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Supplementary Materials: Ageing and water-based processing of $LiFeMnPO_4$ secondary agglomerates and its effects on electrochemical characteristics

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The following section provides the complete list of refined parameter values of the diffraction data used in the manuscript.

All refinements were carried out starting with the following reference data sets for Al and $LiFe_{0.27}Mn_{0.73}PO_4$ (LFMP) from the Inorganic Crystal Structure Database (ICSD) maintained by the FIZ Karlsruhe as given in Tables S1 and S2. The occupancy value for Li1 in LFMP was set to 1 prior to the refinements. Occupancy factors of Fe and Mn were constrained to unity and refined together for neutron diffraction data. The refined parameters and their corresponding standard uncertainties are listed below in Table S3. The refinements were carried out using the Highscore software package [1] with THOMPSON-COX-HASTINGS (TCH) type pseudo-Voigt profile functions either as RIETVELD [2] or PAWLEY [3] fit as implemented in the software.

Table S1: Al Phase starting parameters. B_{iso} is the abbreviation for isotropic displacement parameter.

Al

ICSD #-43423, [4]

Space group: $F\,m\,\bar{3}\,m$ (No. 225)

a = 4.0497(0) Å

V = 66.42(0) Å^3

Z = 4

Atom	Wyckoff position	occupancy	fractional coordinates			B_{iso} (Å^2)
			x	y	z	
Al	4a	1	0	0	0	Not determined

Table S2: LFMP Phase literature parameters, Li occupancy was set to 1 prior to starting the refinements. B_{iso} is the abbreviation for isotropic displacement parameter.

$LiFe_{0.27}Mn_{0.73}PO_4$

ICSD #-54826, [5]*

Space group: $P\ n\ m\ a$ (No. 62)

a = 10.4084(9) Å

b = 6.0705(4) Å

c = 4.7247(2) Å

V = 298.53(3) Å^3

Z = 4

Atom	**Wyckoff position**	**occupancy**	**fractional coordinates**			**B_{iso} (Å^2)**
			x	***y***	***z***	
Li1	4*a*	1	0	0	0	1.59(8)
Mn1	4*c*	0.73	0.28183(3)	¼	0.02747(8)	0.558(6)
Fe1	4*c*	0.27	0.28183(3)	¼	0.02747(8)	0.558(6)
P1	4*c*	1	0.09309(6)	¼	0.5887(1)	0.560(9)
O1	4*c*	1	0.0968(2)	¼	0.2657(4)	0.85(3)
O2	4*c*	1	0.4559(2)	¼	0.7896(4)	0.86(3)
O3	8*d*	1	0.3375(1)	0.5487(2)	0.2213(2)	0.86(2)

* Structure originally published in space group *P b n m*. The structure was transformed to the standard setting *P n m a*. Transformed data set as presented here was used as starting model for the refinements.

Table S3: Instrumental setup. K_{α_2} / K_{α_1} ratios were determined by refinement using a Si-powder standard (NIST 640d).

Experimental setup					
Sample name	**Pristine**	**23 °C/35% rh**	**40 °C/100% rh**	**NMP**	**H_2O**
Method	ND	ND	ND	XRD	XRD
Instrument setup geometry	Debye-Scherrer	Debye-Scherrer	Debye-Scherrer	Debye-Scherrer	Debye-Scherrer
Step size	0.05	0.05	0.05	0.008	0.008
Wavelength (Å)	1.5482	1.5482	1.5482	0.70932	0.70932
Exposure time (h)	7	7	7	3	19
$K_{\alpha_2}/K_{\alpha_1}$ ratio	monochromator	monochromator	monochromator	0.328	0.328

Table S4: R-values of the refinements and not phase-specific (general) parameters.

R-values, %					
Sample name	**Pristine**	**23 °C/35% rh**	**40 °C/100% rh**	**NMP**	**H_2O**
R_{Bragg} (LFMP)	1.14	1.34	0.84	3.15	2.66
R_{Bragg} (Al)	-	-	-	0.78	0.86
R_{wp}	2.3564	2.7104	1.4808	6.6040	4.8384
Refined general parameters					
Zero Shift (° 2θ)	-0.0094(2)	-0.0213(3)	-0.010(4)	-0.0026(3)	-0.0023(4)
Sample displacement (mm)	-	-	-	0.086(2)	0.095(3)

Table S5: refined Al phase parameters.

Refined Al phase parameters (PAWLEY **–Fit)**					
Sample name	**Pristine**	**23 °C/35% rh**	**40 °C/100% rh**	**NMP**	**H_2O**
S/L Asymmetry	-	-	-	0.001(3)	0.00(6)
D/L Asymmetry	-	-	-	0.000(3)	0.00(6)
TCH-Profile *W*	-	-	-	0.001(1)	0.0014(5)
TCH-Profile *U*	-	-	-	0.03(9)	0.04(4)
TCH-Profile *X*	-	-	-	0.0(4)	0.0(2)
TCH-Profile Peak Shape	-	-	-	0.337(8)	0.295(3)

Table S6: Refined LFMP phase parameters. Standard uncertainty values are given in parentheses as refined by Highscore without multiplying the values with the R_{wp}-value of the fit. B_{iso} is the abbreviation for isotropic displacement parameter.

Refined LFMP phase parameters (RIETVELD **-Fit)**					
Sample name	**Pristine**	**23 °C/35% rh**	**40 °C/100% rh**	**NMP**	**H_2O**
Scale Factor	39.99(3)	29.11(3)	23.28(4)	0.00008(1)	0.000391(1)
Cell axis *a* (Å)	10.41128(4)	10.4125(5)	10.41263(7)	10.4119(3)	10.4092(3)
Cell axis *b* (Å)	6.07066(2)	6.07113(3)	6.07085(4)	6.0705(1)	6.06889(8)
Cell axis *c* (Å)	4.72981(2)	4.73063(2)	4.73114(3)	4.73055(9)	4.72931(4)
Preferred Orientation:					
Model: March-Dollase[6]	0.9897(3)	0.9857(4)	0.9839(6)	0.988(1)	0.9935(7)
Direction: (1 0 1)					
S/L Asymmetry	0.0486(1)	0.0335(1)	0.0498(2)	0.013(1)	0.012(10)
D/L Asymmetry	0.0195(1)	0.0355(2)	0.0216(5)	0.013(1)	0.012(10)
TCH-Profile *W*	0.196(1)	0.199(2)	0.196(2)	0.00471(6)	0.0055(1)
TCH-Profile *V*	-0.125(3)	-0.122(4)	-0.123(6)	-	-
TCH-Profile U	0.084(3)	0.080(4)	0.077(5)	-	-
TCH-Profile *X*	0.00(2)	0.00(2)	0.00(3)	-	-
TCH-Profile Peak Shape	0.126(2)	0.077(2)	0.101(4)	0.63(1)	0.55(1)
Overall B_{iso} (Å²)	-	-	-	1.44(2)	1.25(2)
Li1 B_{iso} (Å²)	2.21(2)	3.18(3)	3.08(5)	-	-
Mn1/Fe1 fractional coordinate *x*	0.2820(5)	0.2855(6)	0.2843(10)	-	-
Mn1/Fe1 fractional coordinate *z*	0.026(1)	0.053(1)	0.058(2)	-	-
Mn1/Fe1 B_{iso} (Å²)	0.5(1)	0.5(2)	0.5(3)	-	-
Mn1/Fe1 occupancy factor	0.6933(6)/ 0.3067(6)	0.6933(7)/ 0.3067(7)	0.693(1)/ 0.307(1)	-	-
P1 fractional coordinate *x*	0.09360(4)	0.09375(4)	0.09352(7)	-	-
P1 fractional coordinate *z*	0.58852(7)	0.58826(9)	0.5884(1)	-	-
P1 B_{iso} (Å²)	0.649(5)	0.782(6)	0.708(9)	-	-
O1 fractional coordinate *x*	0.09759(3)	0.09758(4)	0.09776(7)	-	-
O1 fractional coordinate *z*	0.26579(7)	0.2658(9)	0.2661(1)	-	-
O1 B_{iso} (Å²)	0.921(5)	1.078(7)	0.99(1)	-	-
O2 fractional coordinate *x*	0.45591(3)	0.45571(4)	0.45574(6)	-	-
O2 fractional coordinate *z*	0.79014(8)	0.7888(1)	0.7894(2)	-	-
O2 B_{iso} (Å²)	0.960(5)	1.226(7)	1.17(1)	-	-
O3 fractional coordinate *x*	0.33669(2)	0.33637(3)	0.33631(5)	-	-
O3 fractional coordinate *y*	0.54812(3)	0.54845(4)	0.54822(7)	-	-
O3 fractional coordinate *z*	0.22088(5)	0.22037(7)	0.2206(1)	-	-
O3 B_{iso} (Å²)	0.950(3)	1.180(5)	1.100(7)	-	-

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