

Supplementary Information

Controlling the wettability properties of polyester fibers using grafted functional nanomaterials

Boguslavsky Yonit,^a Fadida Tania,^a Talyosef Yossi^b and Lellouche Jean-Paul*^a

1. Silica NPs synthesis

Silica NPs of 60 nm diameter were prepared using the classical Stöber basic hydrolytic method as follows: to a reaction vessel containing 300 ml of EtOH, 16 ml of ammonium hydroxide solution and 18.9 ml (0.085 mol) of tetraethyl orthosilicate (TEOS) were added. Silica NPs of 150 and 300 nm diameter were prepared by changing the amount of ammonium hydroxide solution and TEOS while the amount of EtOH stayed constant (300 ml). For 150 nm NPs, we used 24 ml of ammonium hydroxide solution and 12 ml (0.054 mol) of TEOS and for 300 nm NPs, we used 30 ml of ammonium hydroxide solution and 12 ml (0.054 mol) of TEOS. The reaction was carried out for 24h with constant magnetic agitation at room temperature. The obtained NPs were used after ammonium hydroxide was evaporated by distillation till obtaining a neutral pH.

2. Oxidation of multi-walled carbon nanotubes (MWCNTs)

100 mg of raw MWCNTs were suspended and heated in 16 ml of concentrated HNO₃/H₂SO₄ (1/1) mixture for 2 h at 70°C, followed by decantation of mixture of acids. Resulting MWCNTs_{ox} were continuously washed with distilled water and centrifuged till neutral pH and then dried in vacuum oven (35°C, 2 mBar, 18 h). The obtained MWCNTs_{ox} were immediately used for further functionalization.

3. 1,3-Diaminopropane coupling to MWCNTs_{ox} (NH₂-functionalized MWCNTs_{ox})

50 mg of oxidized MWCNTs were suspended in 5 ml of deionized water. 2 mg (0.01 mmol) of EDAC and 2 drops of TEA were added to this suspension while stirring. The activation reaction was performed for 30 min. 2 ml of 1,3-diaminopropane was then added to the stirred suspension and the reaction was continued for 2h. The resulting NH₂-functionalized MWCNTs_{ox} were washed by extensive centrifugation cycles with water till achieving neutral pH and then dried in a vacuum oven (35°C, 2 mBar, 18 h).

4. Amine group Quantification (Kaiser Test)

Grafted materials (around 2.0 mg, precisely weighted) were placed in a test tube with 75 µl of solution 1, 75 µl of solution 2 and 100 µl of solution 3. The test tube was placed in a heating block, preadjusted to 100°C, for 3 min. A solution of 60% ethanol in water (4.8 ml) was added to the test tube and stirred well. An aliquot (0.5 ml) of this solution was diluted in 4.5 ml of 60% ethanol in water.

Solution 1: 40 g phenol in 10 ml ethanol

Solution 2: 2.5 g ninhydrin in 50 ml ethanol

Solution 3: 65 mg of KCN is dissolved in 100 ml of water. 2 ml of the KCN solution is diluted with 100 ml pyridine.

The amount of grafted amines was calculated by the following equation using the Beer-Lambert law:

$\text{mmol (NH}_2\text{)/g of PET threads} = (\text{Abs} \times 5 \times 10 \times 10^3) / (1.5 \times 10^4 \times \text{mg of resin})$, where

Abs - Absorbance

$1.5 \times 10^4 - \epsilon$