## CLAIMS

1. A method of preparing a layered double hydroxide having a specific surface area of at least 125 $m^2/g$ and a BET pore volume ($N_2$) of at least 0.5 cc/g, and having the formula:

$$[M^{z+}_{1-x}M'^{y+}_{x}(OH)_2]^{a+}(X^{n-})_{a/n}\cdot bH_2O\cdot c(\text{AMO-solvent}) \quad (I)$$

wherein M and M′ are different and each is a charged metal cation (and must be present),

$z$ = 1 or 2;

$y$ = 3 or 4,

$0<x<0.9$,

$b$ is 0 to 10,

$c$ = greater than 0 to 10,

X is an anion selected from carbonate, bicarbonate, hydrogenphosphate, dihydrogenphosphate, nitrite, nitrate, sulphate or phosphate or a mixture of two or more thereof,

$n$ is the charge on the anion,

$a = z(1-x)+xy-2$;

AMO-solvent is aqueous miscible organic solvent,

which method comprises

a) precipitating a layered double hydroxide having the formula

$$[M^{z+}_{1-x}M'^{y+}_{x}(OH)_2]^{a+}(X^{n-})_{a/n}\cdot bH_2O$$

wherein M, M', $z$, $y$, $x$, $a$, $b$ and X are as defined above from a solution containing the cations of the metals M and M' and the anion $X^{n-}$;

b) ageing the layered double hydroxide precipitate obtained in step a) in the original solution;

c) collecting the layered double hydroxide precipitate by filtration, then washing the layered double hydroxide precipitate until the washing solution has a pH which is substantially neutral;

d) dispersing the wet layered double hydroxide precipitate in an AMO solvent so as to produce a slurry of the layered double hydroxide in the solvent, wherein the AMO-solvent is an aqueous miscible organic solvent;

e) maintaining the dispersion obtained in step d); and

f) subjecting the dispersion of layered double hydroxide in the AMO-solvent to spray drying under an inert atmosphere.

2. A method according to claim 1, wherein the AMO solvent is an aqueous miscible organic solvent, preferably having a solvent polarity (P) in the range 3.8 to 9.

3. A method according to either claim 1 or claim 2, wherein, in formula (I), when *z* is 2, M is Mg, Zn, Fe, Ca, Sn Ni, Cu, Co, Mn or Cd or a mixture of two or more of these, or when *z* is 1, M is Li.

4. A method according to any one of claims 1 to 3, wherein, in formula (I), when *y* is 3, M′ is Al, Ga, Y, In, Fe, Co, Ni, Mn, Cr, Ti, V, or La, or when *y* is 4, M' is Sn, Ti or Zr or a mixture thereof.

5. A method according to claim 4, wherein M′ is Al.

6. A method according to claim 5, wherein the layered double hydroxide is selected from Zn/Al, Mg/Al, and Ca/Al, Ni/Al, Cu/Al.

7. A method according to claim 6, wherein the layered double hydroxide is an Mg/Al layered double hydroxide.

8. A method according to any one of the preceding claims, wherein in step a) the layered double hydroxide precipitate is formed by introducing metal ($M^{z+} + M'^{y+}$) solution to the anion ($X^{n-}$) solution with a drop rate in the range of 0.1 to 3.5 {mol($M^{z+} + M^{y+}$)}/{mol(anion)*min}.

9. A method according to any one of the preceding claims, wherein in step a) the layered double hydroxide is precipitated from the cation and anion containing solution which either additionally contains NaOH or to which NaOH solution is added separately to adjust pH of solution to a predetermined value.

10. A method according to any one of the preceding claims, wherein in step b) the layered double hydroxide precipitate is aged in the original solution for less than 24 hours, preferably less than 16 hours, more preferably less than 3 hours.

11. A method according to any of the preceding claims, wherein in step c) the washed layered double hydroxide precipitate is then rinsed with AMO-solvent.

12. A method according to any one of the preceding claims, wherein in step d) the wet layered double hydroxide obtained in step c) is dispersed and maintained in AMO-solvent under stirring.

13. A method according to any one of the preceding claims, wherein in step e) the dispersion obtained in step d) is maintained for up to 96 hours, preferably up to 72 hours, more preferably up to 48 hours.

14. A method according to any of the preceding claims, wherein after step e) the layered double hydroxide is collected as wet form and is dispersed in a fresh volume of the AMO solvent and the dispersion is maintained for at least two hours.

15. A method according to any one of the preceding claims, wherein in step f), the dispersion of layered double hydroxide in the AMO-solvent is fed into the spray dryer at a feed rate of at least 12 ml/min.

16. A method according to any one of claims 1 to 15, wherein in step f), the dispersion of layered double hydroxide in the AMO-solvent is fed into the spray dryer at a feed rate of at least 18 ml/min.

17. A spray-dried layered double hydroxide having a specific surface area of at least 125 $m^2/g$ and a BET pore volume ($N_2$) of at least 0.5 cc/g, the layered double hydroxide having the formula:

$$[M^{z+}_{1-x}M'^{y+}_{x}(OH)_2]^{a+}(X^{n-})_{a/n}\cdot bH_2O\cdot c(\text{AMO-solvent}) \quad (I)$$

wherein M and M′ are different and each is a charged metal cation and must be present,

$z$ = 1 or 2;

$y$ = 3 or 4,

$0<x<0.9$,

$b$ is 0 to 10,

$c$ = greater than 0 to 10,

X is an anion selected from carbonate, bicarbonate, hydrogenphosphate, dihydrogenphosphate, nitrite, nitrate, sulphate or phosphate or a mixture of two or more thereof,

$n$ is the charge on the anion, and

$a = z(1-x)+xy-2$;

AMO-solvent is aqueous miscible organic solvent

wherein the layered double hydroxide is obtainable by a method according to any of the preceding claims.

18. A spray-dried layered double hydroxide according to claim 17, wherein the layered double hydroxide has a specific surface area of at least 240 $m^2/g$.

19. A spray-dried layered double hydroxide according to claim 18, wherein the layered double hydroxide has a BET pore volume ($N_2$) of at least 1.0 cc/g.

20. A spray-dried layered double hydroxide according to any one of claims 17 to 19, wherein the layered double hydroxide has a particle size less than 150 µm.

21. A spray-dried layered double hydroxide according to claim 20, wherein the layered double hydroxide has a particle size less than 30 µm.

22. A spray-dried layered double hydroxide according to any one of claims 17 to 19, wherein the layered double hydroxide has an agglomerated particle size less than 100 µm.

23. A spray-dried layered double hydroxide according to claim 22, wherein the layered double hydroxide has an agglomerated particle size less than 30 µm.