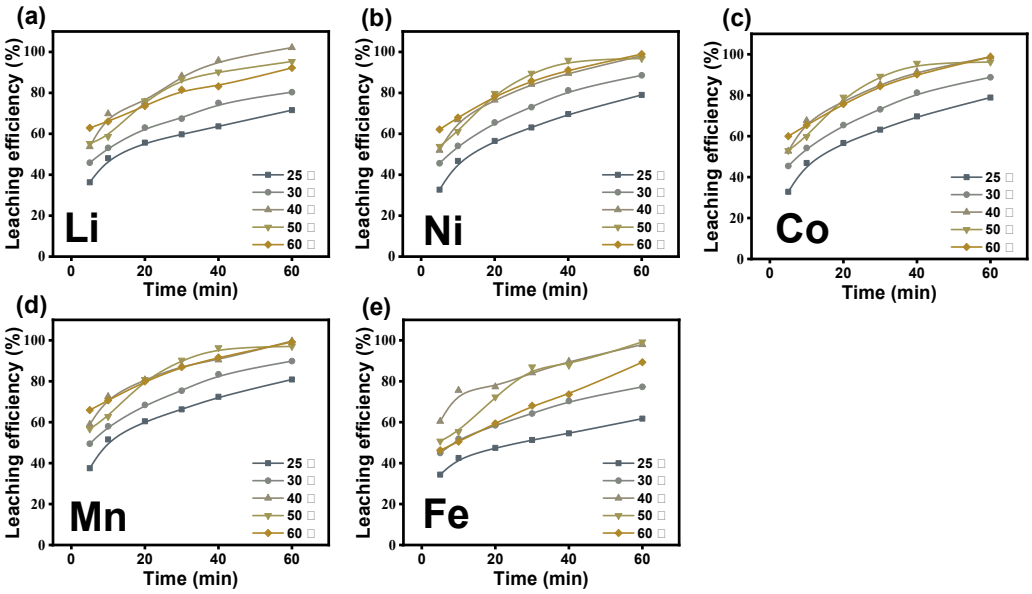


Figure S3 Kinetic data curve for leaching of (a) Li, (b) Ni, (c) Co, (d) Mn, (e) Fe, and (f) Al from spent LIBs at 1 M H_2SO_4, operating voltage of 1.2 V, temperature of 40 °C, leaching time of 1 h, $n_{(Fe)}/n_{(Ni+Co+Mn)}$ = 0.4:1, and solid-liquid ratio of 40 g L^{-1}.

Table S3 Fitting coefficients of kinetic models of Co, Mn, and Li under different temperature parameters

	Equation	Temperature (℃)	Fitting Result	R_1^2	Fitting Result of lnk=-Ea/RT+A	R_2^2	Ea (kJ mol^{-1})
Li	$y = 1-(1-x)^{1/3}$	25	y = 0.002935 x + 0.172007	0.980088	y = -7.790872 x + 20.317386	0.998603	64.77331
		30	y = 0.004723 x + 0.178968	0.984696			
		40	y = 0.010348 x + 0.196695	0.980328			
	$y = 1-(1-x)^{2/3}$	25	y = 0.004451 x + 0.318194	0.974946	y = -5.765635 x + 13.955499	0.991695	47.93549
		30	y = 0.006640 x + 0.337004	0.983417			
		40	y = 0.011412 x + 0.414167	0.974426			
	$y = 1-2/3x-(1-x)^{2/3}$	25	y = 0.001111 x + 0.023295	0.995937	y = -9.000998 x + 23.360122	0.997595	74.8343
		30	y = 0.001708 x + 0.025395	0.992023			
		40	y = 0.004658 x + 0.020471	0.993772			
Ni	$y = 1-(1-x)^{1/3}$	25	y = 0.004279 x + 0.152706	0.996768	y = -4.179018 x + 8.587993	0.990009	34.74436
		30	y = 0.005751 x + 0.180683	0.989986			
		40	y = 0.008476 x + 0.211659	0.99166			
	$y = 1-(1-x)^{2/3}$	25	y = 0.005985 x + 0.298443	0.989157	y = -1.773233 x + 0.861590	0.916094	14.74266
		30	y = 0.007169 x + 0.357092	0.973912			
		40	y = 0.008082 x + 0.452514	0.996026			
	$y = 1-2/3x-(1-x)^{2/3}$	25	y = 0.001857 x + 0.009525	0.994277	y = -4.815397 x + 9.898800	0.984562	40.03521
		30	y = 0.002659 x + 0.017827	0.996093			
		40	y = 0.004096 x + 0.025319	0.994093			
Co	$y = 1-(1-x)^{1/3}$	25	y = 0.004241 x + 0.154189	0.996381	y = -4.710381 x + 10.356114	0.994967	39.16211
		30	y = 0.005794 x + 0.180129	0.991311			
		40	y = 0.009127 x + 0.207978	0.994701			
	$y = 1-(1-x)^{2/3}$	25	y = 0.006707 x + 0.283996	0.993369	y = -1.835003 x + 1.214810	0.747953	15.25621
		30	y = 0.008743 x + 0.323893	0.997116			

		40	y = 0.009288 x + 0.437776	0.999243			
		25	y = 0.001846 x + 0.009914	0.993745			
	$y = 1-2/3x-(1-x)^{2/3}$	30	y = 0.002683 x + 0.017470	0.996572	y = -5.224422 x + 11.261685	0.988926	43.43584
		40	y = 0.004342 x + 0.025144	0.99669			
		25	y = 0.004147 x + 0.178815	0.996702			
	$y = 1-(1-x)^{1/3}$	30	y = 0.005711 x + 0.202828	0.987883	y = -5.289693 x + 12.267193	0.998865	43.97851
		40	y = 0.009751 x + 0.221614	0.985136			
		25	y = 0.005620 x + 0.341041	0.989323			
Mn	$y = 1-(1-x)^{2/3}$	30	y = 0.006884 x + 0.392423	0.996668	y = -1.908013 x + 1.256623	0.900655	15.86322
		40	y = 0.007777 x + 0.503904	0.990164			
		25	y = 0.001785 x + 0.021625	0.999263			
	$y = 1-2/3x-(1-x)^{2/3}$	30	y = 0.002720 x + 0.024997	0.993469	y = -5.719268 x + 12.895694	0.985956	47.54999
		40	y = 0.004563 x + 0.037436	0.99476			
		25	y = 0.002078 x + 0.149584	0.998225			
	$y = 1-(1-x)^{1/3}$	30	y = 0.003508 x + 0.184461	0.99395	y =-8.672584 x + 22.929016	0.998916	72.10386
		40	y = 0.008440 x + 0.209722	0.992145			
		25	y = 0.003231 x + 0.280803	0.996936			
Fe	$y = 1-(1-x)^{2/3}$	30	y = 0.004874 x + 0.345755	0.988066	y = -4.990753 x + 11.057580	0.969893	41.49312
		40	y = 0.007389 x + 0.482744	0.999602			
		25	y = 0.000716 x + 0.017776	0.997201			
	$y = 1-2/3x-(1-x)^{2/3}$	30	y = 0.001473 x + 0.024959	0.997033	y = -10.294137 x + 27.345033	0.990851	85.58546
		40	y = 0.003851 x + 0.034742	0.995552			

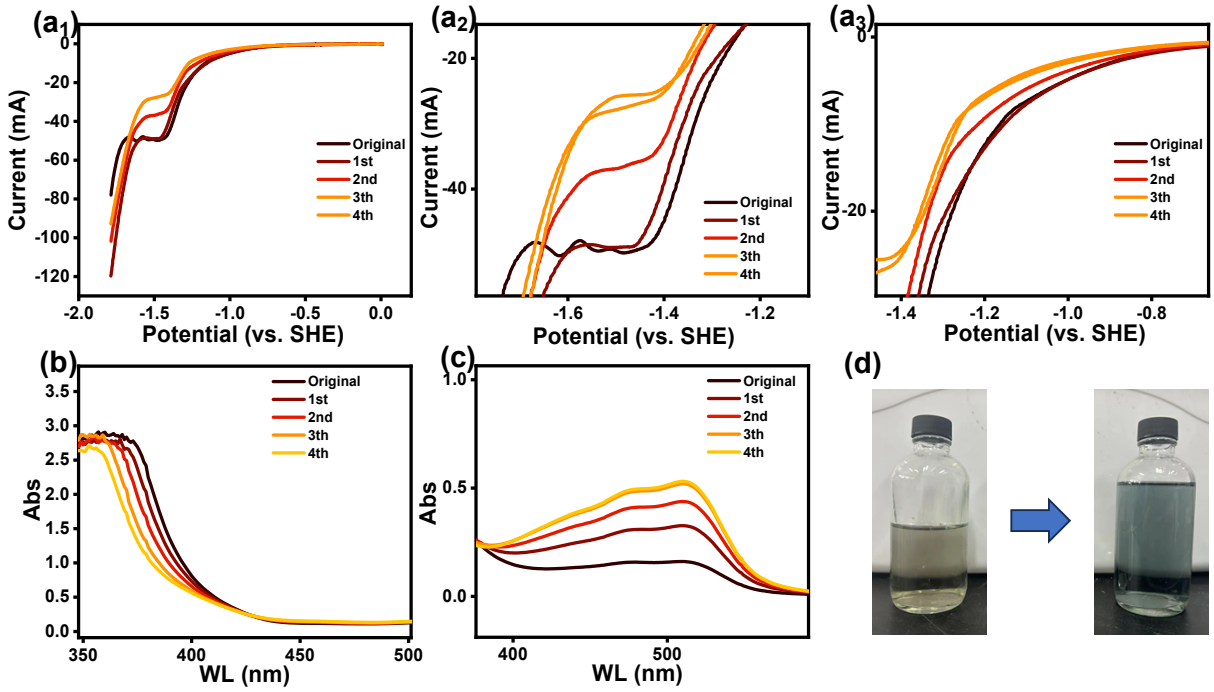


Figure S4 (a) LSV curves of the leachate, (b) UV-Vis tests of the solution under different LSV scan times, (c) UV-Vis tests of the solution after adding o-phenanthroline under different LSV scan times, (d) photos of the solution before and after scanning

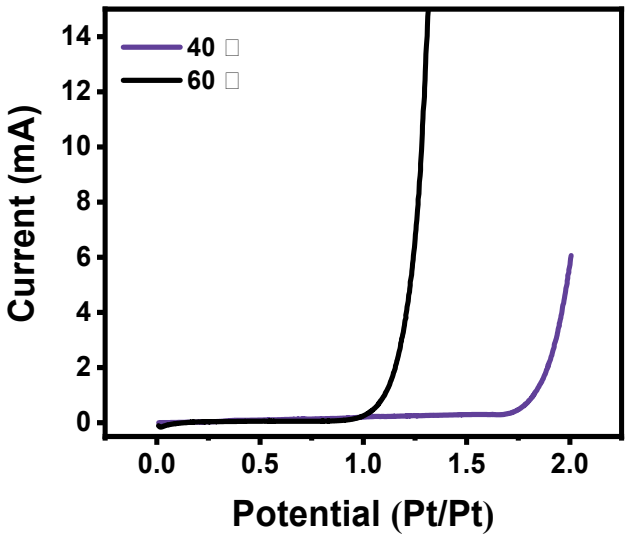


Figure S5 The LSV test results of the Pt/Pt electrode at different temperatures in 1M H_2SO_4 solution。

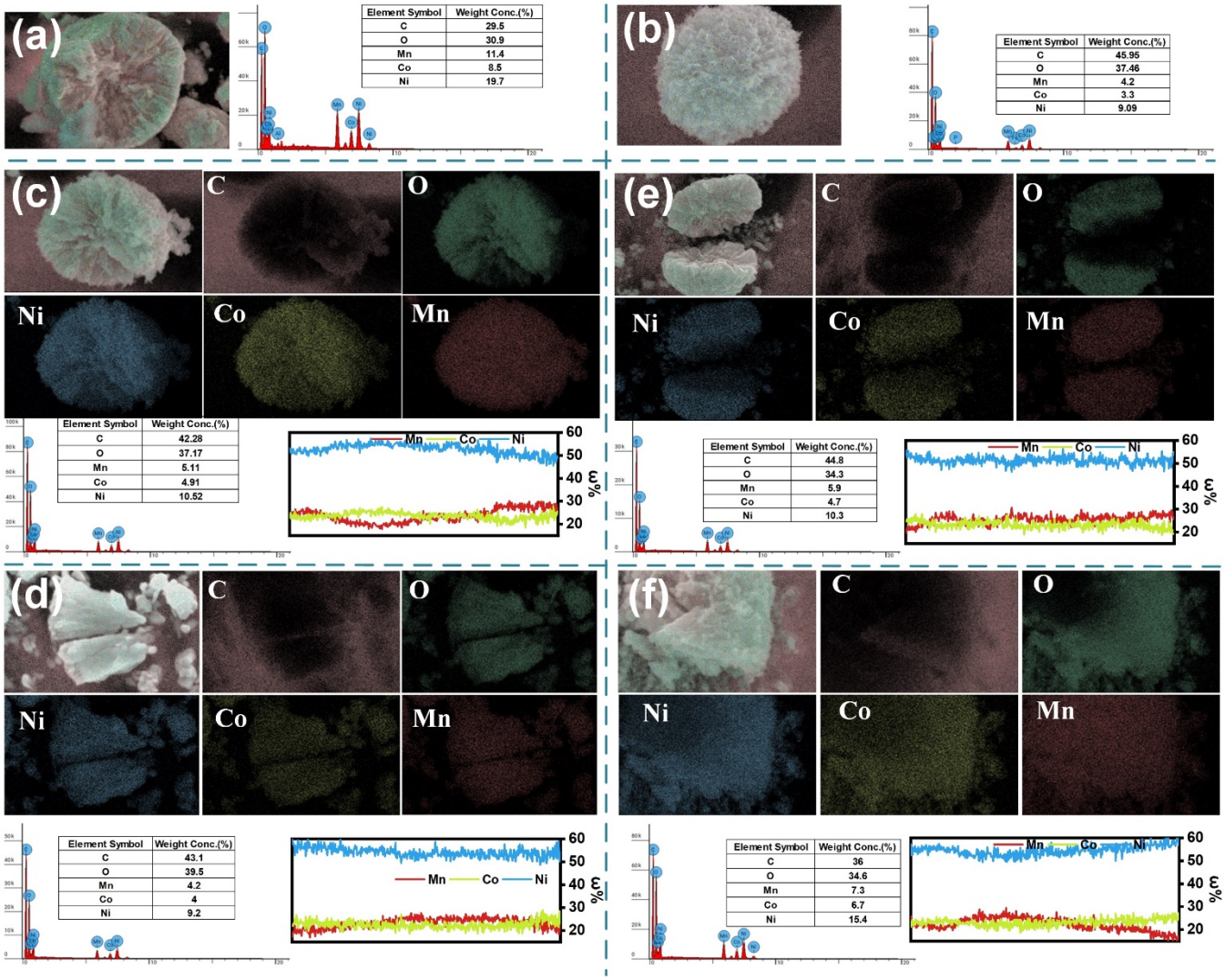
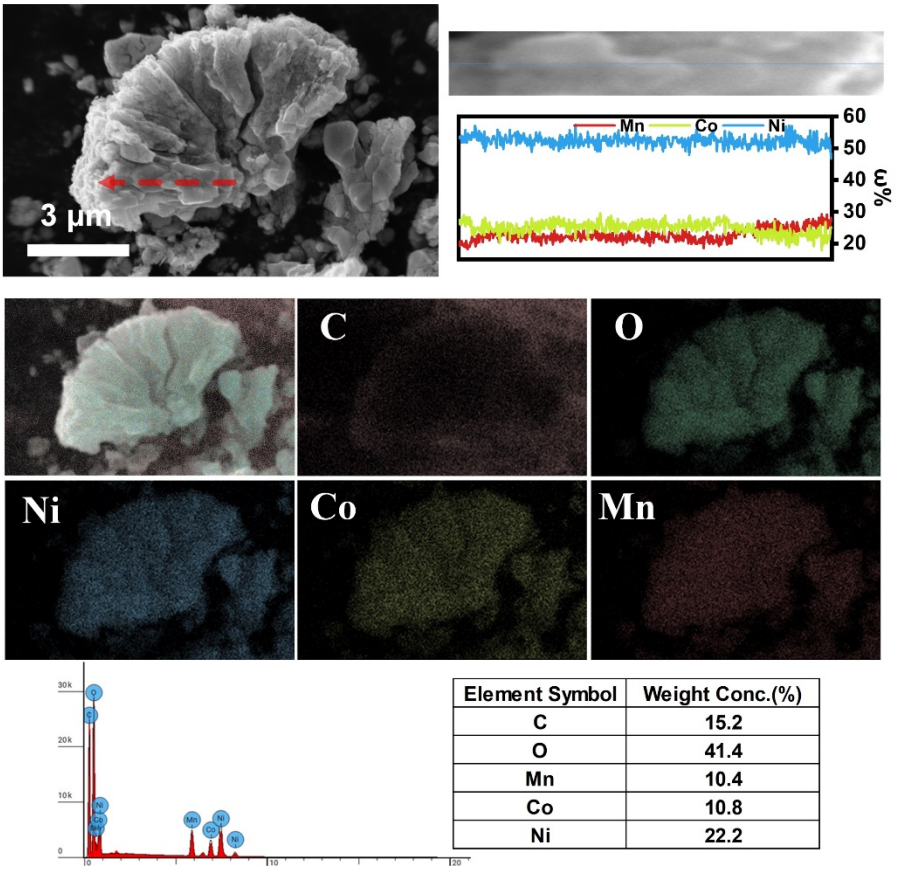


Figure S6 Cross-sectional images of (a) pristine NCM particles, (b) NCM particles after leaching for 10 min under the optimal leaching conditions, and NCM particles after leaching for (c) 10 min and (d) 20 min under the optimal leaching conditions. SEM images, EDS mapping results, and Ni, Co, and Mn line-scan profiles of (e) Group A and (f) Group D after leaching for 10 min.



Element Symbol	Weight Conc.(%)
C	15.2
O	41.4
Mn	10.4
Co	10.8
Ni	22.2

Figure S7 Sectional SEM and EDS linear scanning results of Group F at reaction time 1 h.

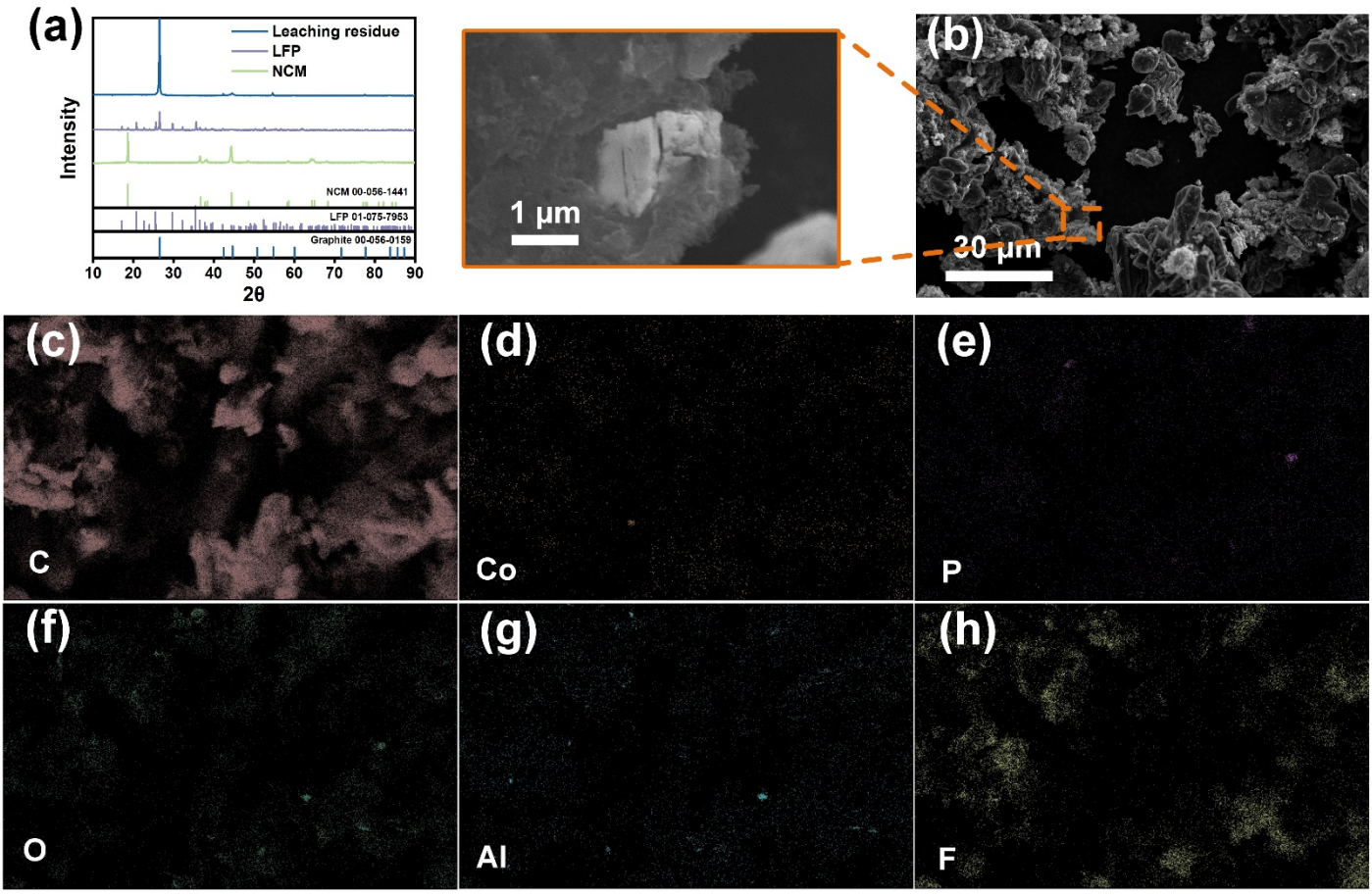


Figure S8 (a) XRD results of raw materials and leaching residue, (b) SEM results of leaching residue and (c-h) EDS results of different elements.

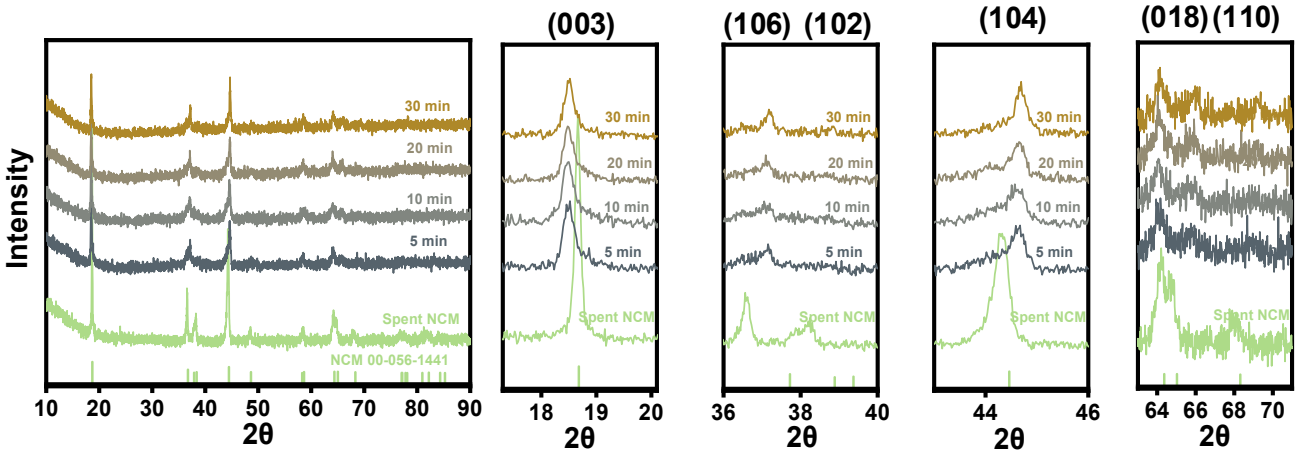


Figure S9 (a) XRD patterns of leaching residues of Group D

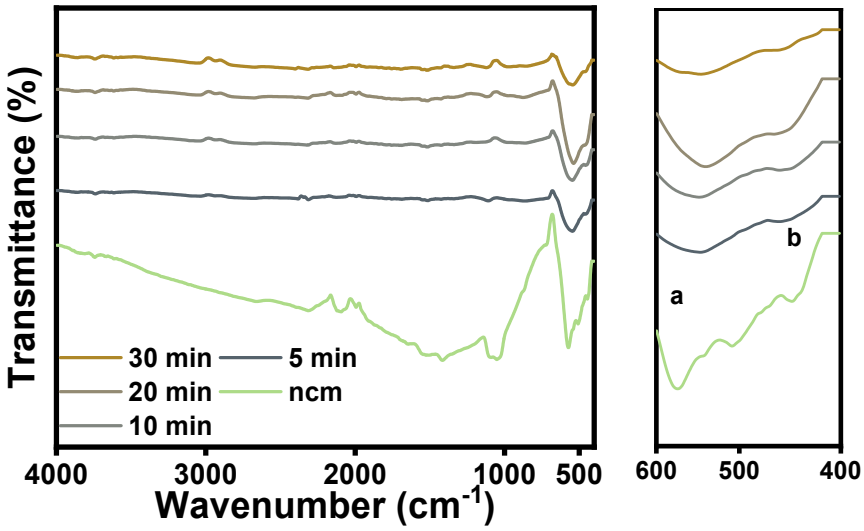


Figure S10 (a) FT-IR patterns of leaching residues after 30 min leaching of Group D.

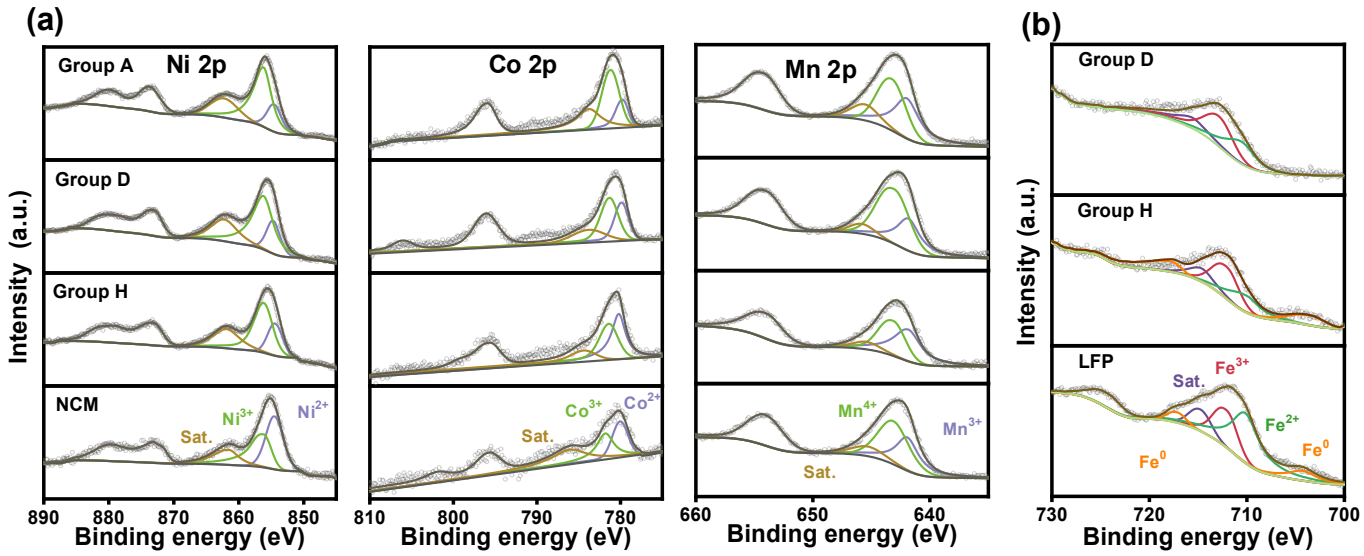


Figure S11 (a) XPS results of Ni, Co and Mn in the leachate residue of NCM and different groups after leaching for 30 min, (b) XPS results of Fe in the leachate residue of LFP and different groups after leaching for 30 min.

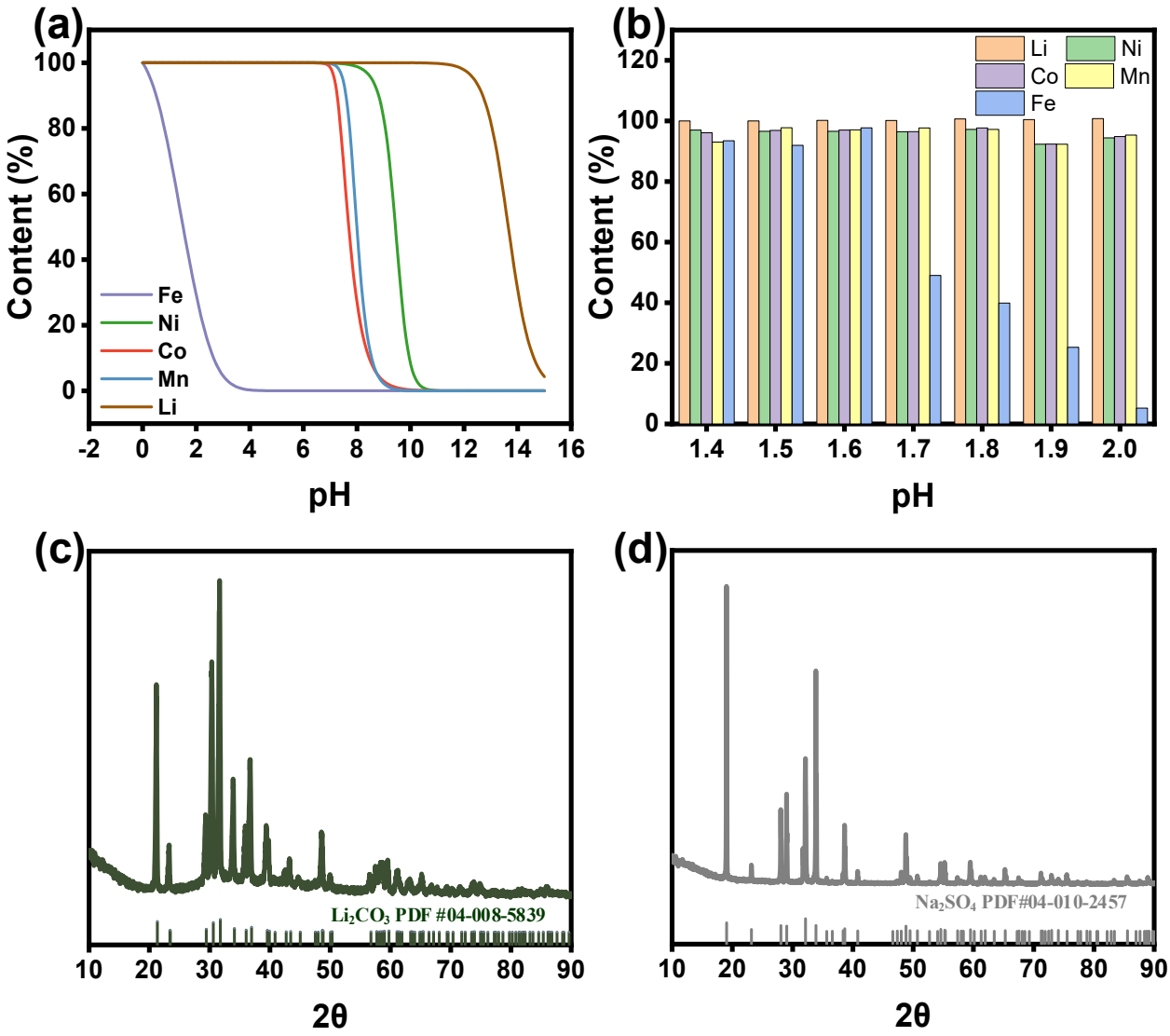


Figure S12 (a) Retention rates of different elements in the leachate at different pH values, (b) Concentration changes of each element in the leachate at different pH values. XRD of (c) Li_2CO_3 and (d) Na_2SO_4 obtained by precipitation

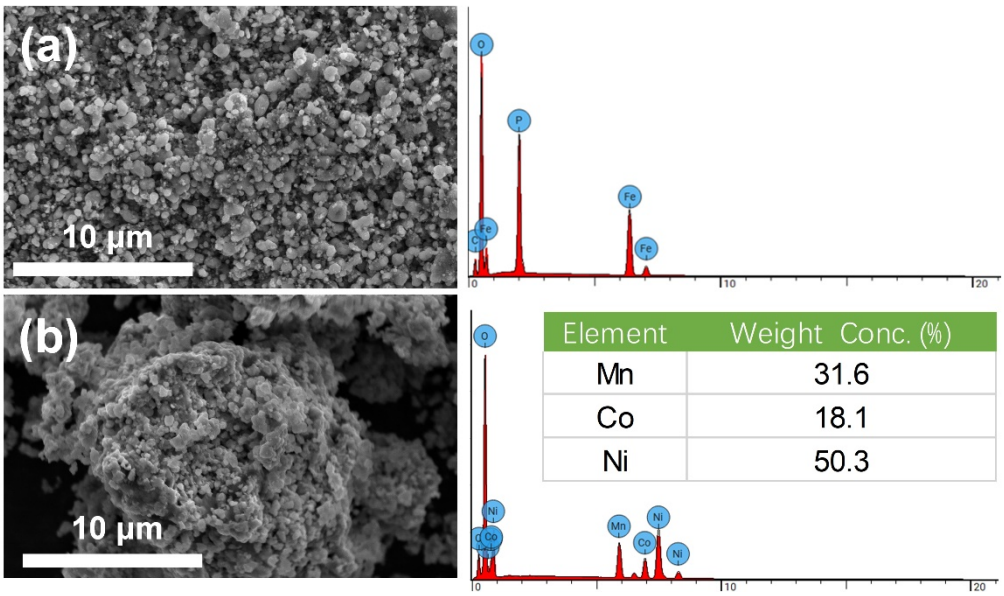


Figure S13 SEM and EDS images of regenerated (a) LFP and (b) NCM;

Table S4 Calculation Process of Economic Analysis

	Substance	Amount (kg)	Price ($/kg)	Costs ($)
Raw materials expenditure	spent NCM	0.58	9.36	5.43
	spent LFP	0.42	1.41	0.59
	H_2SO_4	1.357	0.1	0.13
	NaOH	2.04	0.35	0.71
	$C_2H_2O_4·2H_2O$	1.14	0.39	0.44
	Na_2CO_3	0.89	0.16	0.15
	Li_2CO_3	0.057	10.67	0.6
	Total	8.05		
	Unit operation		Power consumption (kW h)	Costs ($)
Energy expenditure	Suspended electrolysis	Electrolysis	0.006	0.21
		Heating and stirring	3	
		Filtration	0.055	
		Total	3.061	
	Extraction leachate	Stirring	0.2	0.02
		Filtration	0.055	
		Total	0.255	
	Co-precipitation	Heating and stirring	3	0.21
		Filtration	0.055	
		Total	3.055	
	Chemical precipitation	stirring	0.2	0.02
		Filtration	0.055	
		Total	0.255	
	Evaporative crystallization	Heating and stirring	5.6	0.39
		Filtration	0.055	
		Total	5.655	
	Oxidizing roasting	Roasting	5.5	0.38
	Reducing roasting	Roasting	4.5	0.32
	Total	1.55		

	Substance	Amount （kg）	Price ($/kg)	Price ($/t)
Product	NCM	0.55	17.27	9.5
	LFP	0.39	4.78	1.87
	$Na_2SO_4 \cdot 10H_2O$	8.06	0.15	1.21
	NaOH	0.33	0.35	0.12
	Total Benefit ($/kg)	12.70		
Profits ($/kg)	3.10			

In this calculation, the prices of raw materials and products are based on the average prices in February 2026, sourced from https://www.alibaba.com/.

The electricity fee is calculated at the average electricity price in Shaanxi Province, China, of 0.5 CNY per kW·h, and the RMB to USD exchange rate is set at 1 USD = 7.12 CNY.

The energy consumption during battery production is calculated in accordance with the Hunan Provincial Standard of China *Energy Consumption Quota and Calculation Method for Unit Product of Lithium Battery Cathode Materials (DB43/T1591-2019)*.