

Electronic Supplementary Information - ESI

Tuning nano-phase separation and drug delivery kinetics through spray-drying and self-assembly

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ESI 1. Supplementary characterisation data

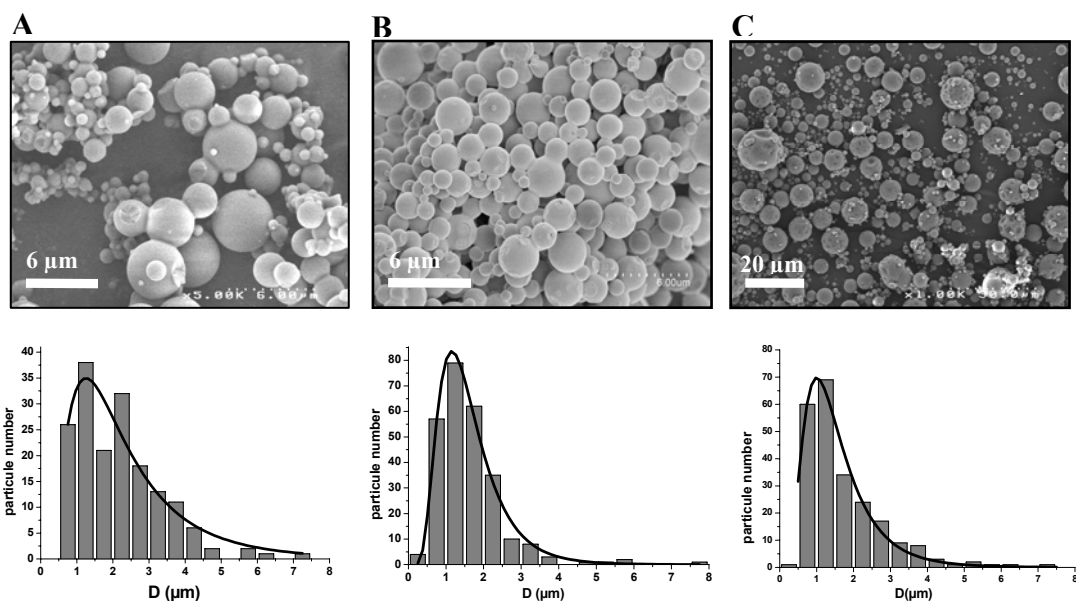
| <i>Sample</i> | A | B | C |
|---|----------|------------------------|----------|
| <i>Synthesis</i> | | | |
| ibuprofen (mg/g SiO ₂) ^a | 156 | 286 | 156 |
| $r_{ibu/tw}$ | 2.0 | 5.0 | ∞ |
| organic volume fraction ϕ_{org} | 0.55 | 0.55 | 0.25 |
| <i>Morphology</i> | | | |
| D_{GM} (μm) | 2.0 | 1.5 | 1.4 |
| <i>Release properties</i> | | | |
| Q_0 (%) | 60 | 59 | 48 |
| b | 0.85 | 1.50 | 2.60 |
| t_{lag} (min) | 0.8 | 2.2 | 8.5 |
| t_{scale} (min) | 8 | 118 | 6483 |
| r^2 | 0.999 | 0.998 | 0.998 |
| <i>Texture</i> | | | |
| d_{corr} (nm) from SAXS | 7.0 | 9.5 | - |
| d_{corr} (nm) from TEM | 6.8 | 9.0 | - |
| spheroid domains (nm) ^b | - | 20-100 | 20-100 |
| ¹³C NMR -COOH peak | | | |
| | Broad | Broad, 78% / Thin, 22% | Thin |
| δ_{iso} (ppm) | 178.9 | 180.2 / 181.3 | 181.4 |
| $\Delta\nu_{1/2}$ (Hz) | 158 | 157 / 24 | 32 |

a: amount of ibuprofen introduced in the sol over the amount of SiO₂ formed after condensation.

b: estimated by TEM, see ESI 4.

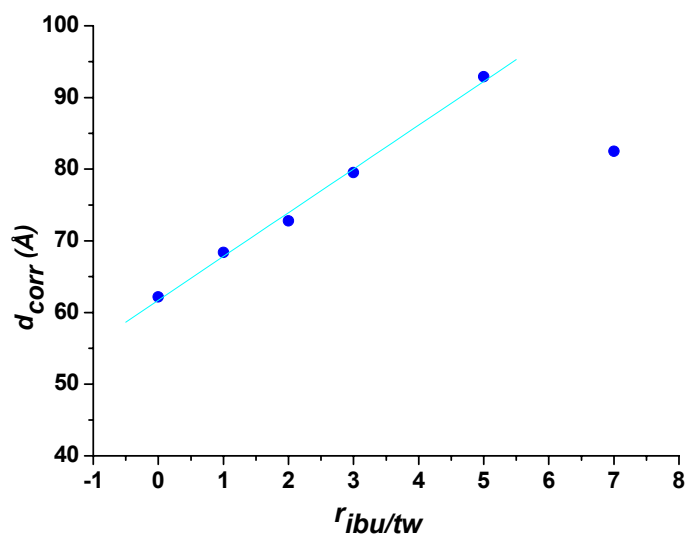
ESI 2. SEM observations.

Examples of morphology (SEM micrographs and statistical analyses) observed for three samples **A**, **B**, and **C**. Size distributions were obtained by counting the number of spheres (well-separated or slightly agglomerated), and were fitted using a Log-Normal distribution.



ESI 3. Variation of d_{corr} as a function of $r_{ibu/tw}$.

Variation of the correlation distance d_{corr} obtained from SAXS data (0.2 - 2 nm⁻¹ q range) as a function of the molar ratio ibuprofen/tween $r_{ibu/tw}$.



ESI 4. TEM analysis.

Example of a statistical analyses of sizes of domains at the core of the microspheres for sample **B** and **C**. (a) TEM micrograph of samples **B**. (b) Related size distributions obtained by counting the number of visible clear spheroids from one side of the particle to the other side.

