

## Supporting Information

### **Pioneering Wound Care Solutions: Triaxial Wet-Spun Fibers with Bioactive Agents for Chronic Wounds – Part I (Production and characterization of the triaxial fibers)**

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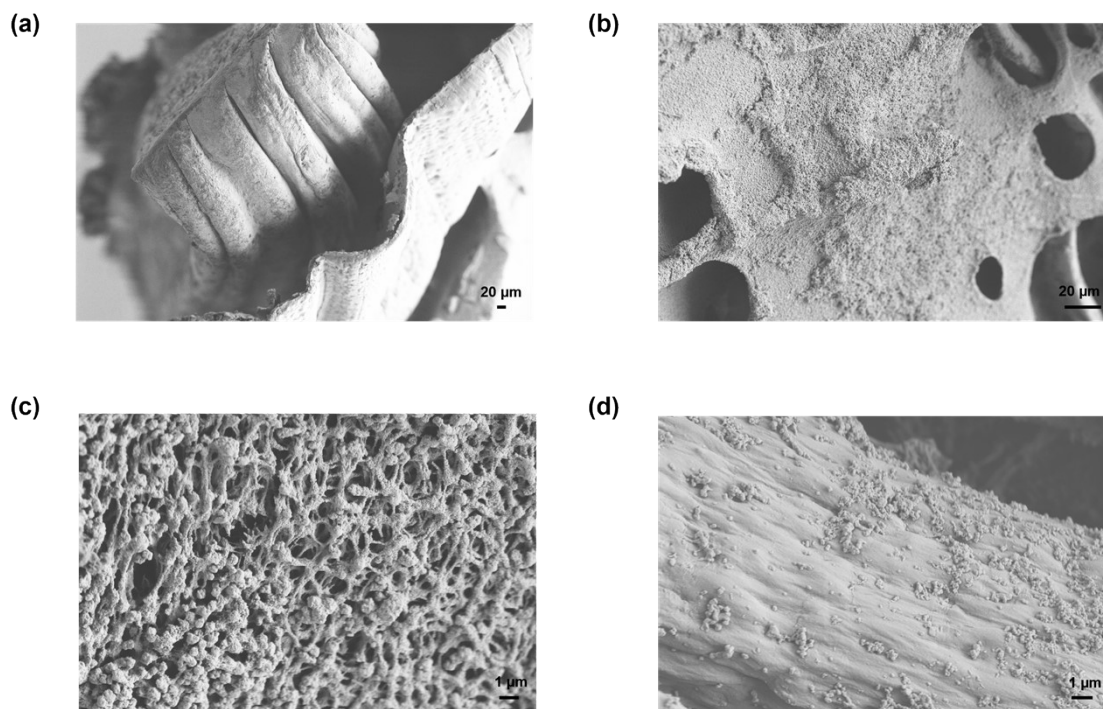
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### Section S1. Morphological Observations of Triaxial Wet-spun Fibers

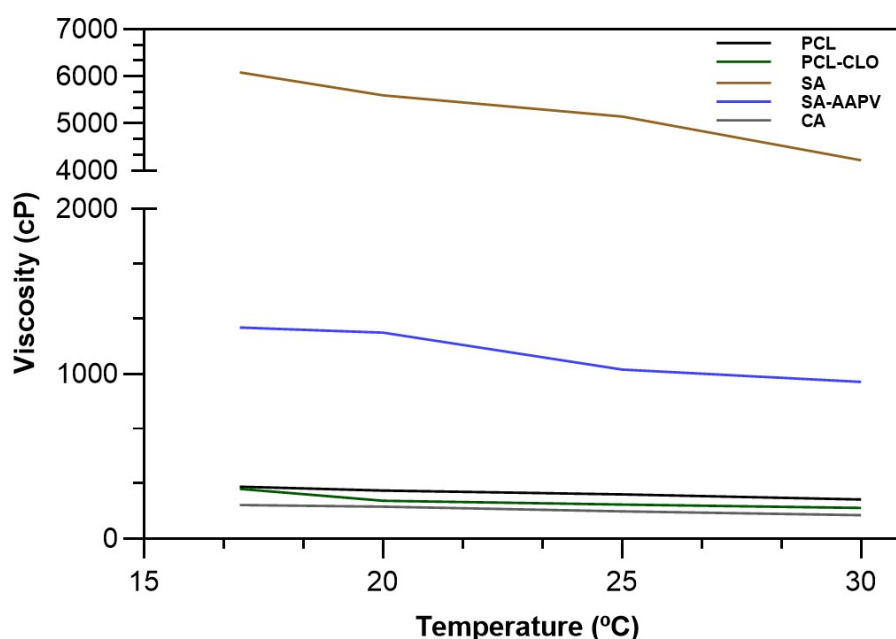
Additional micrographs of the fibers' morphology were acquired with SEM and presented below (Figure S1):



**Figure S1.** Wet-spun PCL-CLO/SA-AAPV/CA fibers morphology attained by SEM: (a), (b) cross section and (c), (d) surface images.

### Section S2. Rheological Properties of Polymeric Solutions

To better understand the influence of both AAPV and CLO additives on the rheological properties of PCL and SA, the viscosities of all solutions with/without additives were measured between 17 and 30 °C (Figure S2).



**Figure S2.** Viscosity variation for PCL, PCL-CLO, SA, SA-AAPV and CA solutions with temperature.

### Section S3. Chemical, Thermal and Mechanical Characterizations of the Wet-spun Fibers

ATR-FTIR analyses were performed on all fiber typologies, in which several characteristic peaks were detected and listed in Table S1, confirming the presence of PCL, SA and CA polymers.

**Table S1.** List of characteristic peaks PCL, SA and CA detected by ATR-FTIR technique.

| Wavenumber (cm <sup>-1</sup> ) | Functional Group Assigned         |
|--------------------------------|-----------------------------------|
| ≈ 3300                         | -OH groups                        |
| 2950                           | C-H stretching vibrations         |
| 2867                           | C-H stretching vibrations         |
| 1725                           | C=O stretching vibrations         |
| 1600                           | COO <sup>-</sup> group vibrations |
| 1440                           | CH <sub>2</sub> vibrations        |
| 1420                           | COO <sup>-</sup> group vibrations |
| 1246                           | C-O-C vibrations                  |
| 1222                           | acetyl ester groups               |
| 1173                           | C-O-C vibrations                  |
| 1044                           | C-O stretching vibrations         |

DSC measurements were also conducted to evaluate the fibers' thermal behavior. Onset and peak temperatures were registered for the polymers composing the fibers, along with the enthalpies for each characteristic peak (Table S2).

**Table S2.** Main DSC thermal events ( $T_O$  onset temperature,  $T_P$  peak temperature and  $\Delta H$  enthalpy).

| <b>Polymer</b>         | <b><math>T_O</math> (°C)</b> | <b><math>T_P</math> (°C)</b> | <b><math>\Delta H</math> (J/g)</b> | <b><math>T_O</math> (°C)</b> | <b><math>T_P</math> (°C)</b> | <b><math>\Delta H</math> (J/g)</b> | <b><math>T_O</math> (°C)</b> | <b><math>T_P</math> (°C)</b> | <b><math>\Delta H</math> (J/g)</b> | <b><math>T_O</math> (°C)</b> | <b><math>T_P</math> (°C)</b> | <b><math>\Delta H</math> (J/g)</b> |
|------------------------|------------------------------|------------------------------|------------------------------------|------------------------------|------------------------------|------------------------------------|------------------------------|------------------------------|------------------------------------|------------------------------|------------------------------|------------------------------------|
| <b>PCL</b>             | 49.89                        | 58.42                        | 63.73                              |                              |                              |                                    | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>PCL-CLO</b>         | 44.54                        | 51.22                        | 42.07                              | 302.76                       | 303.28                       | 4.08                               | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>SA</b>              | 141.87                       | 143.34                       | 403.49                             | -                            | -                            | -                                  | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>SA-AAPV</b>         | 135.13                       | 136.76                       | 1.96                               | 157.27                       | 163.30                       | 390.45                             | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>CA</b>              | 160.69                       | 164.32                       | 123.48                             | 266.42                       | 271.91                       | 29.12                              | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>PCL/SA</b>          | 48.96                        | 55.39                        | 219.54                             | 395.56                       | 419.73                       | 185.05                             | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>PCL/SA-AAPV</b>     | 106.26                       | 106.78                       | 10.86                              | 131.82                       | 134.65                       | -387.24                            | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>PCL-CLO/SA</b>      | 45.32                        | 49.60                        | 41.04                              | 169.87                       | 173.20                       | 12.27                              | 388.60                       | 413.24                       | 169.11                             | -                            | -                            | -                                  |
| <b>PCL-CLO/SA-AAPV</b> | 96.20                        | 124.32                       | 335.46                             | 325.41                       | 345.10                       | 60.21                              | -                            | -                            | -                                  | -                            | -                            | -                                  |
| <b>PCL/CA</b>          | 47.43                        | 51.82                        | 23.23                              | 140.52                       | 144.47                       | 22.65                              | 370.13                       | 387.51                       | 17.26                              | -                            | -                            | -                                  |
| <b>PCL-CLO/CA</b>      | 37.92                        | 46.20                        | 10.24                              | 137.09                       | 141.95                       | 260.32                             | 277.37                       | 280.89                       | 6.43                               | 377.24                       | 393.02                       | 74.78                              |

|                                     |        |        |        |        |        |       |   |   |   |   |   |   |
|-------------------------------------|--------|--------|--------|--------|--------|-------|---|---|---|---|---|---|
| <b>SA/CA</b>                        | 113.93 | 126.74 | 157.77 | 266.02 | 271.73 | 25.38 | - | - | - | - | - | - |
| <b>SA-<br/>AAPV/CA</b>              | 96.48  | 120.77 | 196.61 | -      | -      | -     | - | - | - | - | - | - |
| <b>PCL/SA/C<br/>A</b>               | 102.62 | 123.86 | 340.42 | -      | -      | -     | - | - | - | - | - | - |
| <b>PCL/SA-<br/>AAPV/CA</b>          | 137.43 | 151.34 | 510.53 | -      | -      | -     | - | - | - | - | - | - |
| <b>PCL-<br/>CLO/SA/C<br/>A</b>      | 101.14 | 123.37 | 254.99 | 380.72 | 395.84 | 14.08 | - | - | - | - | - | - |
| <b>PCL-<br/>CLO/SA-<br/>AAPV/CA</b> | 68.84  | 109.64 | 370.01 | -      | -      | -     | - | - | - | - | - | - |

The mechanical properties of the wet-spun fibers were also evaluated by determining their tenacities and maximum elongations at break (Table S3).

**Table S3.** Mechanical examinations of the engineered wet-spun fibers. Results are plotted as mean  $\pm$  SD (n = 6). Statistical significance was determined via Tukey test by applying multiple comparisons between the different fiber typologies (all tenacities from fiber inner, intermediate and outer layers were statistically different:  $p < 0.0001$ ; all maximum elongations from fiber inner and outer layers were statistically different:  $p < 0.0001$ ; significance between the maximum elongation of SA and SA-AAPV, SA/CA, PCL-CLO/SA-AAPV/CA:  $p < 0.0009$ ).

| Fiber typology | Core               |                  | Intermediate Layer |                  | Shell            |                  |
|----------------|--------------------|------------------|--------------------|------------------|------------------|------------------|
|                | Elongation (%)     | Tenacity (MPa)   | Elongation (%)     | Tenacity (MPa)   | Elongation (%)   | Tenacity (MPa)   |
| PCL            | 414.80 $\pm$ 54.74 | 12.50 $\pm$ 0.01 | -                  | -                | -                | -                |
| PCL-CLO        | 610.50 $\pm$ 1.21  | 27.25 $\pm$ 0.01 | -                  | -                | -                | -                |
| SA             | -                  | -                | 28.88 $\pm$ 2.37   | 55.50 $\pm$ 0.06 | -                | -                |
| SA-AAPV        | -                  | -                | 43.12 $\pm$ 6.24   | 57.56 $\pm$ 0.14 | -                | -                |
| CA             | -                  | -                | -                  | -                | 28.56 $\pm$ 5.01 | 41.40 $\pm$ 0.03 |
| PCL/SA         | 584.41 $\pm$ 26.57 | 14.01 $\pm$ 0.01 | -                  | -                | -                | -                |
| PCL/SA-AAPV    | 651.90 $\pm$ 22.61 | 19.18 $\pm$ 0.03 | 20.68 $\pm$ 9.21   | 30.30 $\pm$ 0.03 | -                | -                |

|                           |                |              |              |               |              |              |
|---------------------------|----------------|--------------|--------------|---------------|--------------|--------------|
| <b>PCL-CLO/SA</b>         | 593.53 ± 11.40 | 22.21 ± 0.02 | -            | -             | -            | -            |
| <b>PCL-CLO/SA-AAPV</b>    | 570.36 ± 82.60 | 17.14 ± 0.04 | 21.95 ± 4.29 | 32.21 ± 0.02  | -            | -            |
| <b>PCL/CA</b>             | 635.38 ± 15.87 | 23.28 ± 0.05 | -            | -             | -            | -            |
| <b>PCL-CLO/CA</b>         | 685.09 ± 44.44 | 26.25 ± 0.07 | -            | -             | 10.49 ± 4.09 | 29.32 ± 0.06 |
| <b>SA/CA</b>              | -              | -            | 14.02 ± 0.16 | 28.29 ± 0.04  | -            | -            |
| <b>SA-AAPV/CA</b>         | -              | -            | 27.65 ± 2.54 | 12.43 ± 0.04  | 16.06 ± 3.03 | 26.23 ± 0.09 |
| <b>PCL/SA/CA</b>          | 710.05 ± 23.61 | 2.04 ± 0.02  | -            | -             | 10.96 ± 5.54 | 11.12 ± 0.01 |
| <b>PCL/SA-AAPV/CA</b>     | 620.19 ± 9.40  | 7.05 ± 0.01  | -            | -             | 35.99 ± 5.61 | 5.06 ± 0.01  |
| <b>PCL-CLO/SA/CA</b>      | 558.92 ± 98.49 | 7.09 ± 0.01  | -            | -             | -            | -            |
| <b>PCL-CLO/SA-AAPV/CA</b> | 560.47 ± 1.41  | 4.05 ± 0.01  | 45.61 ± 0.24 | 13.03 ± 21.07 | 15.54 ± 4.89 | 12.15 ± 0.01 |



In order to complement the discussion, a photograph is presented (Figure S3), showing the rupture of the fiber

is presented (Figure S3), to mechanical testing:

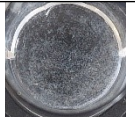


**Figure S3.** Rupture of intermediate and outer layers of the triaxial system, while subjected to the mechanical tests.

#### Section S4. Fibers Swelling and Water Retention

Swelling ratios and water retentions for each fiber typology were determined, after different incubation times in PBS (Table S4):

**Table S4.** Wet-spun fibers' swelling ratios and water retentions over 7 days of incubation in PBS. Data is presented as average percentage of degree of swelling (DS)/average percentage of water retention (WR)  $\pm$  SD (n = 3).

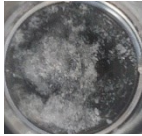
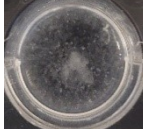
| Fiber typology  | DS at day 1 (%)    | WR at day 1 (%)     | DS at day 2 (%)   | WR at day 2 (%)     | DS at day 3 (%)   | WR at day 3 (%)    | DS at day 7 (%)                                                                     | WR at day 7 (%)                                                                     |
|-----------------|--------------------|---------------------|-------------------|---------------------|-------------------|--------------------|-------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------|
| PCL             | 134.56 $\pm$ 37.97 | 100 $\pm$ 0.01      | 58.47 $\pm$ 0.01  | 240.90 $\pm$ 6.88   | 63.30 $\pm$ 3.54  | 274.26 $\pm$ 27.53 | 66.73 $\pm$ 5.91                                                                    | 307.70 $\pm$ 60.17                                                                  |
| PCL-CLO         | 26.49 $\pm$ 27.85  | 100 $\pm$ 0.01      | 24.43 $\pm$ 11.62 | 134.58 $\pm$ 22.03  | 31.33 $\pm$ 14.24 | 149.47 $\pm$ 27.83 | 20.47 $\pm$ 16.55                                                                   | 129.16 $\pm$ 24.83                                                                  |
| SA              | 67.04 $\pm$ 8.99   | 320.01 $\pm$ 92.50  | 64.63 $\pm$ 15.73 | 322.23 $\pm$ 137.42 | 62.66 $\pm$ 10.28 | 283.22 $\pm$ 84.71 |  |  |
| SA-AAPV         | 87.44 $\pm$ 1.75   | 805.74 $\pm$ 104.08 | 88.11 $\pm$ 0.71  | 842.97 $\pm$ 51.75  | 84.80 $\pm$ 1.31  | 661.37 $\pm$ 57.07 | 86.25 $\pm$ 1.19                                                                    | 730.64 $\pm$ 60.90                                                                  |
| CA              | 22.93 $\pm$ 3.88   | 100 $\pm$ 0.01      | 13.97 $\pm$ 6.19  | 116.62 $\pm$ 8.06   | 7.94 $\pm$ 17.10  | 111.00 $\pm$ 19.25 | 18.79 $\pm$ 0.39                                                                    | 123.14 $\pm$ 0.59                                                                   |
| PCL/SA          | 56.17 $\pm$ 68.63  | 100 $\pm$ 0.01      | 21.95 $\pm$ 49.74 | 162.10 $\pm$ 82.25  | 27.35 $\pm$ 42.56 | 167.90 $\pm$ 79.24 | 40.45 $\pm$ 32.33                                                                   | 197.90 $\pm$ 83.22                                                                  |
| PCL-CLO/SA      | 95.79 $\pm$ 61.38  | 100 $\pm$ 0.01      | 57.374 $\pm$ 2.05 | 234.94 $\pm$ 11.04  | 53.77 $\pm$ 4.68  | 217.87 $\pm$ 22.92 | 22.55 $\pm$ 11.37                                                                   | 130.89 $\pm$ 18.29                                                                  |
| PCL/SA-AAPV     | 18.48 $\pm$ 59.31  | 100 $\pm$ 0.02      | 24.03 $\pm$ 21.89 | 140.56 $\pm$ 46.73  | 47.87 $\pm$ 10.97 | 197.64 $\pm$ 41.84 | 39.64 $\pm$ 16.55                                                                   | 176.15 $\pm$ 57.29                                                                  |
| PCL-CLO/SA-AAPV | 163.78 $\pm$ 30.92 | 100 $\pm$ 0.01      | 66.12 $\pm$ 2.73  | 296.48 $\pm$ 25.04  | 68.48 $\pm$ 2.60  | 318.76 $\pm$ 26.94 | 66.99 $\pm$ 2.06                                                                    | 303.71 $\pm$ 19.20                                                                  |
| PCL/CA          | 29.58 $\pm$ 26.72  | 100 $\pm$ 0.01      | 21.79 $\pm$ 23.11 | 134.68 $\pm$ 34.69  | 24.42 $\pm$ 19.07 | 138.01 $\pm$ 34.25 | 31.62 $\pm$ 14.60                                                                   | 150.42 $\pm$ 29.39                                                                  |

|                           |               |            |                |                |                |                |                |                |
|---------------------------|---------------|------------|----------------|----------------|----------------|----------------|----------------|----------------|
| <b>PCL-CLO/CA</b>         | 50.43 ± 19.24 | 100 ± 0.01 | 35.51 ± 7.91   | 156.59 ± 18.78 | 39.12 ± 9.50   | 166.80 ± 24.53 | 36.96 ± 12.96  | 162.83 ± 30.74 |
| <b>SA/CA</b>              | 39.01 ± 7.51  | 100 ± 0.02 | 28.72 ± 11.19  | 142.87 ± 24.63 | 27.91 ± 5.63   | 139.26 ± 10.60 | 26.65 ± 1.85   | 136.39 ± 3.41  |
| <b>SA-AAPV/CA</b>         | -11.07 ± 3.65 | 100 ± 0.02 | -6.49 ± 4.12   | 94.00 ± 3.68   | -6.13 ± 10.58  | 94.84 ± 9.23   | -22.72 ± 1.14  | 81.49 ± 0.76   |
| <b>PCL/SA/CA</b>          | -3.23 ± 15.89 | 100 ± 0.01 | 0.41 ± 18.50   | 103.03 ± 21.17 | 3.38 ± 24.65   | 109.01 ± 32.42 | -24.30 ± 12.92 | 81.07 ± 8.95   |
| <b>PCL-CLO/SA/CA</b>      | 35.29 ± 4.88  | 100 ± 0.02 | 13.27 ± 3.17   | 115.41 ± 4.23  | 15.60 ± 16.36  | 121.22 ± 21.22 | 7.94 ± 1.35    | 108.64 ± 1.60  |
| <b>PCL/SA-AAPV/CA</b>     | 8.58 ± 10.83  | 100 ± 0.02 | 6.46 ± 12.83   | 108.18 ± 13.89 | 8.99 ± 8.11    | 110.47 ± 10.09 | 7.88 ± 10.65   | 109.60 ± 13.56 |
| <b>PCL-CLO/SA-AAPV/CA</b> | 19.16 ± 27.80 | 100 ± 0.01 | -10.59 ± 19.64 | 92.25 ± 15.50  | -15.92 ± 14.26 | 87.11 ± 10.34  | -7.62 ± 8.11   | 93.26 ± 6.77   |

## Section S5. Fibers Stability in PBS

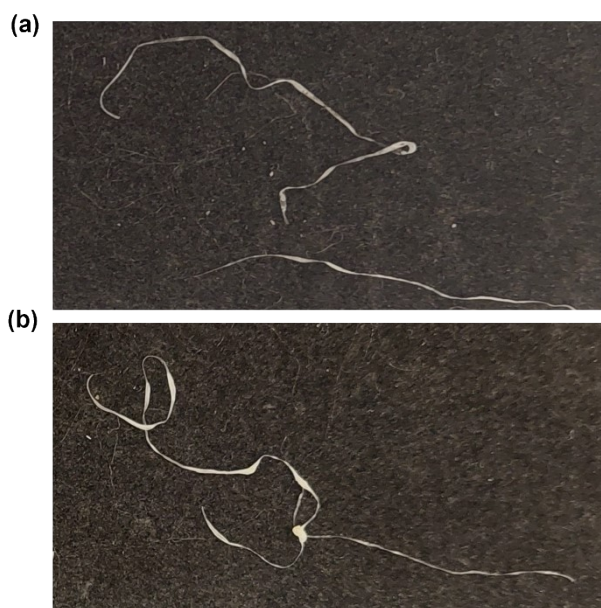
Mass losses of each fiber typology were tracked throughout 28 days of incubation in PBS (Table S5):

**Table S5.** Wet-spun fibers degradation profiles over 28 days of incubation in PBS. Data is presented as average percentage of mass loss  $\pm$  SD (n = 3).

| Fiber           | Mass Loss (%)        |                      |                                                                                      |                                                                                      |                                                                                      |
|-----------------|----------------------|----------------------|--------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------|
|                 | Day 1-3              | Day 3-7              | Day 7-14                                                                             | Day 14-21                                                                            | Day 21-28                                                                            |
| PCL             | $-0.40 \pm 0.10$     | $-48.92 \pm 30.96$   | $-51.30 \pm 25.06$                                                                   | $-65.48 \pm 7.09$                                                                    | $-94.75 \pm 27.33$                                                                   |
| PCL-CLO         | $-35.95 \pm 30.97$   | $-47.56 \pm 28.22$   | $-35.76 \pm 34.63$                                                                   | $-53.16 \pm 30.27$                                                                   | $-43.27 \pm 32.21$                                                                   |
| SA              | $-207.01 \pm 67.13$  | $-378.68 \pm 104.76$ |   |   |   |
| SA-AAPV         | $-647.38 \pm 187.26$ | $-816.61 \pm 33.06$  |  |  |  |
| CA              | $34.26 \pm 7.25$     | $33.46 \pm 13.59$    | $31.03 \pm 18.79$                                                                    | $29.67 \pm 13.12$                                                                    | $19.46 \pm 7.58$                                                                     |
| PCL/SA          | $-153.07 \pm 82.25$  | $-188.31 \pm 77.66$  | $-186.00 \pm 76.57$                                                                  | $-191.55 \pm 72.20$                                                                  | $-224.92 \pm 46.07$                                                                  |
| PCL-CLO/SA      | $-126.13 \pm 20.74$  | $-152.05 \pm 27.84$  | $-122.46 \pm 36.83$                                                                  | $-130.45 \pm 29.08$                                                                  | $-135.26 \pm 44.14$                                                                  |
| PCL/SA-AAPV     | $-197.56 \pm 65.97$  | $-246.34 \pm 84.82$  | $-236.05 \pm 93.41$                                                                  | $-253.33 \pm 97.27$                                                                  | $-292.92 \pm 142.00$                                                                 |
| PCL-CLO/SA-AAPV | $-156.83 \pm 25.44$  | $-180.81 \pm 4.49$   | $-182.76 \pm 19.29$                                                                  | $-160.21 \pm 66.99$                                                                  | $-171.68 \pm 73.35$                                                                  |

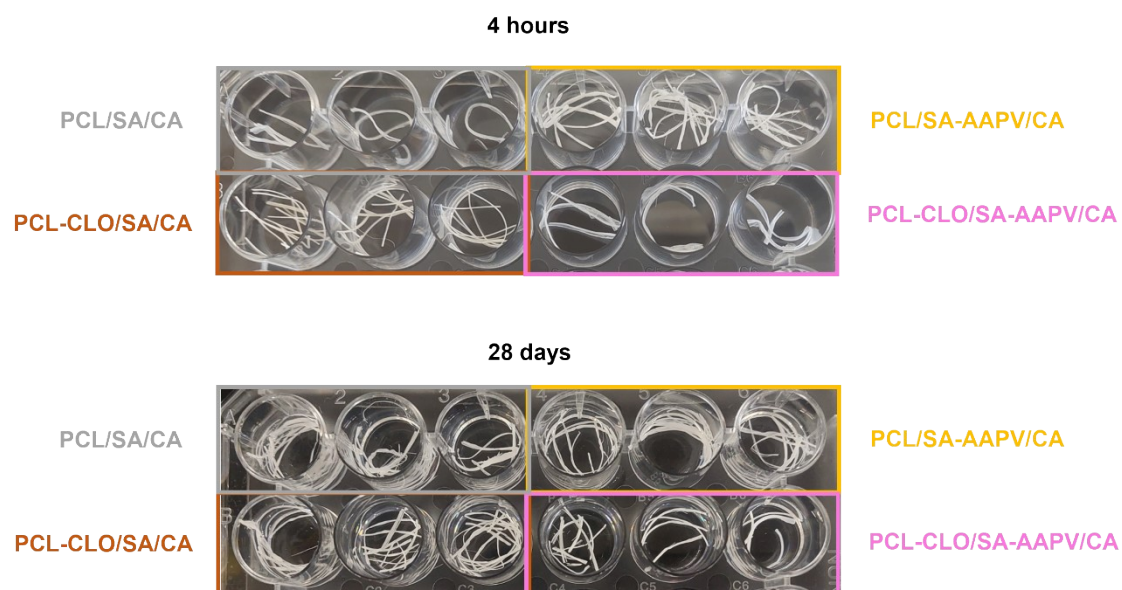
|                           |                    |                    |                    |                    |                    |
|---------------------------|--------------------|--------------------|--------------------|--------------------|--------------------|
| <b>SA/CA</b>              | $-19.12 \pm 51.63$ | $-23.23 \pm 41.71$ | $-8.04 \pm 31.62$  | $-29.32 \pm 26.94$ | $-36.92 \pm 4.44$  |
| <b>SA-AAPV/CA</b>         | $43.22 \pm 9.51$   | $39.38 \pm 13.06$  | $48.51 \pm 2.58$   | $40.92 \pm 21.48$  | $30.55 \pm 26.03$  |
| <b>PCL/CA</b>             | $-2.54 \pm 26.86$  | $-4.02 \pm 31.49$  | $-16.37 \pm 18.09$ | $-8.11 \pm 29.41$  | $-49.44 \pm 34.47$ |
| <b>PCL-CLO/CA</b>         | $-2.50 \pm 17.91$  | $-9.38 \pm 10.24$  | $-16.80 \pm 30.52$ | $-7.67 \pm 10.81$  | $-21.24 \pm 21.44$ |
| <b>PCL/SA/CA</b>          | $3.82 \pm 5.94$    | $8.24 \pm 4.44$    | $5.59 \pm 6.02$    | $11.23 \pm 5.53$   | $-9.61 \pm 4.08$   |
| <b>PCL-CLO/SA/CA</b>      | $-57.39 \pm 68.24$ | $-51.95 \pm 51.82$ | $-19.73 \pm 46.91$ | $-17.14 \pm 28.92$ | $-53.44 \pm 41.25$ |
| <b>PCL/SA-AAPV/CA</b>     | $3.69 \pm 4.72$    | $-2.96 \pm 13.74$  | $1.94 \pm 2.57$    | $-12.41 \pm 17.98$ | $-26.78 \pm 5.91$  |
| <b>PCL-CLO/SA-AAPV/CA</b> | $23.51 \pm 12.47$  | $13.97 \pm 23.08$  | $18.40 \pm 11.13$  | $10.32 \pm 20.60$  | $-11.11 \pm 14.80$ |

Physical images of both SA and SA-AAPV hollow fibers were collected in order to complement the results from this section (Figure S4).



**Figure S4.** Physical images of (a) SA and (b) SA-AAPV hollow wet-spun fibers. Photographs were taken, from a distance of 6.5 cm, and  $3\times$  magnification, with a 64 MP camera of a Samsung Galaxy A72 mobile phone.

CLO/AAPV loaded and unloaded fibers were assessed visually after different periods of incubation in PBS, up to 28 days, to test their structural integrity (Figure S5). Results confirmed that the wet-spun fibers kept their structural integrity until the 28<sup>th</sup> day of incubation, with the exception of SA and SA-AAPV fibers, as described in Section 3.6.



**Figure S5.** Wet-spun fibers (a) prior to their incubation in PBS and (b) after 28 days of incubation in PBS.