Electronic Supplementary Information for Dalton Transactions


# Staging during anion-exchange intercalation into $[LiAl_2(OH)_6]Cl{\cdot}yH_2O$: structural and mechanistic insights

Gareth R. Williams, Andrew M. Fogg, Jeremy Sloan, Christine Taviot-Guèho and Dermot O'Hare

**Table S1:** The approximate formulae of the second stage intercalates studied by TEM. Of the phosphonate guests, only the second stage intercalate of EPA could be synthesised with phase purity; in the case of the other phosphonates, mixtures of second stage intercalate with small amounts of host and first stage product were obtained.

| Guest | Formula of intercalate |
|---|---|
| MPA | $[Li_{0.8}Al_2(OH)_6](MePO_3H)_{0.4}Cl_{0.4}{\cdot}3.2H_2O$ |
| EPA | $[Li_{0.86}Al_2(OH)_6](EtPO_3H)_{0.45}Cl_{0.41}{\cdot}2.3H_2O$ |
| BPA | $[Li_{0.82}Al_2(OH)_6](PhCH_2PO_3H)_{0.41}{\cdot}2.7H_2O$ |
| Terephthalate | $[Li_{0.95}Al_2(OH)_6]Cl_{0.49}(C_8H_4O_4)_{0.23}{\cdot}8H_2O$ |

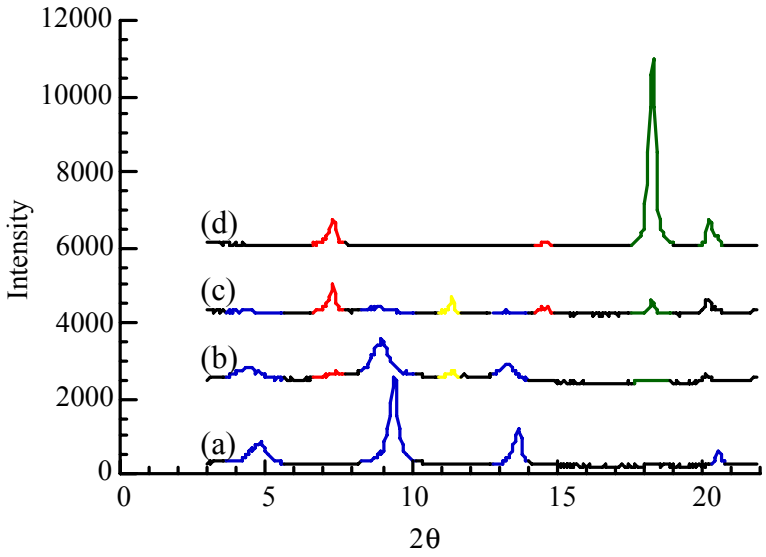



**Figure S1:** XRD patterns showing the temperature dependence of the reaction between $[LiAl_2(OH)_6]Cl.H_2O$ and a stoichiometric amount of disodium succinate for the formation of a second stage intermediate (host:guest ratio 4:1). **(a)** 20˚C, **(b)** 40˚C, **(c)** 60˚C and **(d)** 100˚C. Bragg reflections due to the second stage intermediate are in blue, the first stage product in red, the host lattice in yellow and gibbsite in green.

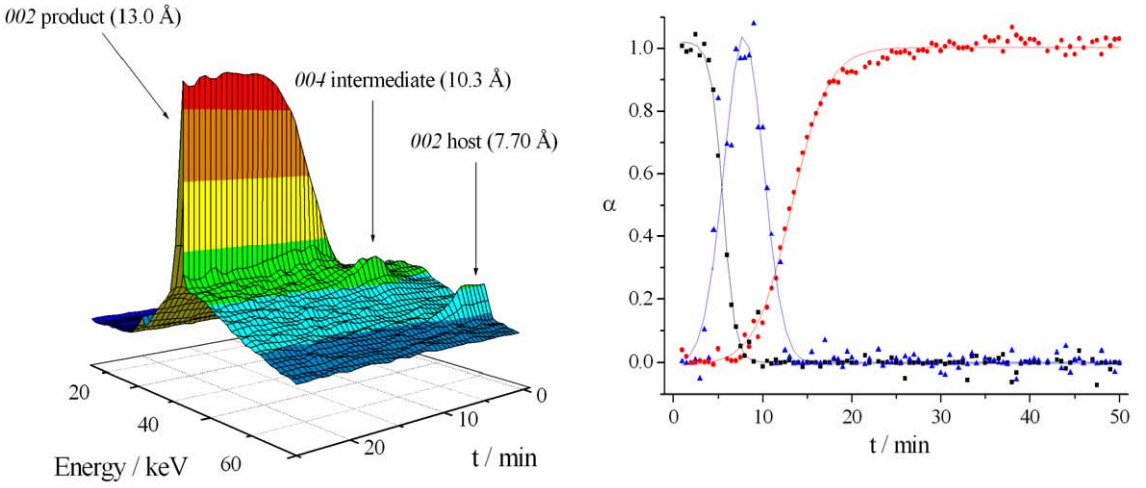



**Figure S2:** *In situ* data for the intercalation of MPA into h-$LiAl_2$-Cl in water at 20 ºC. **(a)** 3D stacked plot showing the formation of a second stage intermediate and **(b)** extent of reaction *vs.* time plot for the host *002* (■), intermediate *004* (▲) and product (●) *002* reflections.

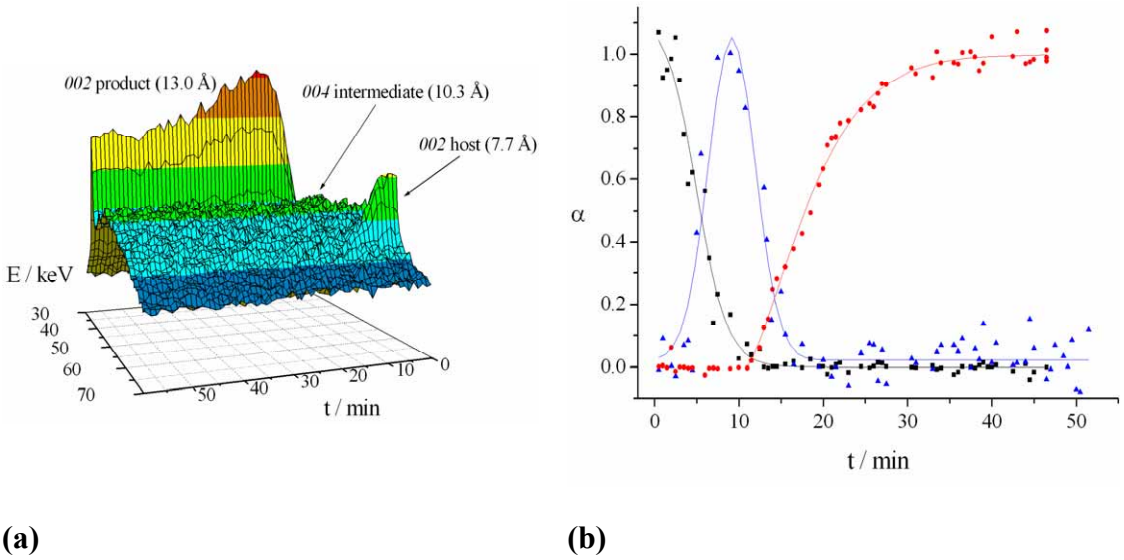
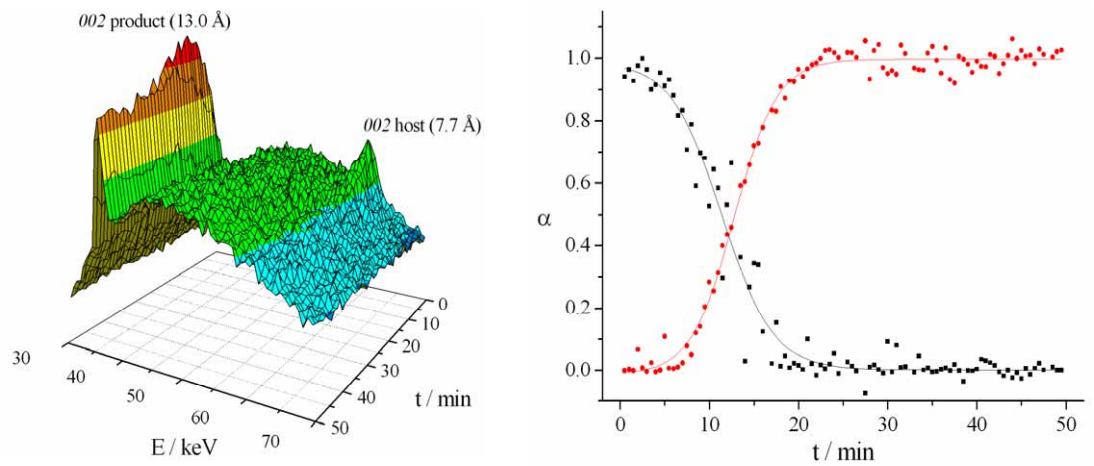



**Figure S3**

*In situ* data for the intercalation of MPA into h-$LiAl_2$-Cl in a 95:5 b.v. mixture of water and acetone. **(a)** 3D stacked plot showing the presence of a second stage intermediate, and **(b)** extent of reaction *vs.* time plot for the host *002* (■), intermediate *004* (▲) and product (●) *002* reflections.




**(a)** **(b)**

**Figure S4**

*In situ* data for the intercalation of MPA into h-$LiAl_2$-Cl a in a 50:50 b.v. mixture of water and acetone. **(a)** 3D stacked plot showing the direct conversion of the host to the product phase and **(b)** extent of reaction *vs.* time plot for the host (■) and product (●) *002* reflections.