*materials*

MDPI

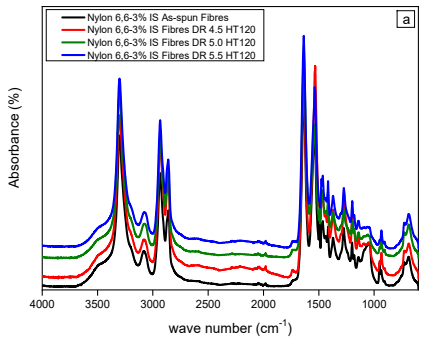
*Supplementary Materials*

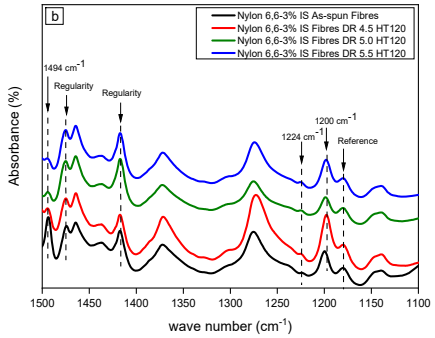
# Transient Confinement of the Quaternary Tetramethylammonium Tetrafluoroborate Salt in Nylon 6,6 Fibres: Structural Developments for High Performance Properties

**Ahmed Dawelbeit and Muhuo Yu ***

State Key Laboratory for Modification of Chemical Fibers and Polymer Materials, College of Materials Science and Engineering, Donghua University, Shanghai 201620, China; 413004@mail.dhu.edu.cn
* Correspondence: yumuhuo@dhu.edu.cn

## 1. Supplemental Figures




**Citation:** Dawelbeit, A.; Yu, M. Transient Confinement of the Quaternary Tetramethylammonium Tetrafluoroborate Salt in Nylon 6,6 Fibres: Structural Developments for High Performance Properties. *Materials* **2021**, *14*, 2938. https://doi.org/10.3390/ma14112938

Academic Editor: Jānis Andersons

Received: 24 April 2021
Accepted: 26 May 2021
Published: 10 June 2021

**Publisher's Note:** MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.

**Copyright:** © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (http://creativecommons.org/licenses/by/4.0/).

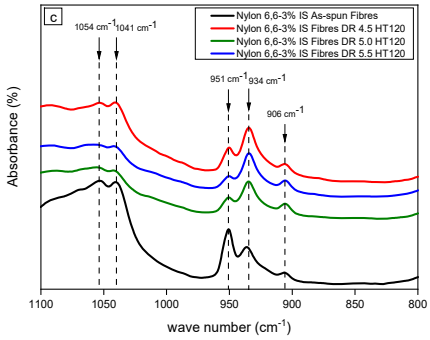



**Figure S1.** The FTIR spectrum of the as-spun and drawn salt-confined nylon 6,6 fibres at the drawing temperature of 120 °C at the band regions of (**a**) 4000–600 $cm^{-1}$, (**b**) 1500–1100 $cm^{-1}$ and (**c**) 1100–800 $cm^{-1}$.

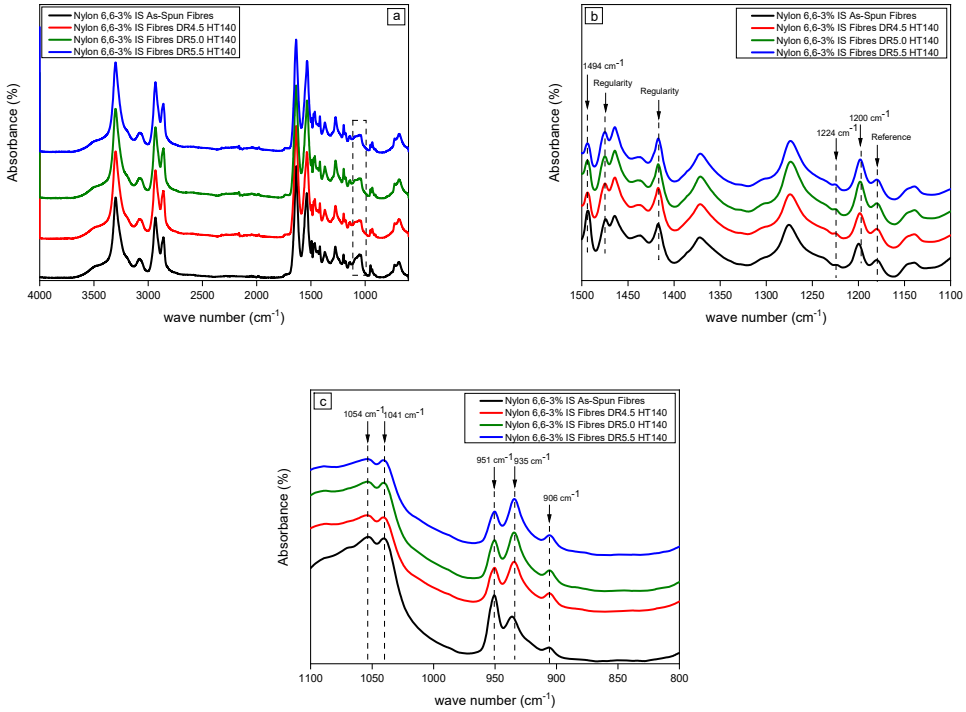



**Figure S2.** The FTIR spectrum of the as-spun and drawn salt-confined nylon 6,6 fibres at the drawing temperature of 140 °C at the band regions of (**a**) 4000–600 $cm^{-1}$, (**b**) 1500–1100 $cm^{-1}$ and (**c**) 1100–800 $cm^{-1}$.

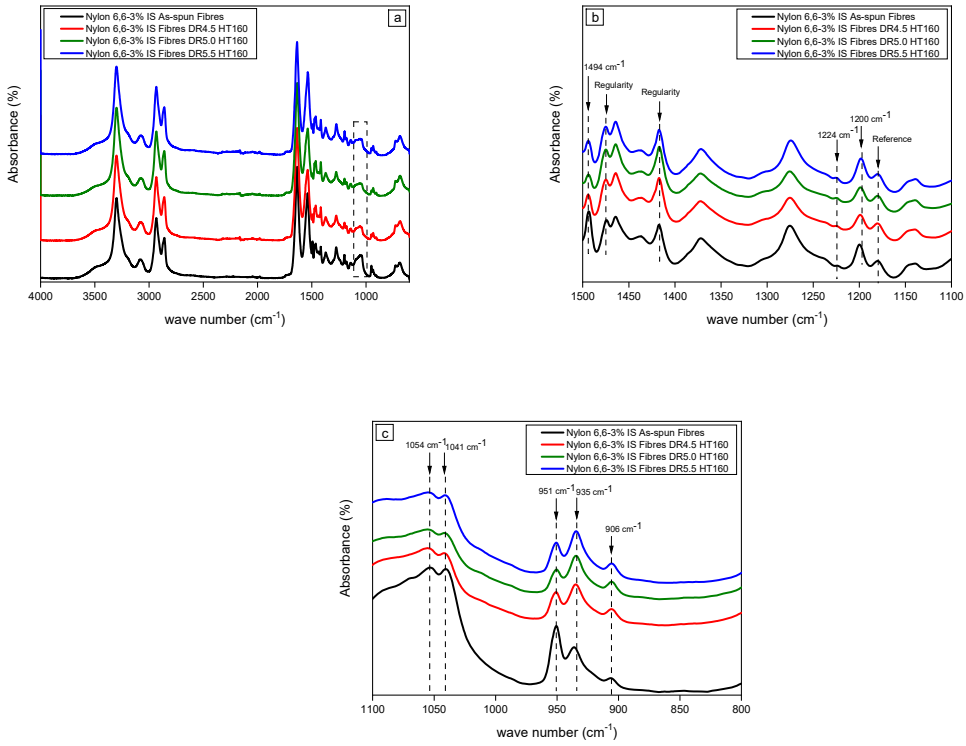



**Figure S3.** The FTIR spectrum of the as-spun and drawn salt-confined nylon 6,6 fibres at the drawing temperature of 160 °C at the band regions of (**a**) 4000–600 $cm^{-1}$, (**b**) 1500–1100 $cm^{-1}$ and (**c**) 1100–800 $cm^{-1}$.

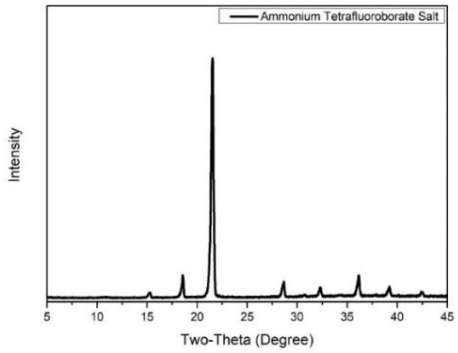



**Figure S4.** XRD of the TMA $BF_4$ salt.

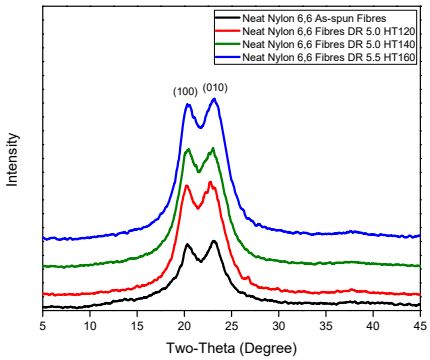



**Figure S5.** XRD pattern as-spun and drawn neat nylon 6,6 fibres, drawn at maximum draw ratio and at drawing temperature of 120, 140 and160 °C.

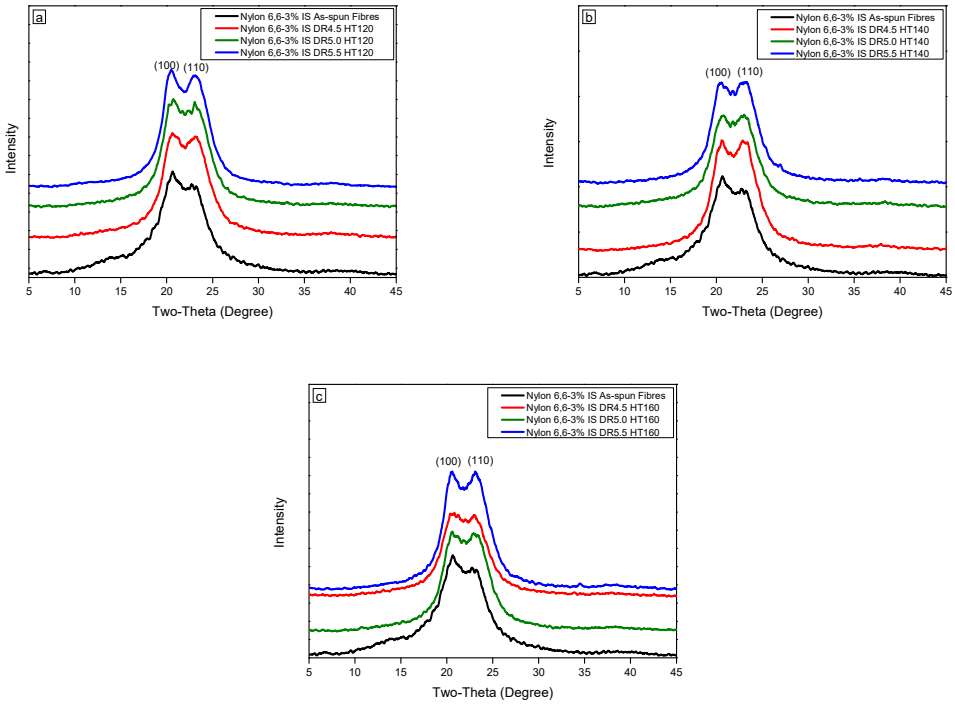



**Figure S6.** XRD pattern of 3% salt-confined as-spun and drawn nylon 6,6 fibres at drawing temperature of (**a**) 120, (**b**) 140 and (**c**) 160 °C.

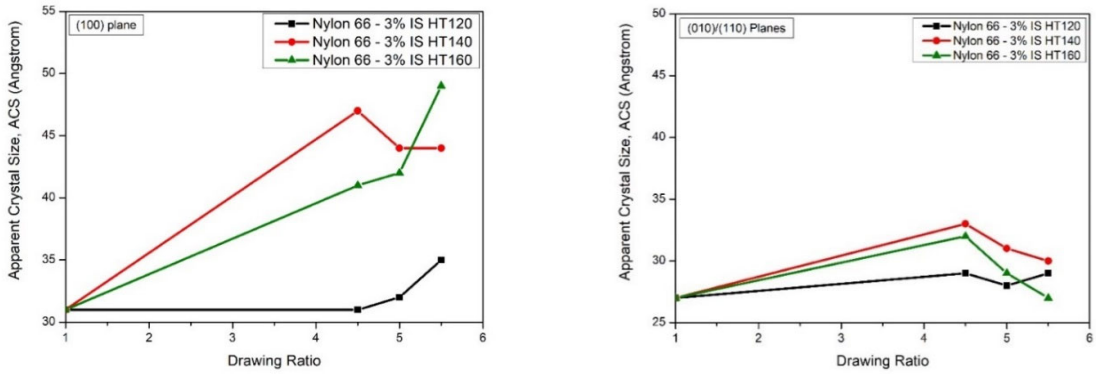



**Figure S7.** The ACS of the salt-confined nylon 6,6 fibres for the (100) and the (010/110) diffraction planes versus the drawing ratio at different drawing temperatures.

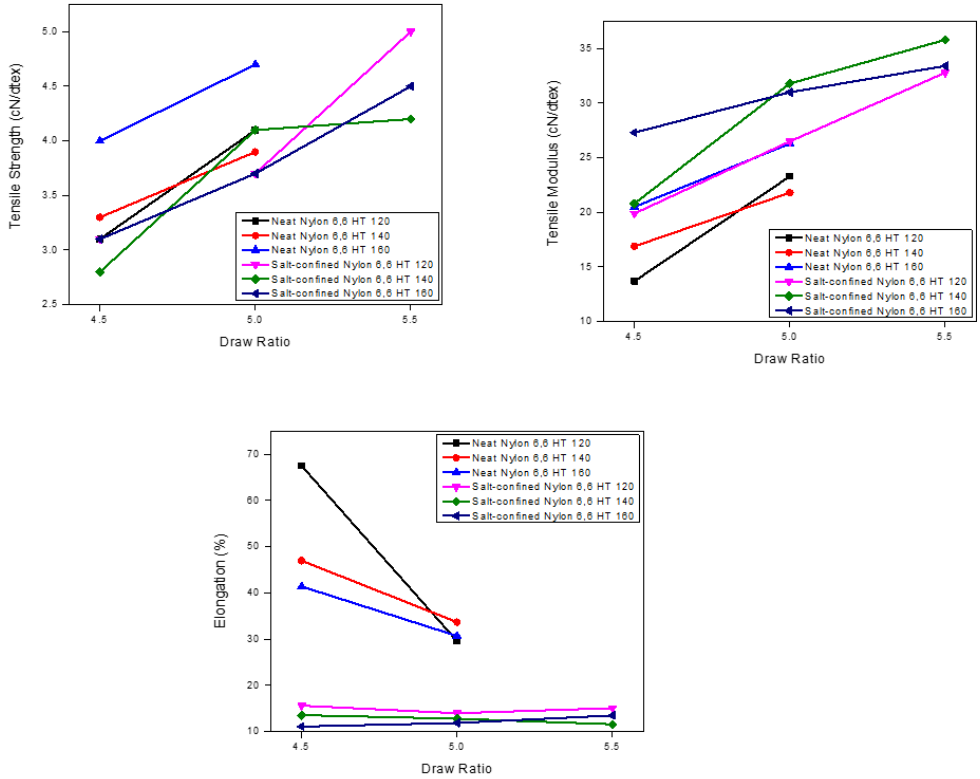



**Figure S8.** The tensile strengths, tensile moduli and elongation of the neat and the salt-confined nylon 6,6 fibres versus the drawing ratio at drawing temperatures of 120, 140 and 160 °C.

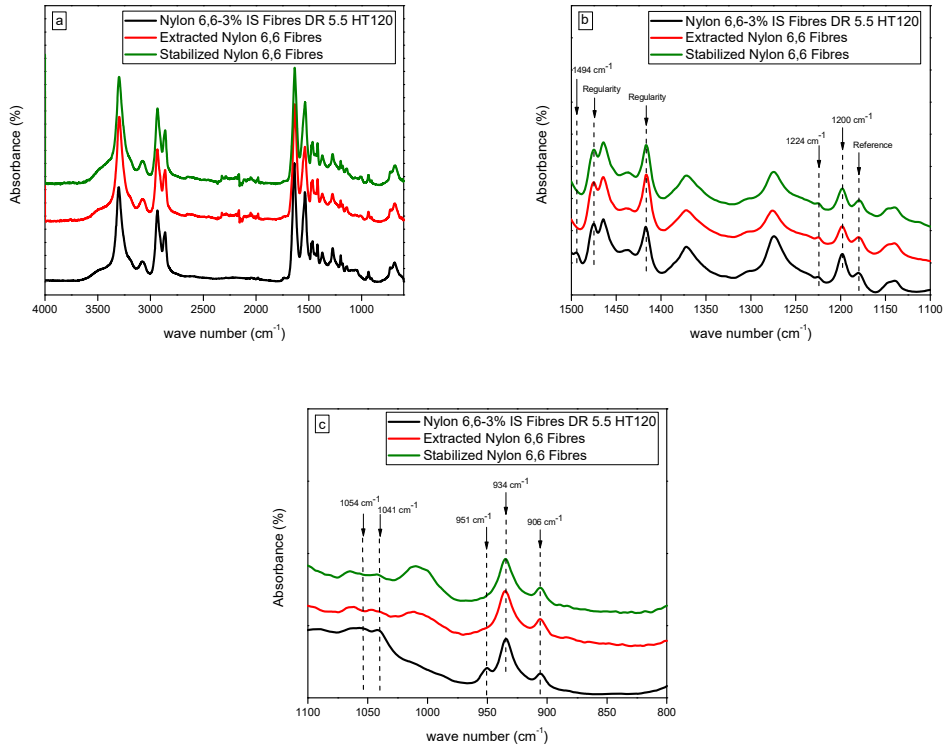
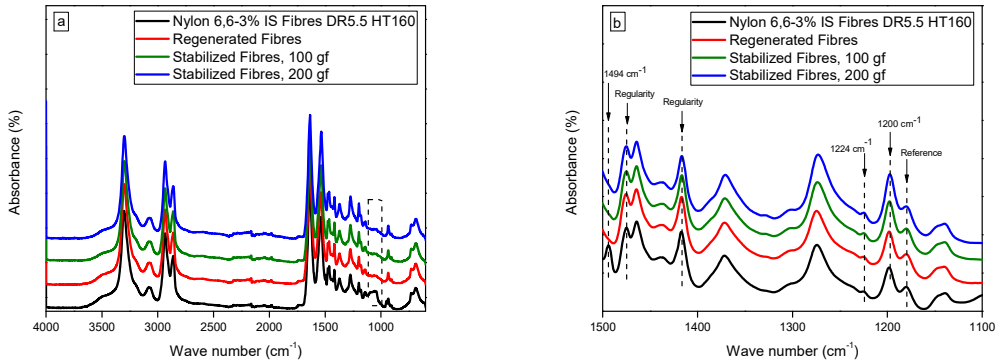



**Figure S9.** The FTIR spectrum of the salt-confined (drawn at, maximum, 5.5 draw ratio and at drawing temperature of 120 °C) and reverted nylon 6,6 fibres at the band regions of (**a**) 4000–600 $cm^{-1}$, (**b**) 1500–1100 $cm^{-1}$ and (**c**)1100–800 $cm^{-1}$.



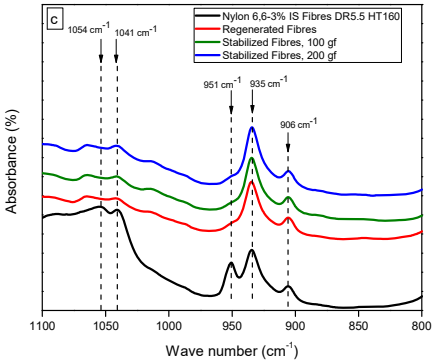



**Figure S10.** The FTIR spectrum of the salt-confined (drawn at, maximum, 5.5 draw ratio and at drawing temperature of 160 °C) and reverted nylon 6,6 fibres at the band regions of (**a**) 4000–600 $cm^{-1}$, (**b**) 1500–1100 $cm^{-1}$ and (**c**)1100–800 $cm^{-1}$.

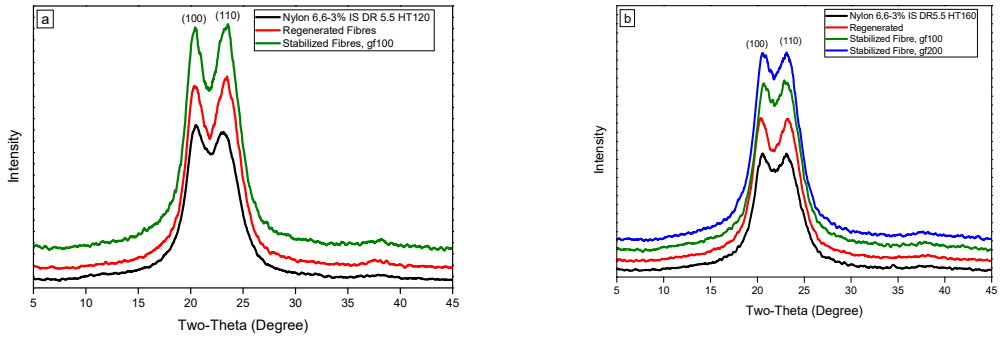
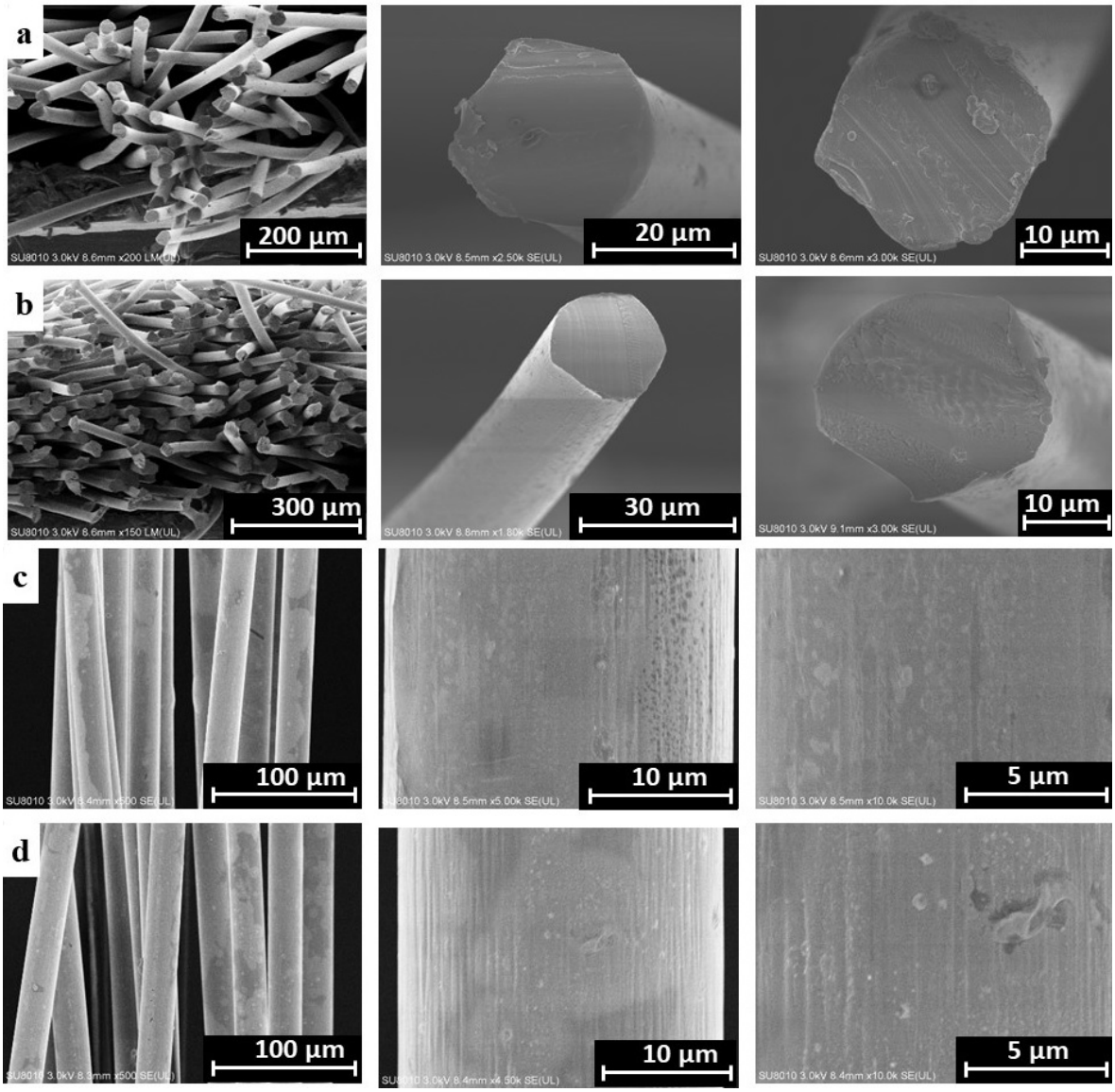



**Figure S11.** XRD pattern of salt-confined nylon 6,6 fibres that drawn at, maximum, 5.5 draw ratio and at drawing temperature of (**a**) 120 and (**b**) 160 °C and its reverted fibres.


a
200 µm
20 µm
10 µm
b
300 µm
30 µm
10 µm
c
100 µm
10 µm
5 µm
d
100 µm
10 µm
5 µm

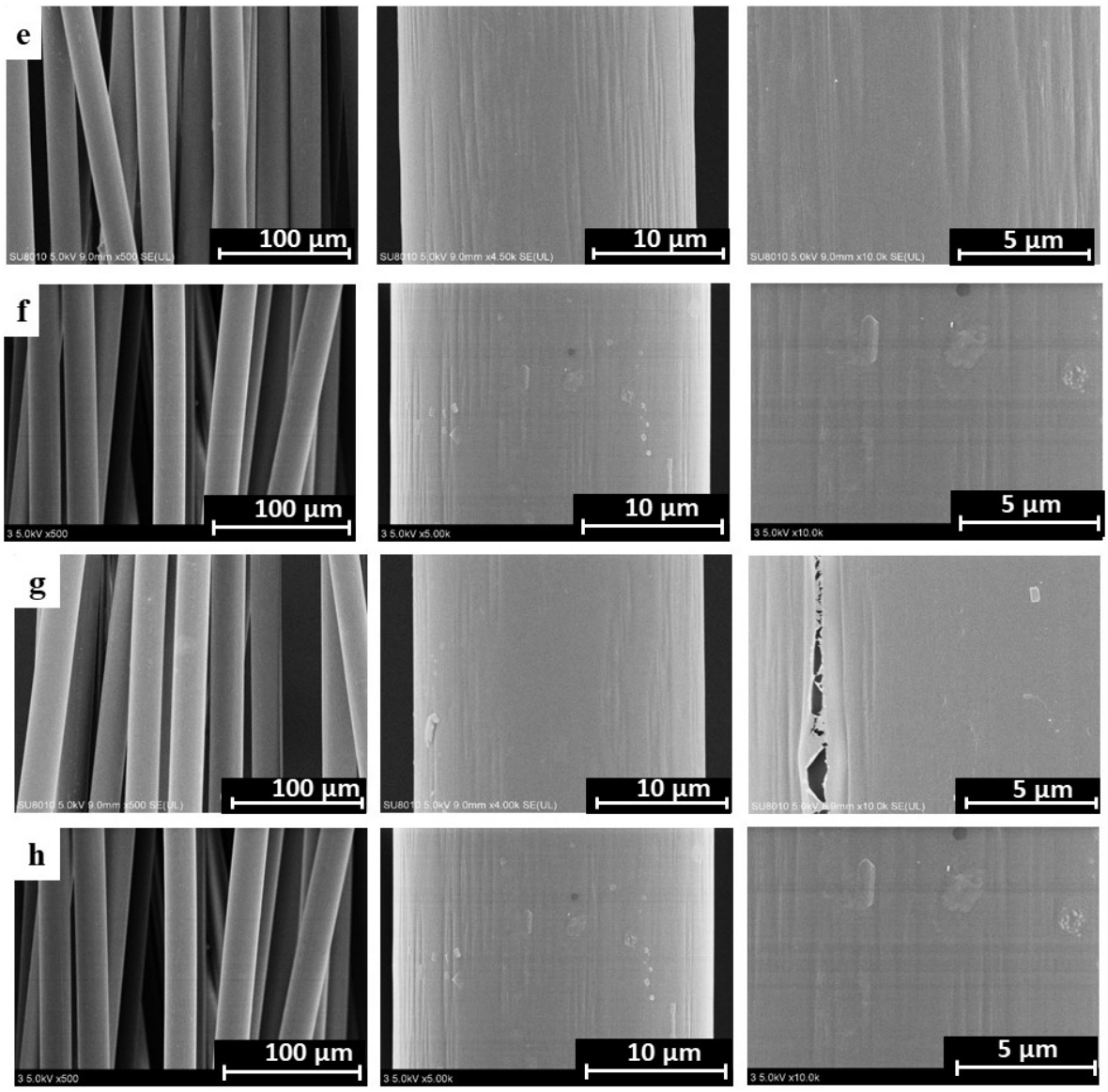



**Figure S12.** SEM micrographs of the reverted nylon 6,6 fibres. The morphological cross-section structures of (**a**) the regenerated and (**b**) the stabilized nylon 6,6 fibres (the regeneration have been done for the 3% salt-confined nylon 6,6 fibres drawn at drawing ratio of 5.5 and drawing temperature of 120 °C). The morphological surfaces structures of (**c**) the regenerated and (**d**) the stabilized nylon 6,6 fibres (the regeneration have been done for the 3% salt-confined nylon 6,6 fibres drawn at drawing ratio of 5.5 and drawing temperature of 120 °C). The morphological surfaces structures of (**e**) the regenerated and (**f**) the stabilized nylon 6,6 fibres (the regeneration have been done for the 3% salt-confined nylon 6,6 fibres drawn at drawing ratio of 5.5 and drawing temperature of 140 °C). The morphological surfaces structures of (**g**) the regenerated and (**h**) the stabilized nylon 6,6 fibres (the regeneration have been done for the 3% salt-confined nylon 6,6 fibres drawn at drawing ratio of 5.5 and drawing temperature of 160 °C).

## 2. Supplemental Tables

**Table S1.** Crystallographic properties of the as-spun and drawn neat nylon 6,6 fibres.

| Samples | Crystallinity (%) | Interplanar space (d-spacing), Å | | Crystal perfection, CPI (%) | Apparent crystal size, ACS (Å) | | $\cos^2\emptyset_{hkl}$ | Degree of Orientation ($f_c$) | Orientation angle (degree) |
|---|---|---|---|---|---|---|---|---|---|
| | | *100* | *010* | | *100* | *010* | | | |
| As-spun Neat Nylon 6,6 Fibre | 38.24 | 4.3671 | 3.8541 | 70.426 | 70 | 56 | 0.238 | -0.144 | 60.84 |
| Neat Nylon 6,6 Fibres DR 5.0 HT120 | 62.81 | 4.3959 | 3.8946 | 68.104 | 51 | 34 | 0.913 | 0.87 | 17.14 |
| Neat Nylon 6,6 Fibres DR 5.0 HT140 | 59.35 | 4.3763 | 3.8854 | 66.85 | 51 | 32 | 0.810 | 0.715 | 25.83 |
| Neat Nylon 6,6 Fibres DR 5.5 HT160 | 67.72 | 4.3680 | 3.8402 | 72.72 | 34 | 31 | 0.750 | 0.625 | 30.00 |

**Table S2.** Crystal Structural Parameters of Salt-confined and Reverted Nylon 6,6 Fibres.

| Samples | Treatment Temperature (°C) | Tension (gram force) | Crystallinity (%) | Apparent crystal size, ACS (Å) | | Crystal perfection, CPI (%) | $\cos^2\emptyset_{hkl}$ | Degree of Orientation ($f_c$) | Orientation angle (degree) |
|---|---|---|---|---|---|---|---|---|---|
| | | | | *100* | *010* | | | | |
| Salt-confined Fibres | 120 | DR 5.5 | 72.58 | 35 | 29 | 72.22 | 0.76 | 0.640 | 29.34 |
| Regenerated Fibres | RT | - | 79.31 | 38 | 30 | 80.08 | 0.728 | 0.592 | 31.46 |
| Stabilized Fibres | 190 | 100 | 82.36 | 39 | 29 | 79.79 | 0.924 | 0.886 | 16.01 |
| Salt-confined Fibres | 140 | DR 5.5 | 58.02 | 44 | 30 | 72.35 | 0.928 | 0.892 | 15.55 |
| Regenerated Fibres | RT | - | 60.82 | 33 | 31 | 72.75 | 0.785 | 0.677 | 27.64 |
| Stabilized Fibres | 190 | 100 | 74.99 | 34 | 32 | 67.92 | 0.774 | 0.661 | 28.40 |
| Stabilized Fibres | 190 | 200 | 70.82 | 40 | 29 | 67.09 | 0.931 | 0.897 | 15.20 |
| Salt-confined Fibres | 160 | DR 5.5 | 54.86 | 49 | 27 | 67.74 | 0.886 | 0.829 | 19.72 |
| Regenerated Fibres | RT | - | 71.98 | 37 | 30 | 75.43 | 0.902 | ddd | 18.25 |
| Stabilized Fibres | 190 | 100 | 71.15 | 36 | 30 | 65.75 | 0.861 | 0.791 | 21.92 |
| Stabilized Fibres | 190 | 200 | 68.37 | 36 | 30 | 69.90 | 0.860 | 0.790 | 21.97 |