Summary of data supporting "Chiral Self-Assembly of Cellulose Nanocrystals is Driven by Crystallite Bundles"

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General comments

This summary is organised by measurement type. Below each sub-heading is a reference to Figures and Supplementary Figures where the data have been used.

In file and folder names, the label "main series" refers to the data obtained from the core set of sonicated CNC suspensions at varying sonication doses for which photonic films were made (see e.g. Figure 1 of the main text).

Some "main series" data files are named according to the duration of sonication in seconds, rather than the sonication dose in J/mL used in the published work. The conversion between these two quantities (which, coincidentally, are almost identical) is given in the table below. In a few samples an older labelling convention is used, which is also indicated in the table below.

Sonication duration (s)	Sonication dose (J/mL)	Older labelling
0	0	TS0000
3	3	TS0011
12	12	TS0045
48	48	TS0180
192	193	TS0720
768	772	TS2880
1536	1544	N/A
3072	3087	TS11520
12288	12349	N/A

AFM Atomic force microscopy

Supplementary Table 8, Supplementary Figures 12-14. Data implicitly used elsewhere based on particle thickness estimate in Supplementary Figure 14.

Original image files and data extracted using Gywddion software are provided (see table below for definition of column names). Note that isolated objects on the AFM grid are referred to as "grains" (synonymous with particles).

Column name	Definition
x_c, y_c	X-coordinate of the centre of a grain. In metres.
z_max	Y-coordinate of the centre of a grain. In metres.
z_m	Mean z-coordinate (i.e. height of a grain). In metres.
z_med	Median z-coordinate. In metres.
z_rms	Root mean square z-coordinate. In metres.
A_px	Area of grain in pixels squared.
A_s	Surface area of grain in metres squared.

A_h	Area at half max height in metres squared.
A_c	Area of the convex hull of the grain in metres squared.
D_min	Minimum caliper size (Feret width) in metres.
D_max	Maximum caliper size (Feret length) in metres.

Capillaries

Figure 3, Supplementary Figure 16.

The sub-folder “phase main series” contains photographs of backlit capillaries with unpolarised illumination and with illumination under crossed polarised, with photos labelled L(ight) and D(ark) respectively. The sub-folder also contains the phase data (i.e. the relative amount of anisotropic phase for biphasic samples, and phase information used to construct the phase map in Figure 3), which was extracted manually using ImageJ.

The sub-folder “pitch main series” contains optical microscopy images of capillaries, as well as pitch values manually obtained from those images using ImageJ. Images of a standard microscope scale bar at different magnifications is also provided for scaling the images. Note that for accuracy, the pitch was measured as half the distance of two pitch repeats, so measured values are twice the pitch value.

The sub-folder “pitch mixes” contains optical microscopy images of capillaries contains mixtures of the 772 J/mL and 3087 J/mL samples, as well as pitch values manually obtained from those images using ImageJ. Images of a standard microscope scale bar at different magnifications is also provided for scaling the images. Note that for accuracy, the pitch was measured as half the distance of two pitch repeats, so measured values are twice the pitch value. In the file name labelling, the value after “mix” indicates the relative amount of the low-dose suspension, so “mix_00” is fully 3087 J/mL suspension and “mix_100” is fully 772 J/mL.

Conductivity and pH

Supplementary Tables 1-2, Supplementary Figure 3.

A single data file (data_cond_pH_200704.csv) contains the (1) electrolytic conductivity and (2) pH values obtained for the main series samples before and after sonication, as well as (3) conductivity and (4) mass fraction values for the main series after dialysis.

Cryogenic transmission electron microscopy (cryoTEM)

Supplementary Figure 7.

The original raw images files are provided.

Dynamic light scattering (DLS) and zeta potential

Supplementary Figure 2, Supplementary Figure 15, Supplementary Table 3

For DLS measurements, two datasets are provided: one for the “main series” of samples, and one for the samples used for calibration off the sonication dose (see Supplementary Figure 2). For each dataset, the “raw” data (z-average size and PDI width) are provided for each measurement run, while the “averaged” data contains the mean values.

The folder also contains zeta potential measurements for the “main series” samples.

Photonic films

Figure 1B, Supplementary Figure 6.

The sub-folder “images_POM” contains polarised optical microscopy (POM) images of photonic films made from the “main series”, including (1) crossed-polarisers (XP) images of the films; (2) images from regions viewed under left circular-polarised (LCP) and right circular-polarised (RCP) light, which were the regions used for micro-spectroscopy; (3) reference images of a scalebar, white balance of the diffusive material and an image of the collection spot size for micro-spectroscopy.

The sub-folder “data_POM_spectra” contains the LCP and RCP optical spectroscopy data obtained for each photonic film, as well as spectra for a mirror as reference.

Scanning electron microscopy (SEM)

Figure 1C, Supplementary Figures 5-6.

The sub-folder “images_SEM” contains TIF files of the cross-sections of the photonic films from the “main series” of suspensions. The folder also includes pitch values obtained by manually measuring the layer spacing from SEM images.

Sonication calibration

Supplementary Figure 2. Implicitly used throughout as sonication dose unit.

The folder contains dynamic light scattering data for sonicated 0.1 wt% CNC suspensions, and calorimetry data used to convert duration of sonication into energy per suspension volume and other dose units. Note that some data is repeated from the DLS folder for completeness.

Transmission electron microscopy (TEM)

Images shown: Figure 1D-E, Figure 2A. Analysis of TEM data used extensively throughout.

The folder contains TIF stacks for each sonication dose in the “main series” of samples. Each stack contains (1) the original image file (2) a contrast-enhanced image used for outline tracing (3) the same as 2 with the manually traced outlines overlaid. There is a sub-folder of the raw morphological data collected from each image stack, and a sub-folder of collated morphological data for each sonication dose.

Titration

Supplementary Figure 4, Supplementary Table 3

The folder contains raw conductivity data from the auto-titrator used to measure the surface charge per CNC dry mass.

UV-vis spectroscopy (Turbidity)

Supplementary Figure 15, Supplementary Figure 21.

The folder contains raw spectral data from the transmission optical spectrometer, and the mean particle cross-section values obtained by fitting the spectra as described in the Supplementary Information.