

# Supplementary Materials: Solvent-Free Fabrication of Biphasic Lipid-Based Microparticles with Tunable Structure

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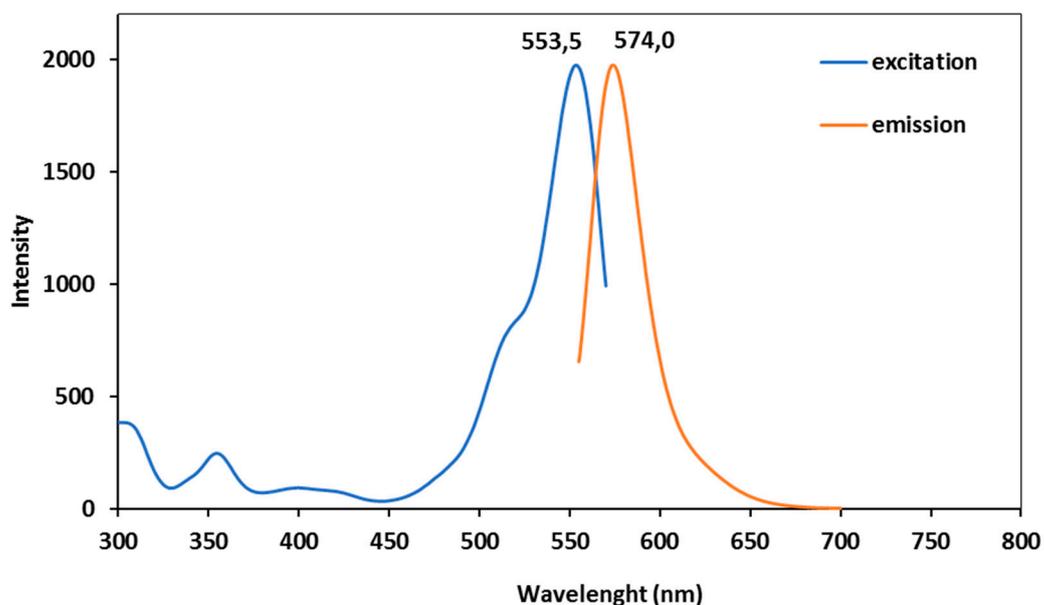


Figure S1. Excitation and emission fluorescence spectra of Rho.

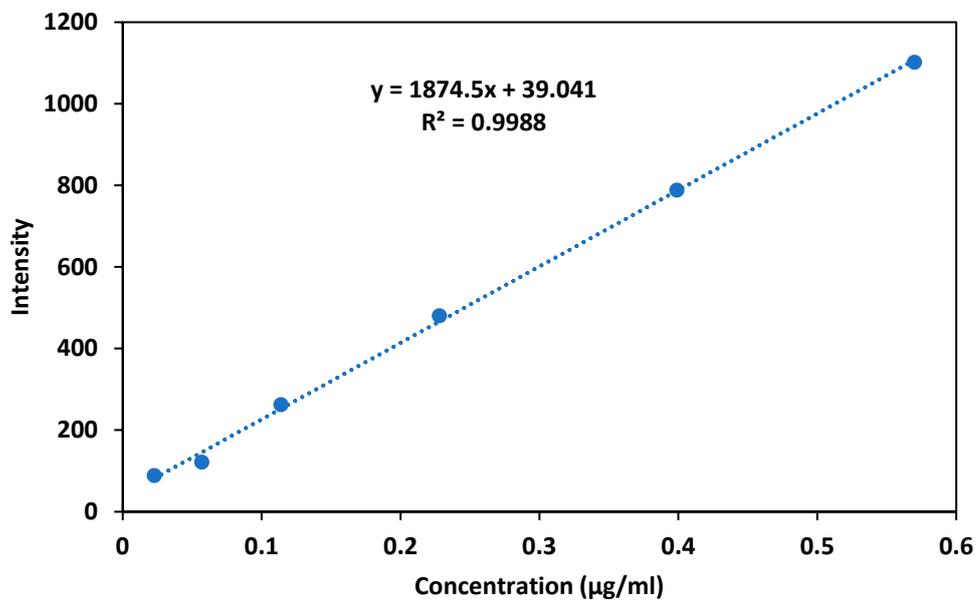
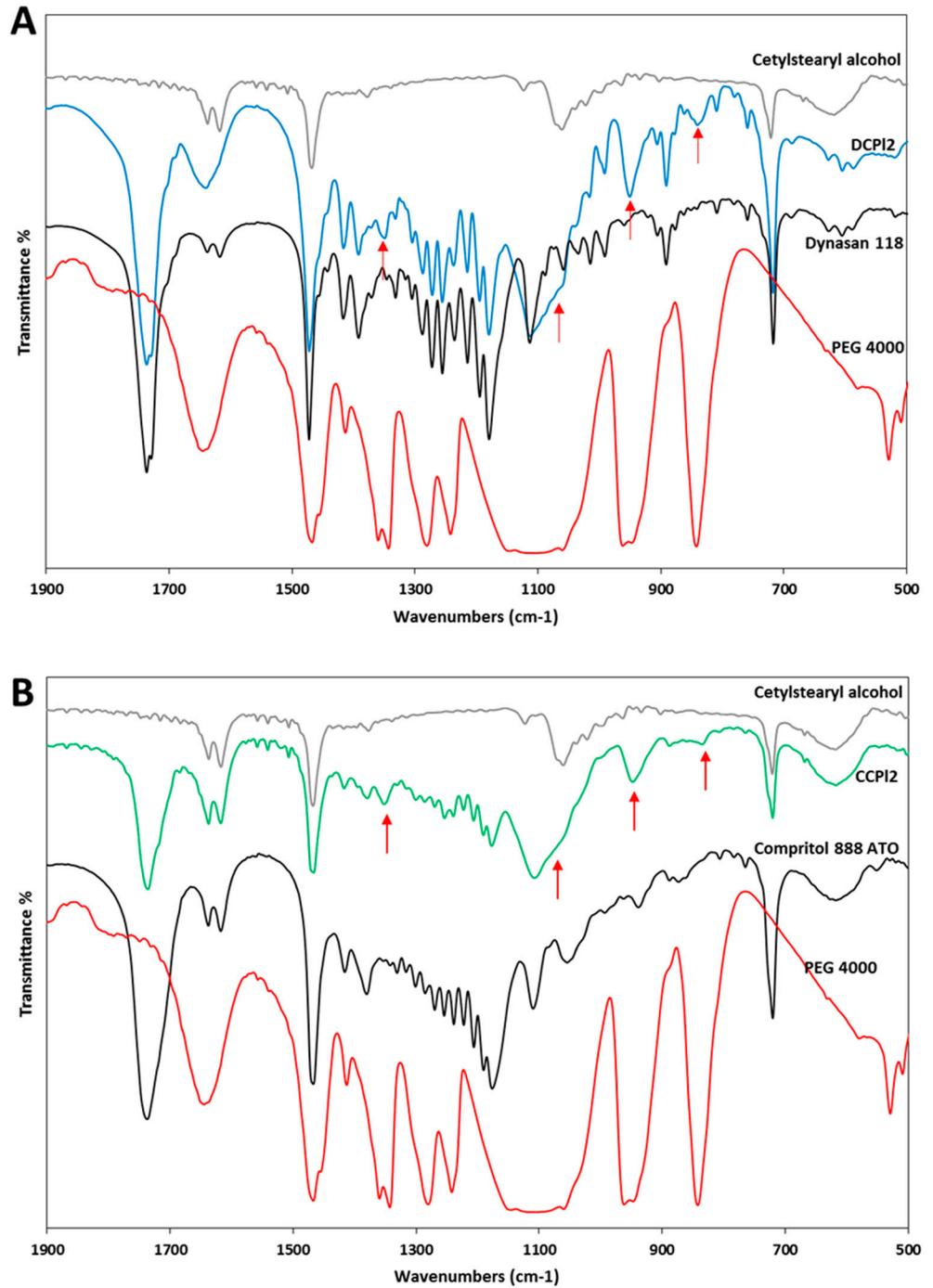
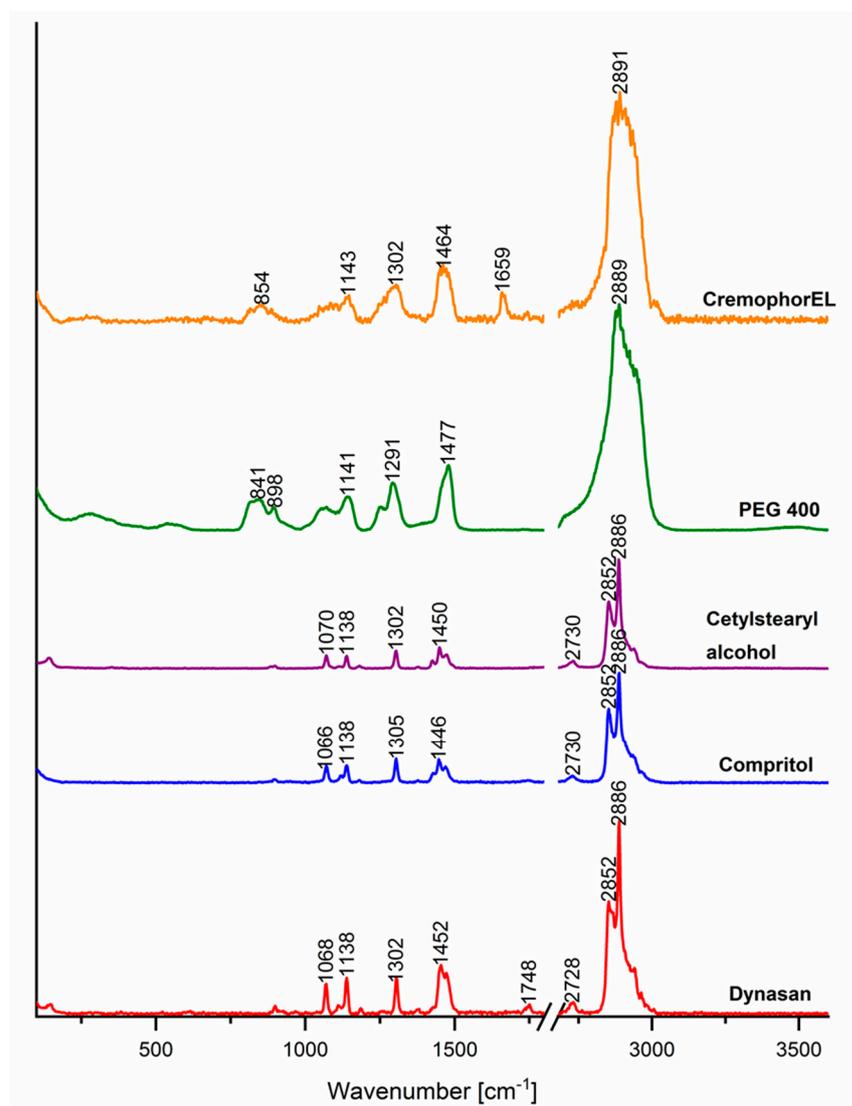


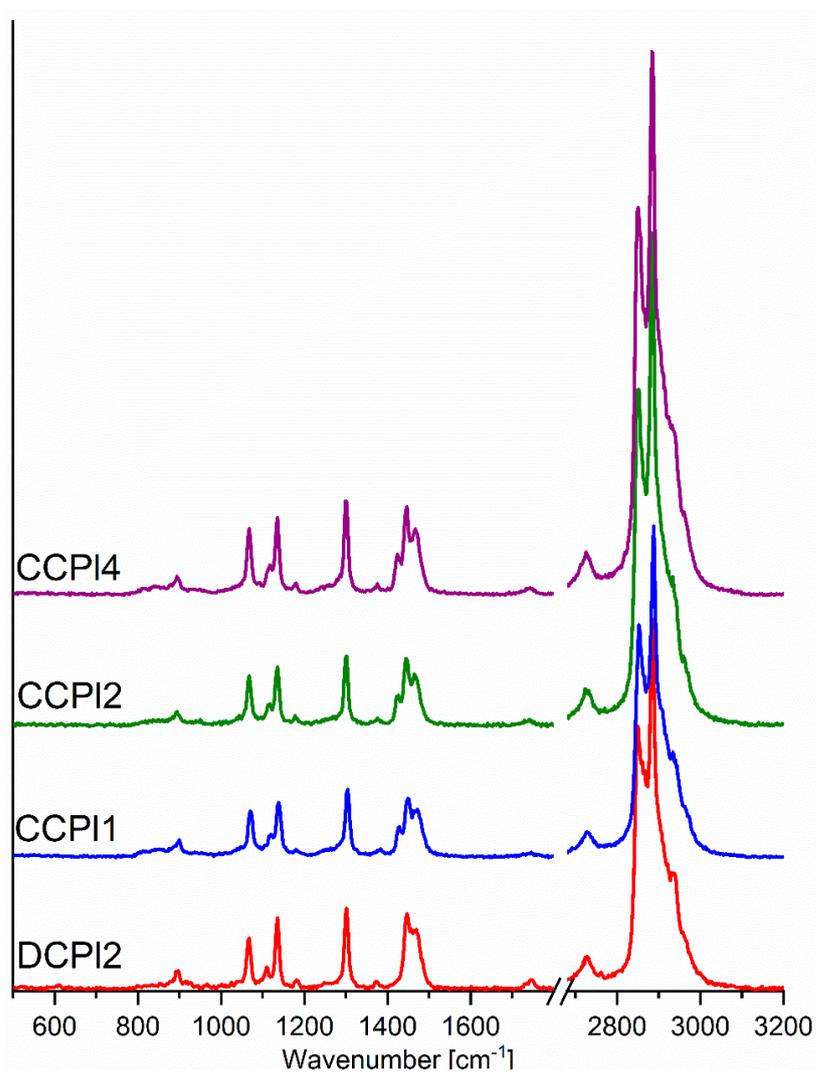
Figure S2. Calibration curve of Rho using fluorescence measurements.



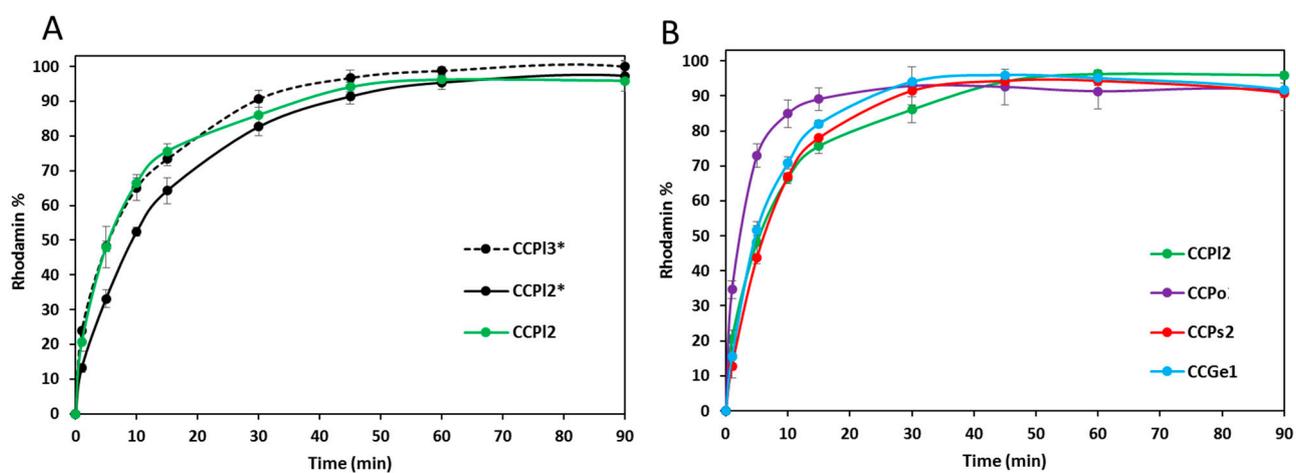
**Figure S3.** FT-IR spectra of DCPI2 b-MPs compared to the respective solid raw excipients (A) and CCPI2 b-MPs compared to the respective solid raw excipients (B). Red arrows indicate the bands in the b-MPs spectra attributed to the hydrophilic phase.



**Figure S4.** Raman spectra recorded for raw materials: Dynasan®118 (red line), Compritol® 888 ATO (blue line), cetylstearyl alcohol (purple line), PEG 400 (green line) and Cremophor®EL (orange line).



**Figure S5.** Raman spectra recorded for b-MPs: DCPI2 (red line), CCPI1 (blue line), CCPI2 (green line), CCPI4 (purple line).



**Figure S6.** Effect of the lipid:surfactant ratio (A); and of various hydrophilic phases (B) on drug release from Compritrol-based biphasic MPs (250-355  $\mu\text{m}$ ).