

Supporting Information

Passive optical wave guiding tubular pharmaceutical solids and Raman spectroscopy/mapping of nano/micro scale defects

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Experimental Sections:

Materials: The materials caffeine (CF), carbamazepine (CBZ) and glibenclamide (GB) were purchased from Sigma-Aldrich, India and directly used for self-assembly studies without further purification. The spectroscopic grade solvents of tetrahydrofuran (THF), methanol (MEOH) were used for preparation of pharmaceutical 1D- self-assembled nano/micro tubular solids under ambient conditions.

Instrumental methods:

Solid state absorbance studies: The solid state absorbance spectra were collected from a Shimadzu UV-3600 spectrometer in a diffuse reflectance UV-visible (DR-UV-vis) mode. The reflectance spectra were converted to an absorbance spectra using Kubelka-Munk function.

Electron microscopy studies: Size and morphology of the organic tubes were examined by using a Carl-Zeiss (Model Ultra55) field emission scanning electron microscope (FESEM) operating at 5 kV. Transmission Electron Microscope (TEM) measurements were performed on a Tecnai G² FEI F12 instrument operating at an accelerating voltage of 200 kV. Carbon coated TEM grids (200 Mesh Type-B) were purchased from Ted Pella Inc. USA.

Atomic Force Microscopy (AFM) studies: Atomic force microscopy studies was carried out on NT-MDT Model Solver Pro M microscope using a class 2R laser of 650 nm wavelength having maximum output of 1 mW. All calculations and image processing were carried out by using NOVA 1.0.26.1443 software provided by the manufacturer. The images were recorded in a semi-contact mode using a noncontact super sharp silicon cantilever (NSG 10_DLC) with a diamond like carbon tip (NT-MDT, Moscow). The dimension of the tip is as follows: Cantilever length = 100 (± 5) μm , Cantilever width 35 (± 5) μm , and Cantilever thickness = 1.7-2.3 μm , Resonate frequency = 190-325 kHz, Force constant = 5.5-22.5 N/m, Chip size = 3.6 \times 1.6 \times 0.4 mm, Reflective side = Au, Tip height = 10-20 μm and DLC Tip curvature radius = 1-3 nm.

Passive optical wave guiding studies: The passive optical wave guiding experiments were carried out on a transmission mode laser confocal optical microscope (T-LCOM) facility of the WiTec alpha 200 SNOM instrument. The Nd:YAG laser operating at 532 nm (maximum output power is 40 mW) light source was point illuminated at one of the open ends of the tube, and the bright and dark field images were captured by using a color eyepiece video camera. To determine the defect sites *via* Raman scattered photons a T-LCOM setup was used (see Chart S1). It consists of a bottom-fix illumination stage, in which the illuminating source (laser) was fixed at the bottom of the piezo scanner and the laser light was point focused (~ 5 μm spot size) on to a particular feature of the sample by using a 5 \times objective. The top 100 \times objective lens was used for the collection of inelastic scattered (Raman) photons and the collected inelastic signal was sent to a CCD detector through a multimode optical fiber of diameter 100/140 μm (core/cladding). The inelastic Raman spectrum/imaging was recorded using a 532 nm long pass edge filter (LPEF). For the image scans in Figures 3 and 4, we commonly employed 240 \times 240 data points with a 0.5 s integration time over the selected scan area of the tube. After completion of the scan, the average spectrum was obtained from the image scan data profile. Finally, a particular Raman peak of the average spectrum was selected and used to generate three dimensional (3D) image using a Witec 2.08 software. This process involved baseline correction, background subtraction, and line integration of the selected peak. All measurements were carried out under ambient conditions.

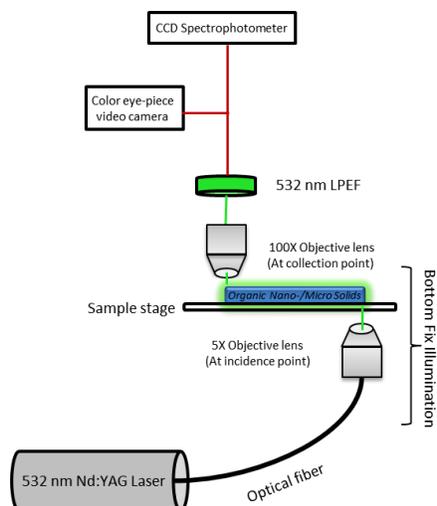


Chart S1: Transmission mode laser confocal Raman microscope (T-LCRM) setup.

Fabrication of self-assembled nano/micro pharmaceutical tubular solids:

Caffeine (CF) tube preparation: 1 mg of CF was taken in a test tube and dissolved in 2 mL of tetrahydrofuran (THF), and then 1 mL of H₂O (Millipore Milli-Q, resistivity = 18 MΩ cm) rapidly injected into above solution and left it for 20 min to form micro tubes.

Carbamazepine (CBZ) tube preparation: 1 mg of CBZ was taken in a test tube and dissolved in 2 mL of methanol (MeOH) and leave it for 5 min to form micro tubes.

Glibenclamide (GM) tube preparation: 1 mg of GM was taken in a test tube and dissolved in 2 ml of methanol (MeOH) and leave it for 5 min to form micro tubes.

For sample preparation, one drop of the solution (CF or CBZ or GM) was drop-casted on a clean glass slide by a capillary/ μ -syringe, air dried and the formation of micro-tubes of CF observed through SEM, AFM and TEM studies.