

Supplementary Information:

TEMPO-oxidized cellulose nanofibril/polyvalent cations hydrogels: A multifaceted view of network interactions and inner structure

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S1 – X-Ray diffraction methods

X-Ray diffractogram patterns were measured in the 2θ range $5^\circ - 30^\circ$ (step size 0.02° , counting time 2 s per step, room temperature) by means of D2 Phaser (Bruker) Benchtop X-ray powder diffractometer equipped with a Bragg-Brentano reflection geometry (Cu-K α 1 radiation; $\lambda = 0.154056$ nm. The crystallinity index (CI) was determined applying the Segal approach as shown in Equation 1:

$$CI = \frac{(I_{200} - I_{am})}{I_{200}} \times 100 \quad (1)$$

Where CI is crystallinity index (%), I_{200} the maximum intensity of the (200) reflection ($2\theta = 22^\circ - 23^\circ$), and I_{am} is the lowest intensity of diffraction for amorphous part ($2\theta = 18^\circ - 19^\circ$).¹

Fig. S1 – X-Ray diffractogram of TOCNFs dispersion.

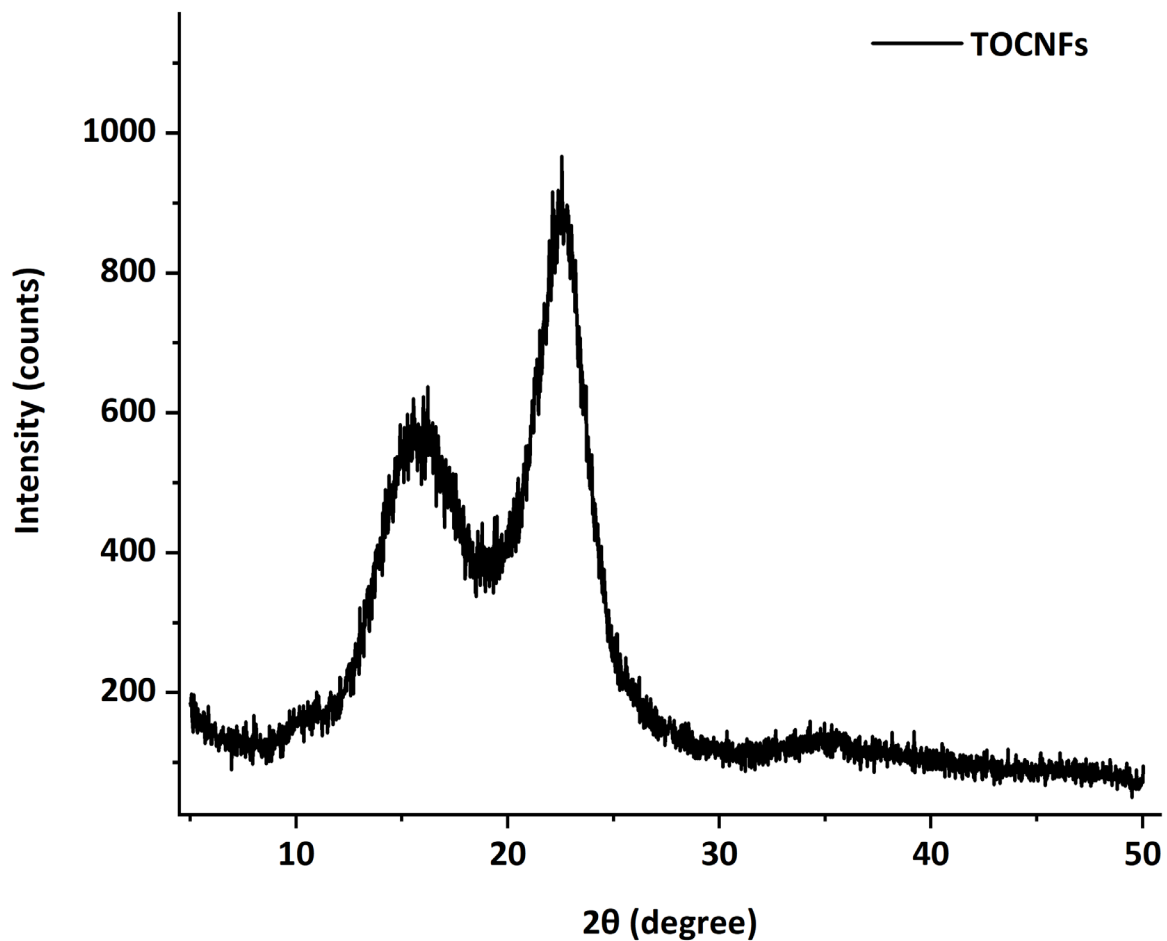


Fig. S2 – TEM and lateral size distribution.

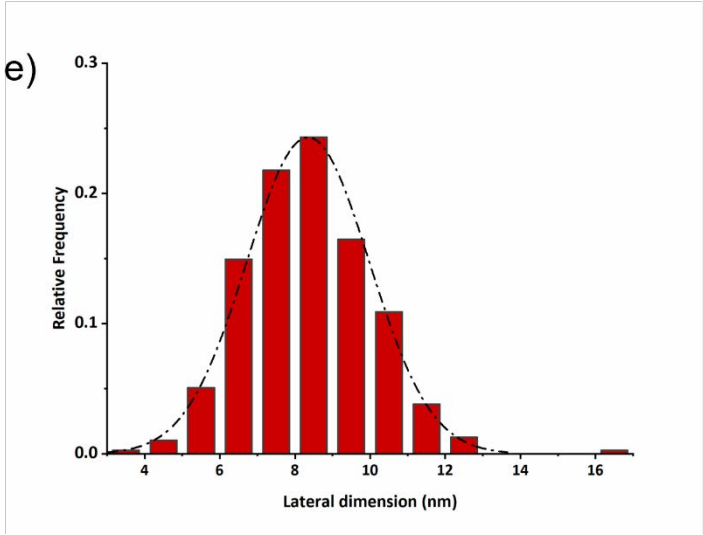
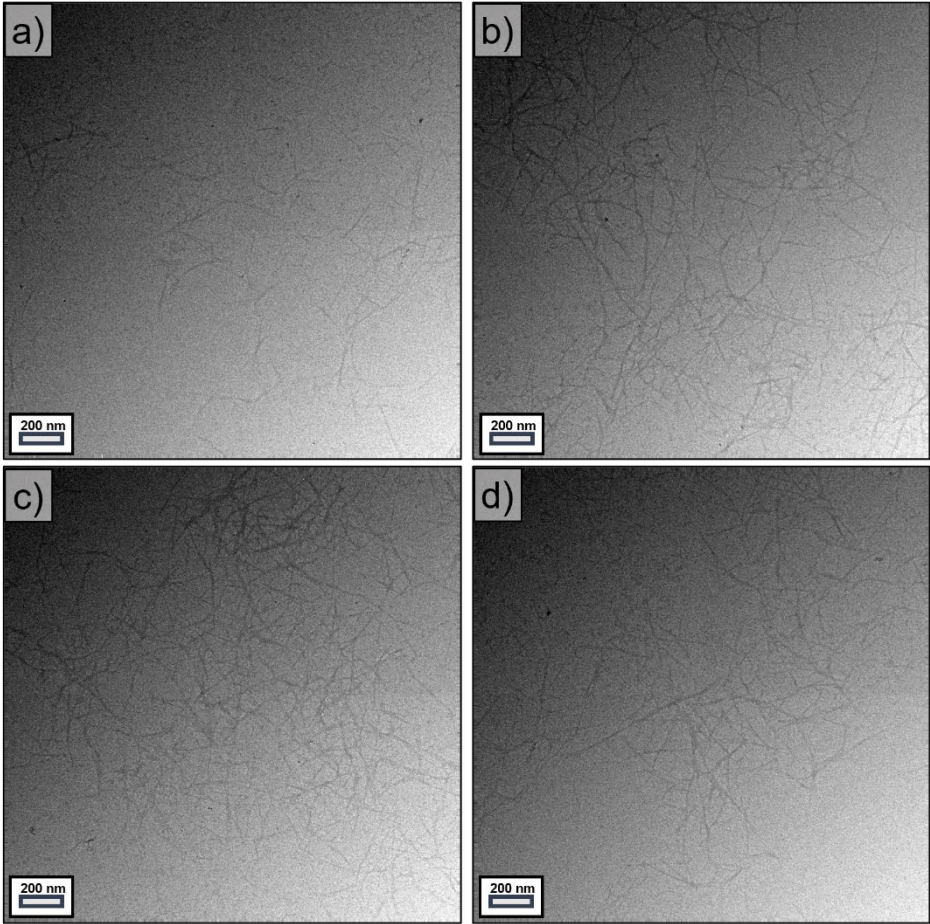


Fig. S3 – SANS data set and fitting details.

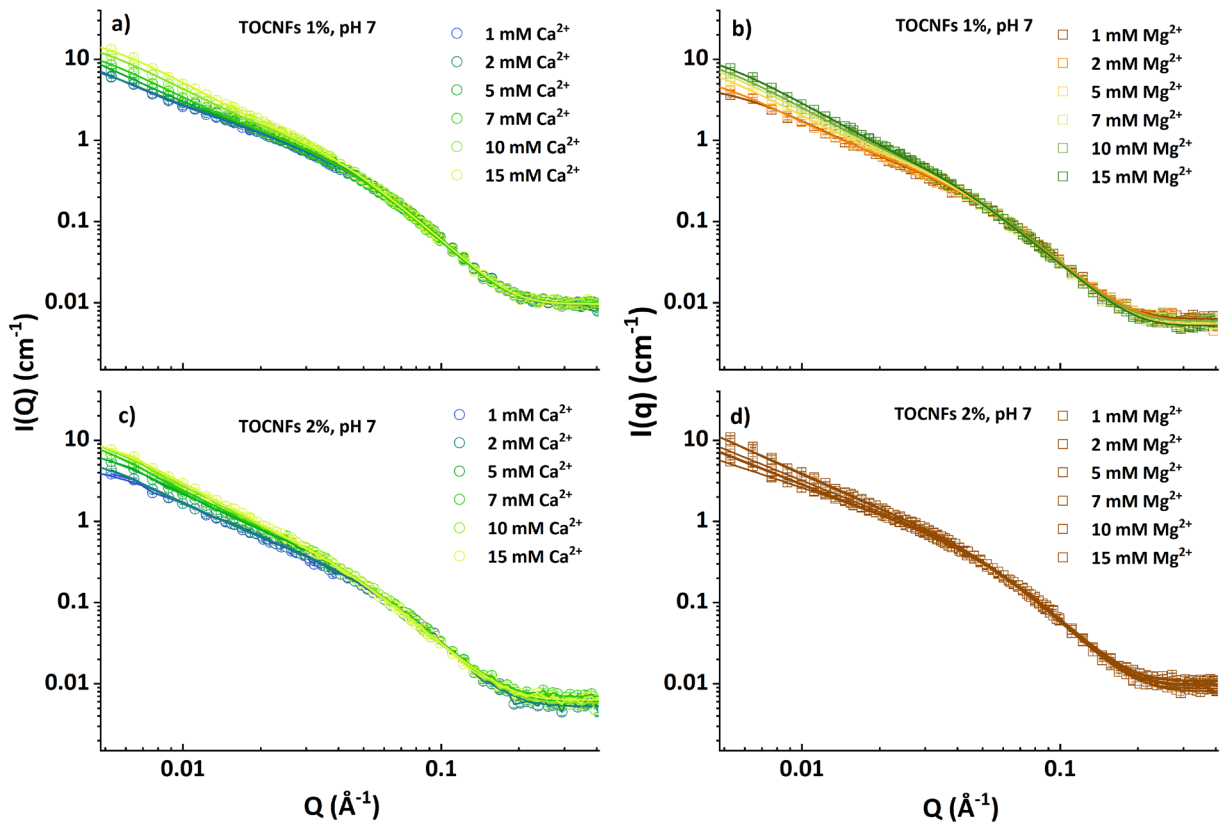


Fig. S4 – UVRR spectra and fitting details.

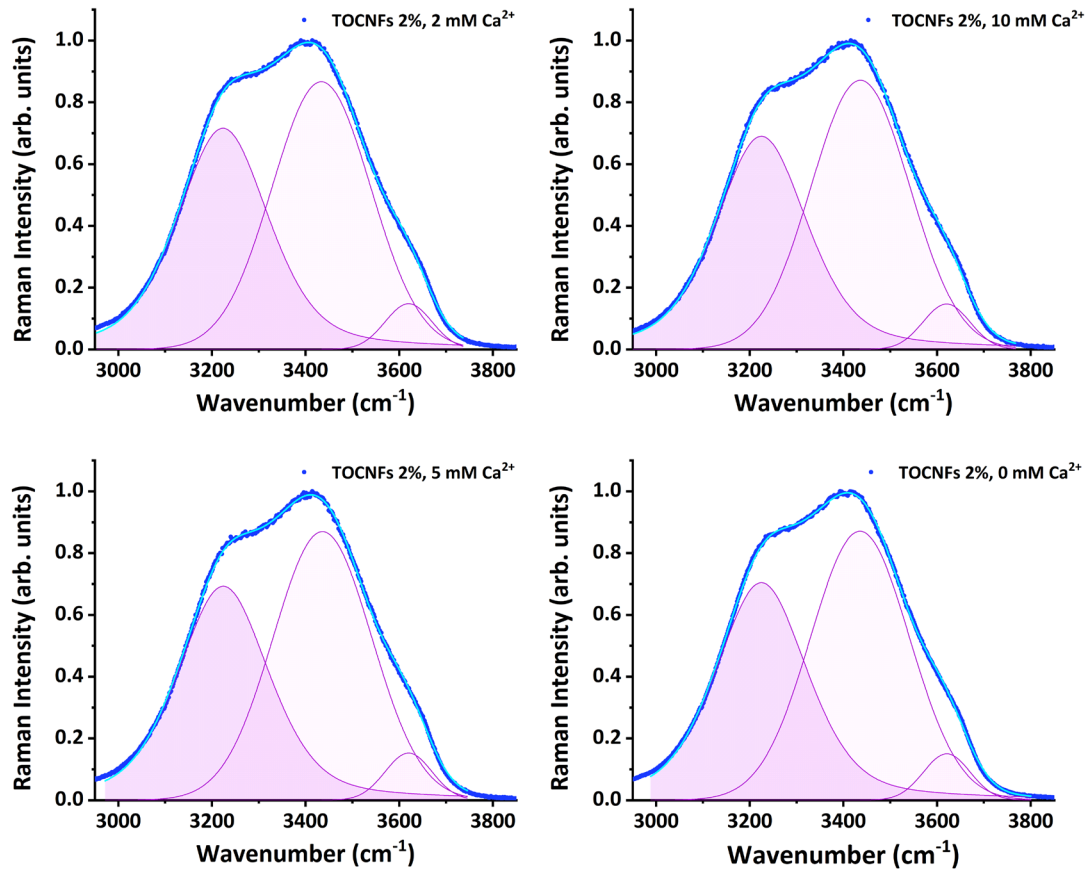
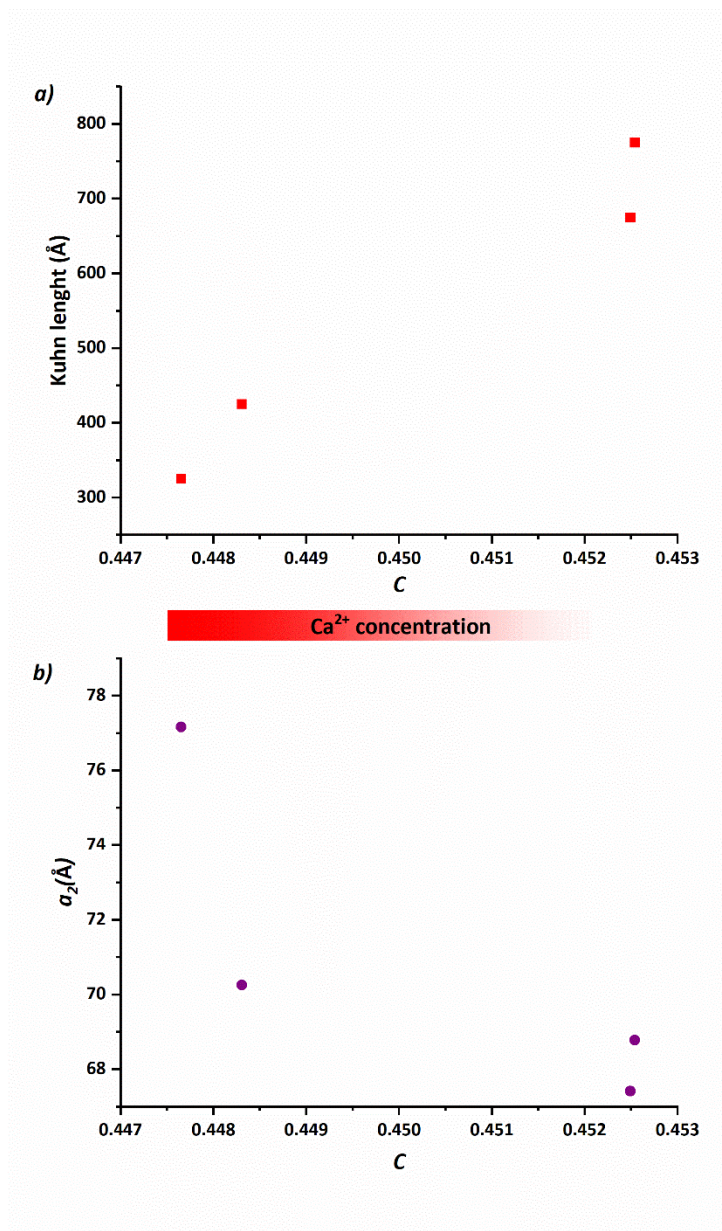


Fig. S5 – UVRR and SANS parameters correlations.



References

1. Segal L, Creely JJ, Martin AE, Conrad CM. An Empirical Method for Estimating the Degree of Crystallinity of Native Cellulose Using the X-Ray Diffractometer. *Textile Research Journal*. 1959;29(10):786-794. doi:10.1177/004051755902901003