Electronic Supplementary Material (ESI) for Nanoscale.

High Strength Nanostructured Films Based on Well-Preserved β-chitin Nanofibrils

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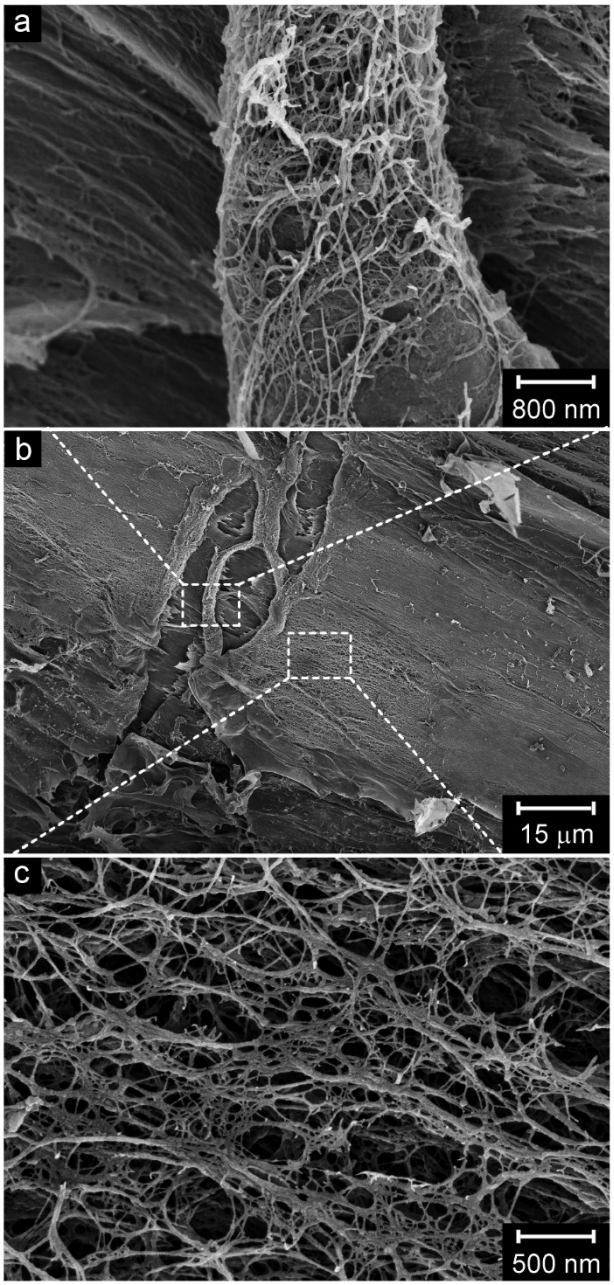

Figure S1. SEM images of the freeze-dried squid pen surfaces.

Table S1. Single-pulse MAS ^{13}C NMR spectral data of extracted chitin nanofibrils.

Resonances	Chemical shift [ppm]
C=O	173.8
C1	103.9
C4	83.4
C5/C3	75.1
C6	61.3
C2	55.7
CH3	23.1

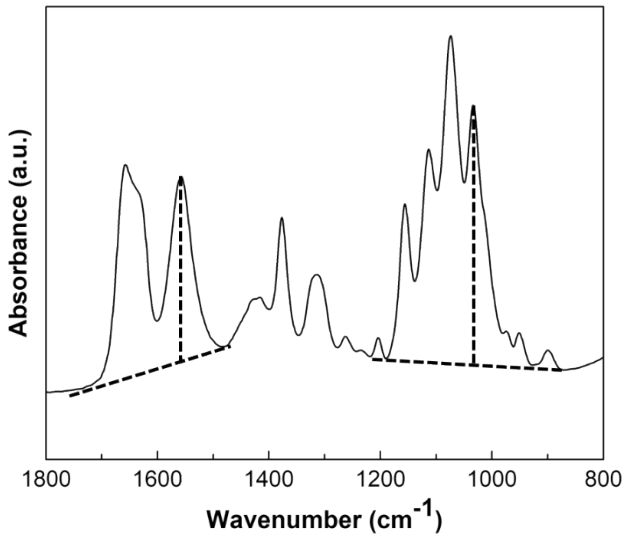

Figure S2. FTIR spectrum of chitin nanofibrils.

The degree of acetylation (DA) was evaluated by FTIR. The two band at 1030 and 1560 cm^{-1} are due to C−O stretching vibration of the chitin skeleton and amide II groups, respectively. According to the work done by Shigemasa et al,[1] the intensity ratio of A_{1560}/A_{1030} has a linear relation with the value of DA:

$$\frac{A1560}{A1030} = -0.7(100-DA) + 0.8 \qquad (1)$$

The baselines were also chosen according to Shigemasa et al, as shown in Figure S2. The resulted DA of chitin nanofibrils extracted by mild treatment was 97 ± 1.4 %.

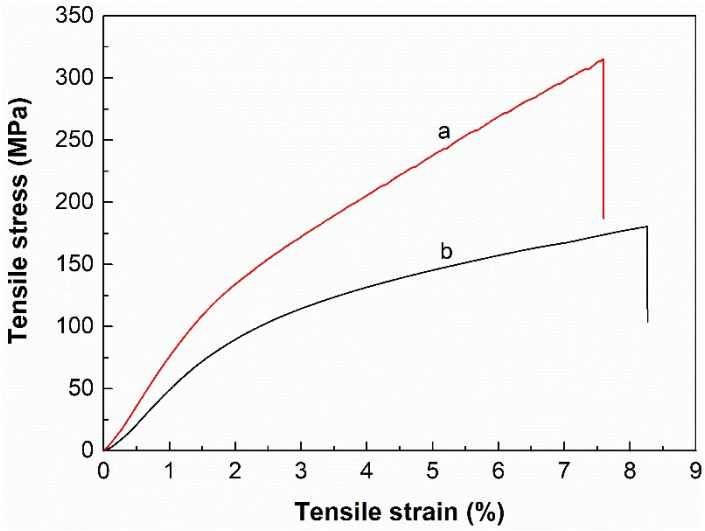

Figure S3. Tensile stress-strain curves of chitin nanostructured films prepared from chitin nanofibrils based on mild treatment (a) and more aggressive treatment (b).

The more aggressive treatment was done by repeating 5 times of deproteinization (each time: 10 % NaOH for 12 hrs and room temperature), where fresh NaOH was used at each time. The rest preparation steps are the same as ChNF described in the article. The resulted chitin nanofibrils by more aggressive treatment were denoted as D-ChNF (surface-deacetylated chitin nanofibrils). The FTIR analysis showed the D-ChNF has a lower DA of 92%, which is due to the more aggressive extraction conditions. The average diameter of the D-ChNF measured from TEM images is 3.8 ± 1 nm (ChNF : 4.1 ± 1 nm). The tensile stress-strain curves in Figure S3 reveal that the nanostructured films prepared from D-ChNF have much lower mechanical properties, as listed in Table S2.

Table S2. Young's modulus and maximum stress at break of films prepared from ChNF and D-ChNF, respectively.

	Young's modulus (GPa)	**Maximum stress at break (MPa)**
ChNF film	6.7 ± 1.2	277 ± 27
D-ChNF film	5.7 ± 0.5	178 ± 9

References

1. Y. Shigemasa, H. Matsuura, H. Sashiwa and H. Saimoto, *Int J Biol Macromol*, 1996, **18**, 237-242.