CLAIMS

1. A method of making silica-layered double hydroxide microspheres having the formula I

$$(SiO_2)_p@\{[M^{z+}_{(1-x)}M^{y+}_x(OH)_2]^{a+}(X^{n-})_{a/n}\bullet bH_2O\bullet c(AMO\text{-}solvent)\}_q \quad (I)$$

wherein,

M^{z+} and M^{y+} are two different charged metal cations;

$z = 1$ or 2;

$y = 3$ or 4;

$0 < x < 0.9$;

b is 0 to 10;

c has a value that is greater than zero and up to 10;

$p > 0$,

$q > 0$,

X^{n-} is an anion; with $n > 0$

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

the AMO-solvent is an 100% aqueous miscible organic solvent;

which method comprises the steps:

(a) contacting silica microspheres and a metal ion containing solution containing metal ions M^{z+} and M^{y+} in the presence of a base and an anion solution;

(b) collecting the product; and

(c) treating the product with AMO-solvent and recovering the solvent treated material to obtain the silica-layered double hydroxide microspheres of formula (I).

2. The method according to claim 1, wherein M′ is one or more trivalent metal cations.

3. The method according to claim 1 or 2, wherein M is one or more divalent metal cations.

4. The method according to any of the preceding claims, wherein X^{n-} is selected from CO_3^{2-}, OH^-, F^-, Cl^-, Br^-, I^-, SO_4^{2-}, NO_3^- and PO_4^{3-}, preferably from CO_3^{2-}, Cl^- and NO_3^-, or a mixture of two or more thereof.

5. The method according to any of the preceding claims, wherein M is Mg, M is Al and X^{n-} is CO_3^{2-}.

6. The method according to any of the preceding claims, wherein the pH of the metal ion containing solution in step (a) is within a range from 8 to 12.

7. The method according to claim 6, wherein the pH is of from 9 to 10.

8. The method according to any one of the preceding claims, wherein the product collected in step (b) is dispersed in the AMO-solvent and wherein c in the formula I has a value which is greater than zero and up to 10.

9. The method according to claim 8, wherein the dispersion of the product in the AMO-solvent is maintained for 2 or more hours and then the treated material is separated from the solvent.

10. The method according to any of the preceding claims, wherein the AMO-solvent is acetone, ethanol or methanol or a mixture thereof, preferably acetone or ethanol.

11. The method according to any of the preceding claims, wherein the temperature of the metal ion containing solution in step (a) is within a range of from 20 to 150°C.

12. The method according to claim 11, wherein the temperature is from 20 to 80°C.

13. The method as claimed in any one of the preceding claims, wherein the silica-layered double hydroxide microspheres have specific surface area as determined by N_2 BET of at least 177 m^2/g, preferably at least 200 m^2/g.

14. The method as claimed in any of the preceding claims, wherein the silica-layered double hydroxide microspheres have at least one structure from hollow-shell, yolk-shell and solid core-shell structures.

15. The method as claimed in any of the preceding claims, wherein the silica-layered double hydroxide microspheres have a thickness of layered double hydroxide layer larger than 65 nm, preferably larger than 110 nm, more preferably larger than 160 nm.

16. Silica-layered double hydroxide microspheres having the formula I below

$$(SiO_2)_p@\{[M^{z+}_{(1-x)}M^{y+}_x(OH)_2]^{a+}(X^{n-})_{a/n}\bullet bH_2O\bullet c(AMO\text{-}solvent)\}_q \quad (I)$$

wherein,

M^{z+} and M^{y+} are two different charged metal cations;

$z = 1$ or 2;

$y = 3$ or 4;

$0 < x < 0.9$;

b is 0 to 10;

c is 0 to 10;

$p > 0$,

$q > 0$;

X^{n-} is an anion; with $n > 0$;

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

AMO-solvent is an 100% aqueous miscible organic solvent,

wherein the silica-layered double hydroxide microspheres each comprise a SiO_2 microsphere having solid layered double hydroxide attached to its surface, and wherein:

i. the silica-layered double hydroxide microspheres are core-shell materials wherein the SiO_2 microsphere is a solid sphere, and wherein the specific surface area of the core-shell materials is at least 100 m^2/g, or

ii. the silica-layered double hydroxide microspheres are yolk-shell materials wherein the SiO_2 microsphere comprises an outer shell and a smaller SiO_2 sphere contained within the outer shell, wherein there is a hollow portion between the smaller sphere and the inner surface of the

outer shell, and wherein the specific surface area of the yolk-shell materials is at least 100 m^2/g, or

iii. the silica-layered double hydroxide microspheres are hollow shell materials wherein the SiO_2 microsphere has a hollow interior, and wherein the hollow shell materials have a specific surface area of at least 130 m^2/g.

17. Silica-layered double hydroxide microspheres according to claim 16, wherein M' is one or more trivalent cations.

18. Silica-layered double hydroxide microspheres according to claim 16 or 17, wherein M is one or more divalent cations.

19. Silica-layered double hydroxide microspheres according to any one of claims 16 to 18, wherein X^{n-} is selected from CO_3^{2-}, OH^-, F^-, Cl^-, Br^-, I^-, SO_4^{2-}, NO_3^- and PO_4^{3-}, preferably from CO_3^{2-}, Cl^- and NO_3^-, or mixtures thereof.

20. Silica-layered double hydroxide microspheres according to any one of the claims 16 to 19, wherein M is Mg, M' is Al and X^{n-} is CO_3^{2-}.

21. Silica-layered double hydroxide microspheres according to any one of claims 16 to 20, wherein the AMO- solvent is acetone, ethanol, methanol or mixtures thereof, preferably acetone or ethanol.

22. Silica-layered double hydroxide microspheres according to claim 16, wherein

M^{z+} is a divalent metal cation,

M'^{y+} is a trivalent metal cation, and

each of b and c is a number > zero.