

Synthesis and electrochemical studies of layer-structured metastable α_1 -LiVPO₄

A. Shahul Hameed,^a Mangayarkarasi Nagarathinam,^a M. V. Reddy,^{*b} B. V. R. Chowdari^b and Jagadese J. Vittal^{**ac}

Received (in XXX, XXX) Xth XXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX
DOI: 10.1039/b000000x

Table S1 Crystallographic data and structure refinement details for LiVPO₄·2H₂O

| | |
|--|---|
| Formula | H4 Li O7 P V |
| Formula weight | 204.88 |
| T (K) | 223(2) |
| Crystal system | Orthorhombic |
| Space group | Cmca |
| Unit cell dimensions (Å) | a = 8.9454(7) b = 9.0406(7) c = 12.7473(10) |
| V/ Å ³ | 1030.90(14) |
| Z | 8 |
| Density (calculated, g. cm ⁻³) | 2.640 |
| μ (mm ⁻¹) | 2.206 |
| Crystal size (mm ³) | 0.40 x 0.36 x 0.16 |
| Theta range | 3.20 to 27.48°. |
| Reflections collected | 3374 |
| Independent reflections | 633 [R(int) = 0.0230] |
| Absorption correction | Semi-empirical from equivalents |
| Max. and min. transmission | 0.7192 and 0.4724 |
| Refinement method | Full-matrix least-squares on F ² |
| Data / restraints / parameters | 633 / 0 / 61 |
| Goodness-of-fit on F ² | 1.175 |
| Final R indices [I>2σ(I)] | R ₁ = 0.0250, wR ₂ = 0.0679 |
| R indices (all data) | R ₁ = 0.0252, wR ₂ = 0.0681 |
| Largest diff. peak and hole | 0.261 and -0.718 e.Å ⁻³ |

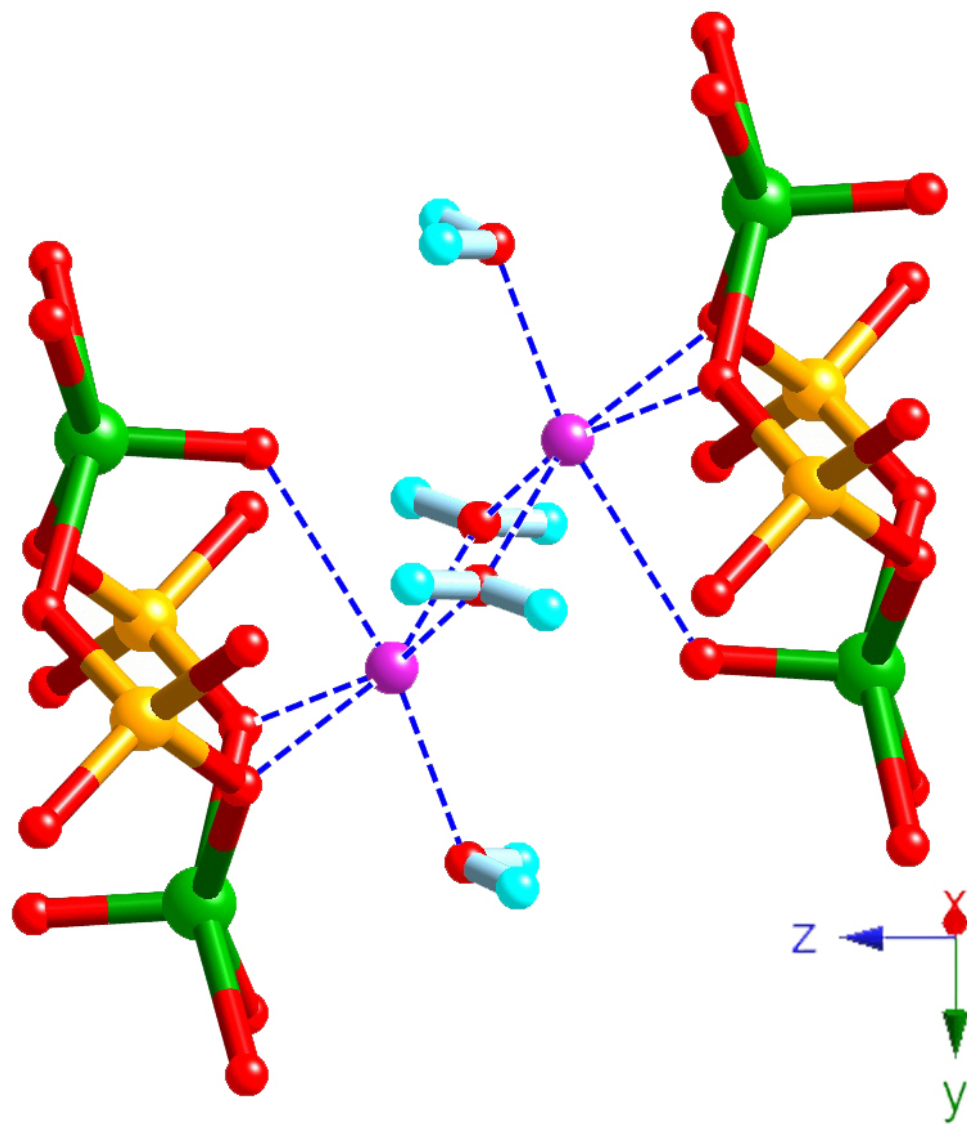


Fig. S2 Crystal structure of $\text{LiVOPO}_4 \cdot 2\text{H}_2\text{O}$ showing coordination of lithium with oxygen atoms. The V, P, O, H and Li atoms are shown in green, orange, red, blue and purple respectively.

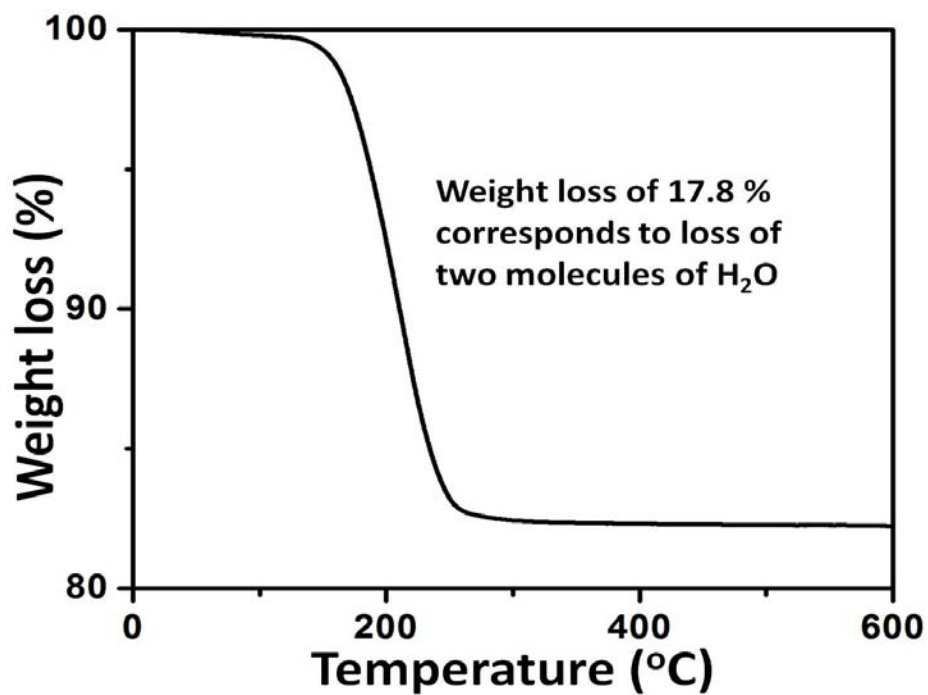


Fig. S3 TGA of $\text{LiVOPO}_4 \cdot 2\text{H}_2\text{O}$ in air at a rate of 5°C min^{-1}

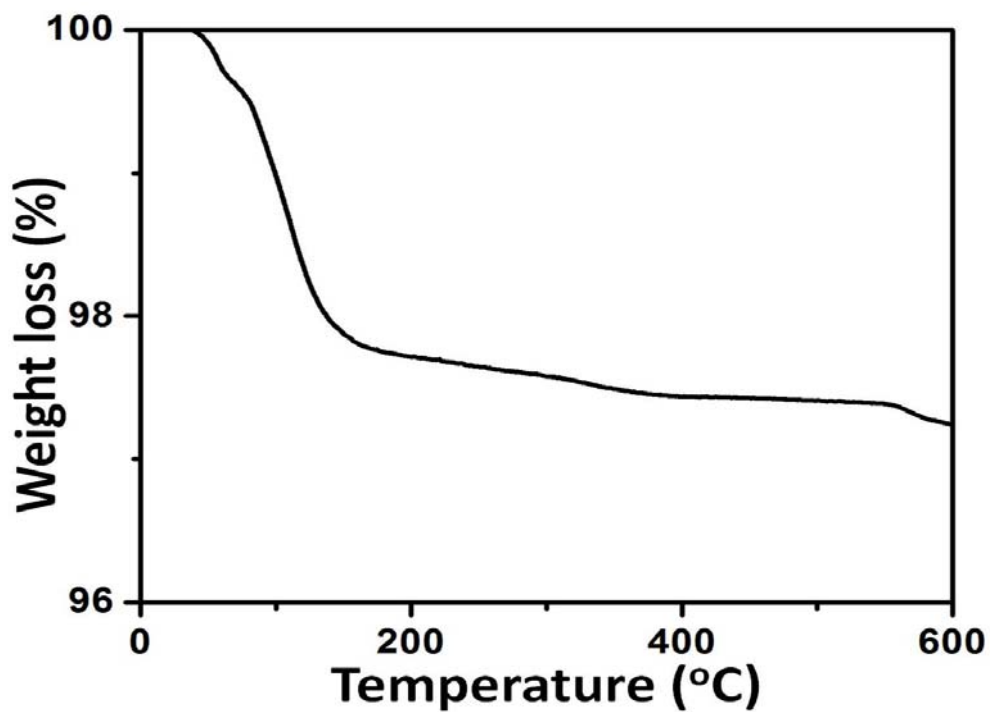


Fig. S4 TGA of compound obtained by dehydration of $\text{LiVOPO}_4 \cdot 2\text{H}_2\text{O}$ in air at a rate of 5°C min^{-1}

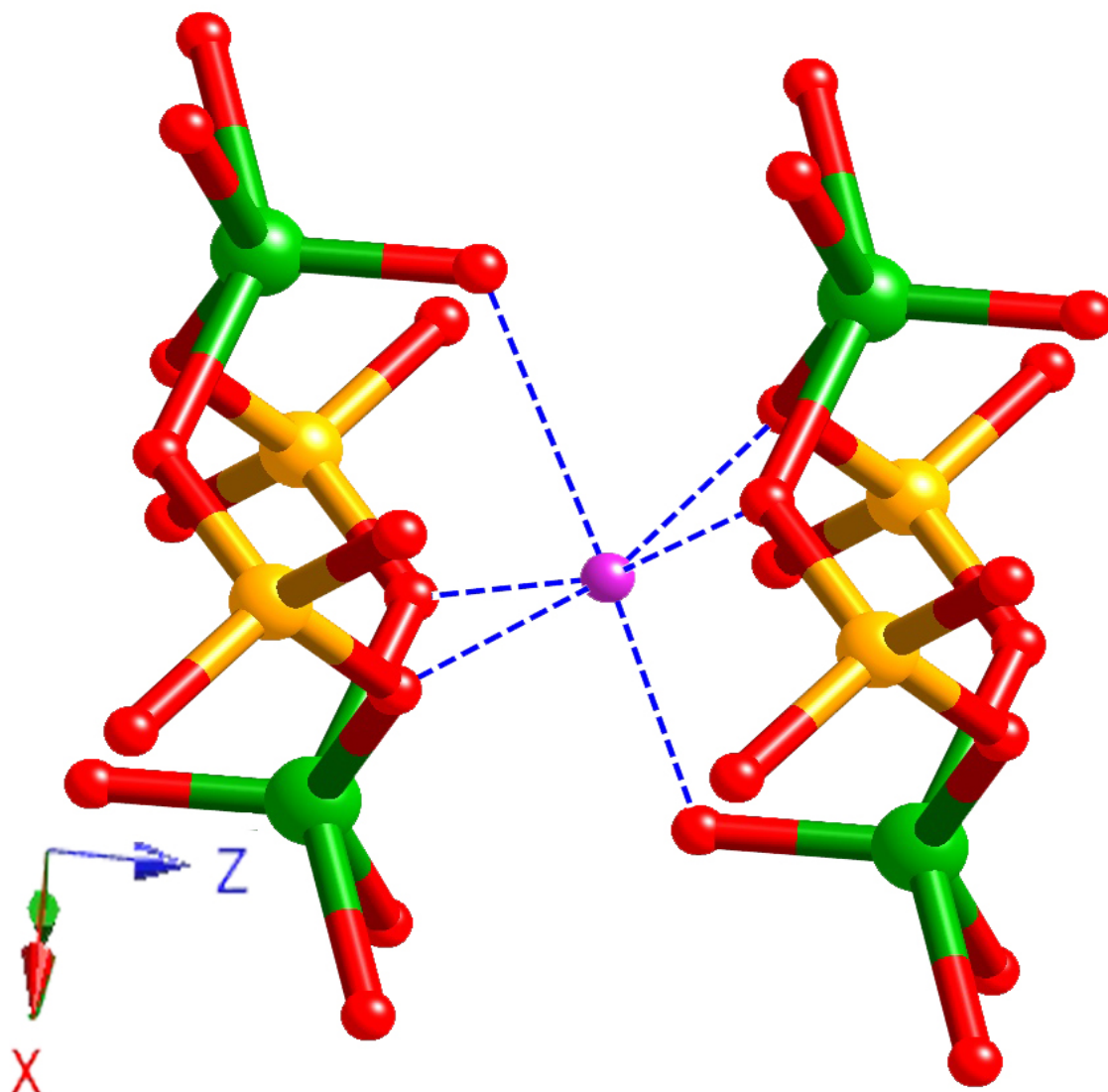


Fig. S5 Crystal structure of α_1 -LiVOPO₄ showing coordination of lithium with oxygen atoms. The V, P, O and Li atoms are shown as green, orange, red and purple colored balls respectively.

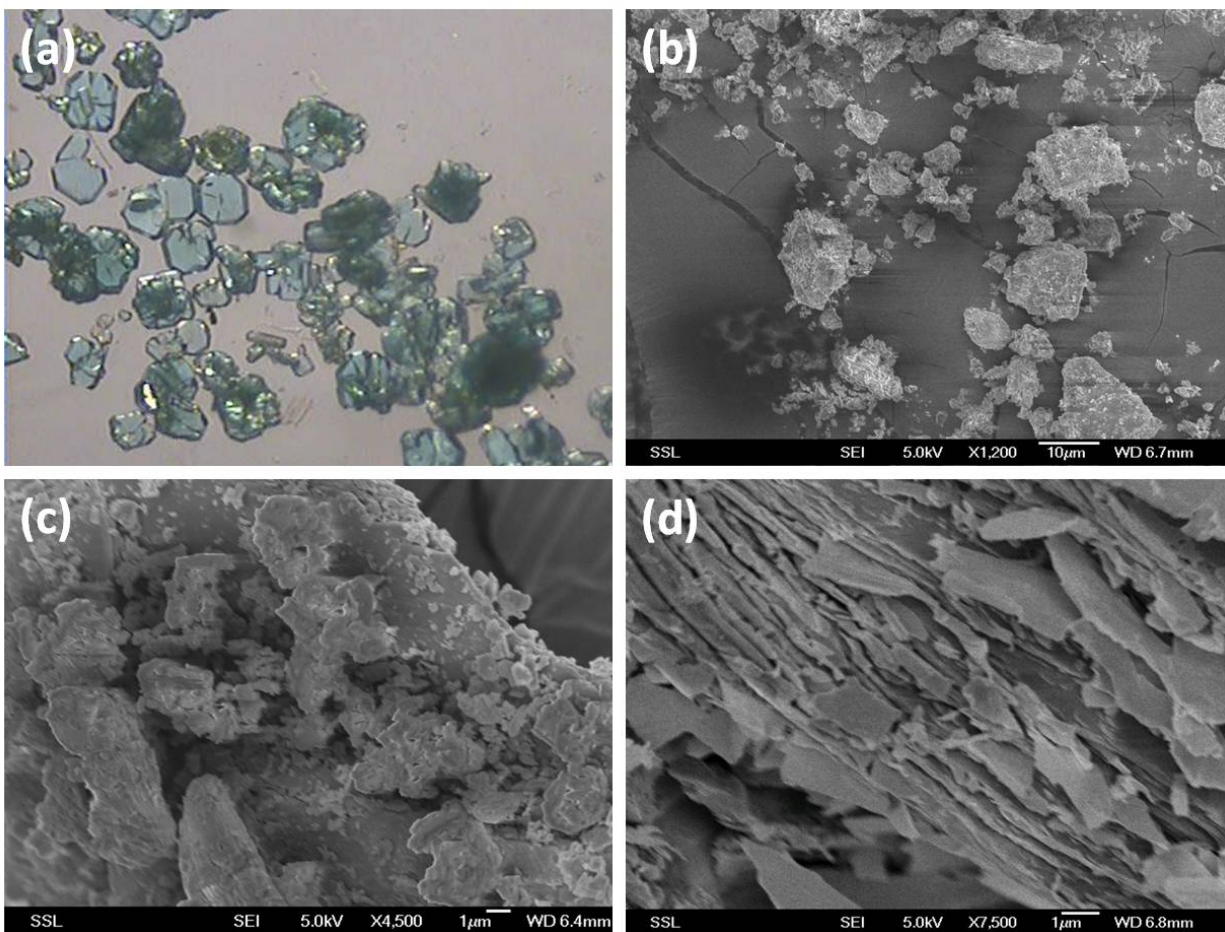


Fig. S6. SEM micrographs of (a) single crystals of $\text{LiVOPO}_4 \cdot 2\text{H}_2\text{O}$, (b) ball-milled $\text{LiVOPO}_4 \cdot 2\text{H}_2\text{O}$, (c) $\alpha_1\text{-LiVOPO}_4$ obtained by dehydration of ball-milled $\text{LiVOPO}_4 \cdot 2\text{H}_2\text{O}$ at low magnification and (d) $\alpha_1\text{-LiVOPO}_4$ at high magnification

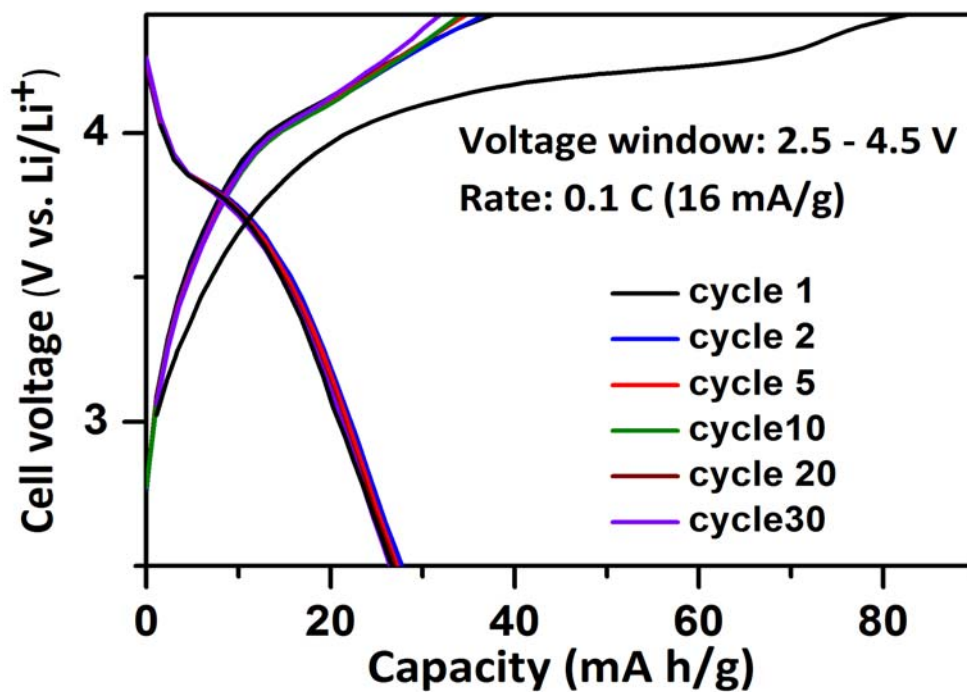


Fig. S7 Galvanostatic charge-discharge cycles of α_I -LiVOPO₄ obtained from as synthesized LiVOPO₄·2H₂O

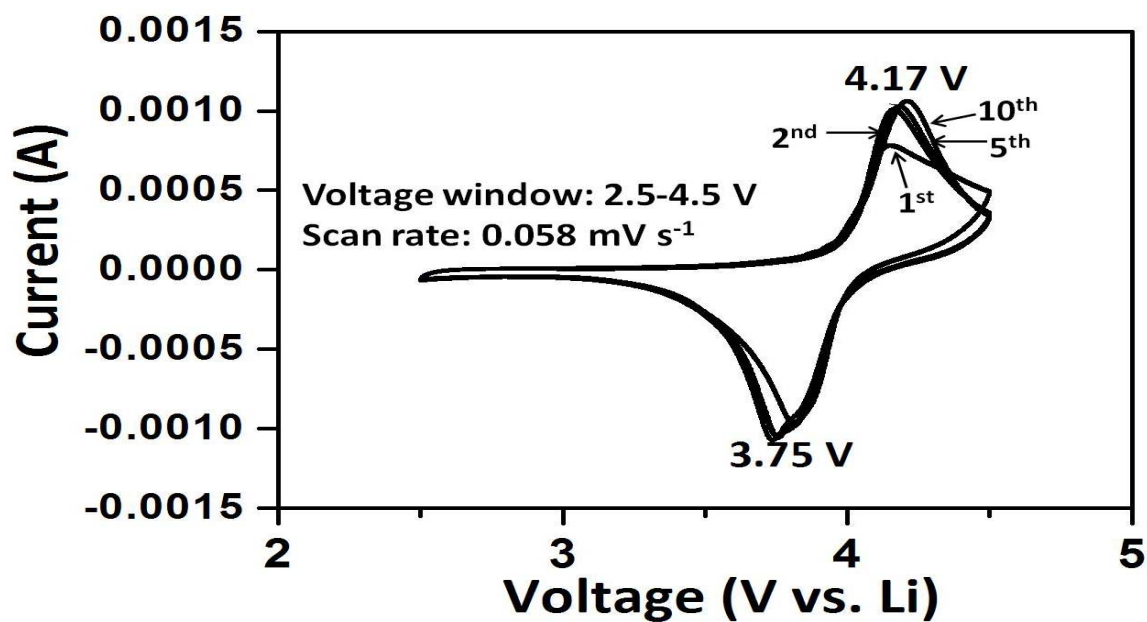


Fig. S8 Cyclic voltammograms of α_I -LiVOPO₄ in the voltage window of 2.5-4.5 V at a scan rate of 0.058 mV s⁻¹

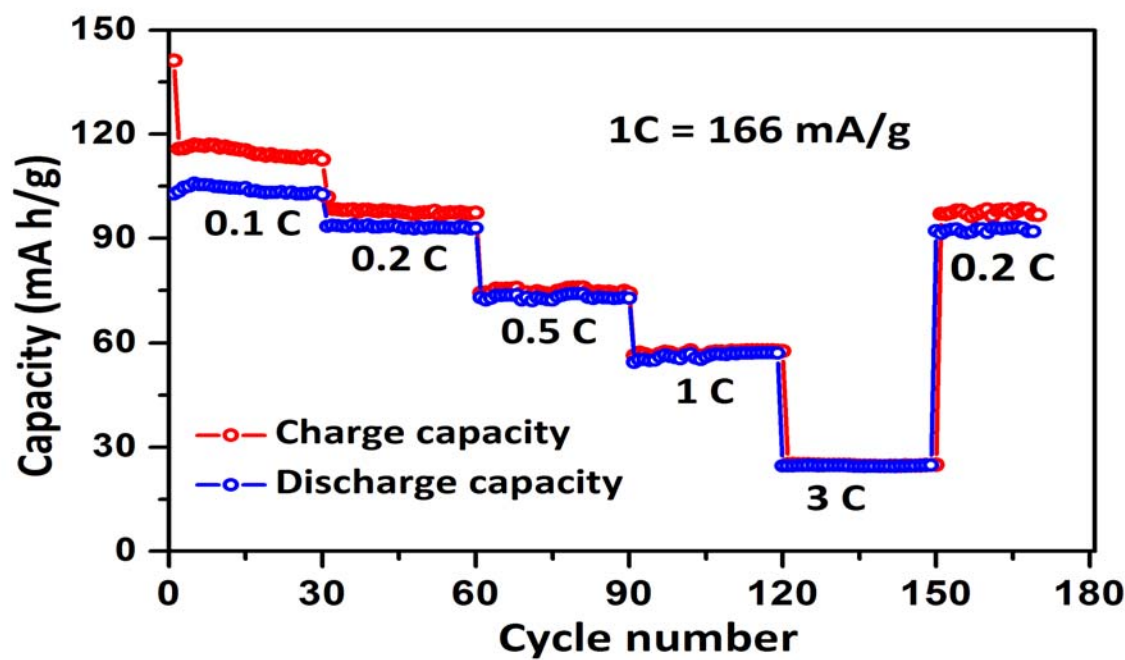


Fig. S9 Rate capability study of α_1 -LiVOPO₄ at various current rates