Supporting Information

Iron Oxide Nanoparticle-containing Main-Chain Liquid Crystalline Elastomer: Towards Soft Magnetoactive Networks

Alfonso Garcia-Marquez, Arnaud Demortière, Benoît Heinrich, Daniel Guillon, Sylvie Bégin-Colin, Bertrand Donnio*

Institut de Physique et Chimie des Matériaux de Strasbourg (IPCMS), UMR 7504, CNRS-Université de Strasbourg, 23 rue du Loess, BP43, 67034, Strasbourg cedex 2, France. Fax: +33 388107246; Tel: +33 3107156; bdonnio@ipcms.u-strasbg.fr

Instrumentation

Microscopic, spectroscopic, and thermal characterization of NPs and hybrid

Evaluation of the soluble content (sol.-%), and swelling behaviour

Characterization of the mesophase by small-angle XRD

Magnetic properties

Synthetic part

Instrumentation

Nuclear magnetic resonance (NMR) (1 H) spectra were recorded on a Bruker AVANCE 300 (300 MHz) spectrometer. The internal references of the spectrum correspond to the peak of the solvent CDCl₃ (ppm).

The optical textures of the mesophases were studied with a Leitz optical polarizing microscope equipped with a Mettler FP80 hot-stage and a Mettler FP80 central processor.

The thermal properties were investigated using a differential scanning calorimetry (DSC) with a TA Instruments DSC-Q1000 instruments operated at scanning rates in a range from 2 to 10°C.min⁻¹.

The TGA measurements were carried out on a SDTQ 600 apparatus at scanning rate of 10°C min⁻¹, under air.

The oriented XRD patterns were recorded by using a pinhole Cu-K α ($\lambda = 1.542$ Å) parallel beam from a sealed-tube generator (900 W) equipped with a home-mounted Kirkpatrick-Baez optics, the detector to sample distance being 107.3 mm. Phosphor imaging screens were used for detection, scanned with a Storm820 scanner of Molecular Dynamics and analyzed using homemade ImageJ plug-ins. The sample was oriented by shearing and introduced in a 1 mm Lindemann capillary. During the experiment, the temperature was controlled within ±0.03°C.

The magnetic characterization and measurements of the frequency-dependent AC susceptibility of the sample were performed with a Superconducting Quantum Interference Device (SQUID) magnetometer (Quantum Design, MPMS XL), at temperatures between 5K and 300K under a field of 75 G. The applied field can reach a maximum value of 5 T. The sample was contained in a small gelatin capsule, which was inserted into the SQUID chamber by means of a plastic straw. The temperature variation of χ ' and χ '' were measured at fixed frequencies between 1Hz and 1kHz.

Transmission electron microscopy (TEM), a TOPCON 002B, operating at 200 kV, (point resolution 0.18 nm) and equipped with a GATAN GIF 200 electron imaging filter, has been used to study the morphology and crystalline structure of nanoparticles.

Microscopic, spectroscopic, and thermal characterization of NPs and hybrid

Morphology, size, dispersity of NP@OA

The nanoparticles (NPs) coated by oleate groups (OA) were characterized by transmission electron microscopy (TEM). TEM observations at high magnification (Figure S1a) revealed NPs with a narrow size distribution and quasi-spherical shape. The average diameter of the NPs, evaluated on a population of c.a. 400 NPs, was estimated to 3.3 nm with a polydispersity of 7% (Figure S1b, the polydispersity being defined as $\sigma/\langle D \rangle$). The Selected Area Electron Diffraction (SAED) pattern (Figure S1c) exhibits clearly six diffraction rings associated to the *hkl* reflections of the spinel structure and can be indexed as indicated on the figure. Conventional characterization techniques show that the NPs have a composition close to that of maghemite as usually observed with NPs with so small sizes (Reference 17).



Figure S1. 3.3 nm iron oxide NP@OA: a) TEM image, b) SAED pattern, c) size distribution.

Spectroscopic characterization of NP@CL





Thermal decomposition of the coated NPs and elastomer (TGA)

Figure S3. TGA-TDA curves of NP@OA (top), NP@CL (middle), NP@LCE (bottom).

Determination of the density of grafting on the surface of the nanoparticle

The average diameter was determined by transmission electron microscopy (see above). The diameter is 3.3 nm with a polydispersity of 23% and the available surface has been calculated considering quasi-spherical nanoparticles ($S_{NP} = 4\pi (\langle D \rangle/2)^2$). Thus, the number of OA chains adsorbed to the NP surface, N_{OA} , can be estimated by the average cross-section area of S_{OA} chains (20 Å²) as $N_{OA} = S_{NP}/S_{OA}$. The number of ligands per particle was determined from the TGA. For the TGA measurements, we made the hypothesis that the event occurring at ca 300°C corresponds to the degradation of the oleate chains, and to cross-linker (at ca 380°C, TDA). TGA measurements permit the estimation of the ratio of the organic shell and inorganic core, and thus the number of molecules grafted. The ratio are 42.25:57.75 OA:NP for NP@OA, 21.0:22.6:56.4 OA:CL:NP for NP@CL and 87.5:12.5 LCE:NP for NP@LCE. The number of chains (OA) and cross-linker (CL), n_i , per particle, NP, can obtained by the general equation: $n_i = (\% i / \% NP) \times (M_{NP} / M_i)$, where the % are deduced from TGA and M_i and M_{NP} are the molecular weights of the ligands ($i = OA = 281.45 \text{ g.mol}^{-1}$; $CL = 290.40 \text{ g.mol}^{-1}$) and of the NP ($M_{NP} = 62181 \text{ g.mol}^{-1}$) i.e. Z×Fe₂O₃×N_{cell}, where Z = 12 and N_{cell} is the number of elementary cells within the NP. Both calculations converge towards similar values, and are presented in the table below.

| | <u> </u> | | . / | | |
|---------|-------------|-----------------------|-----------------|-----------------------|--|
| Samples | n_i (TEM) | N _{OA} (TGA) | N _{CL} | $/n_i*$ | |
| NP@AO | 170±5 | 160±5 | - | 20±1.5 Å ² | |
| NP@CL | | 80±10 | 85±10 | $20\pm 2 \text{ Å}^2$ | |

Table S1. Average repartition of the ligands (i = OA, CL) adsorbed on the NP' surface.

* Average cross-section area per ligand adsorbed ($\langle S \rangle = S_{NC}/n_j$, $n_j = N_{OA}+N_{CL}$).

Thermal behaviour of the NP@LCE (DSC)



Figure S4. DSC traces of both heating/cooling (rate of 2 °C.min⁻¹) for the elastomer with 12.5% w/w of functionalized 3.3 nm-diameter ferrite NP (NP@LCE).

Table S2. Thermal behaviour (DSC data) for the hybrid liquid crystalline elastomer (compared to that of the closely related organic LCE with 4% w/w HD5 cross-linker*).

| Elastomer | Thermal Behavior |
|--------------|-------------------------------------|
| NP@LCE | X 16.5 [8.75] Smectic 34.3 [10.5] I |
| LCE (4% HD5) | X 16.4 [4.6] SmC 41.8 [10.3] I |

Transition temperatures (onset) are expressed in °C with the corresponding latent heats ΔH in brackets in [J.g⁻¹]. Temperatures were estimated as the mean of heating and cooling maxima of the 2 cycles with a rate of 2°C/min. X: unidentified phase; SmC: smectic C mesophase, I: isotropic state. * Data taken from PhD thesis, CNRS-University of Strasbourg, Alfonso Garcia Marquez, 2009.

Evaluation of the soluble content (sol.-%), and swelling behaviour

Soluble content (sol.-%), swelling degree, q, and swelling anisotropy for the hybrid elastomer were measured to evaluate the efficiency of the cross-linking reaction.

The quantity of the soluble content, i.e. corresponding to the non-cross-linked parts, e.g. unreacted monomers, free oligomers, etc.., of the obtained gel was performed for 3 days in a soxlet, using cyclohexane as solvent. The cleaned gel was then dried under vacuum for 3 days. The soluble content was calculated with the following equation:

$$\operatorname{sol.-}{}^{\circ}_{o} = V_{initial} - V_{final},$$

where $V_{initial}$ is the initial weight of the dried elastomer and V_{final} is the weight of the dried elastomer after washing and removal of soluble species.

Swelling experiment was performed in two solvents (heptane and toluene), starting with heptane and finishing with pure toluene. The degree of swelling was calculated with the following equation:

$$q = V_{swollen} / V_{dry},$$

in which q is the swelling factor, $V_{swollen}$ is the volume of the swollen gel and V_{dry} is the initial volume of the dry gel.

The swelling ratio in a given direction of the gel is expressed by the equation:

$$\alpha_i = l_i \, swollen / l_i \, dry,$$

where α_i is the swelling ratio in the *i* direction, and *l* is the size in the swollen dry state. In the case of bulk elastomers, the direction parallel to the director $(l_{//})$ was arbitrarily defined as the height of the initial gel and the perpendicular one (l_{\perp}) was defined as the width. Therefore, $\alpha_{//}$ is the swelling coefficient parallel to the director and α_{\perp} is the swelling coefficient parallel to the director that the swelling in the two perpendicular directions is the same, the degree of swelling can be rewritten as:

$$q = \alpha_{//} \times \alpha_{\perp} \times \alpha_{\perp}$$

In order to determine the difference of swelling as a function of the direction, the swelling anisotropy (Δq) can be determined by:

$$\Delta q = \alpha_{\perp} / \alpha_{//}$$

Table S3. Soluble content, sol.-%, swelling coefficients, $\alpha_{//}$ and α_{\perp} , degree of swelling, q, and swelling anisotropy, Δq .

| sol% <i>bulk</i> (±1) | $\alpha_{//} (\pm 0.5)$ | $\alpha_{\!\perp}(\pm 0.5)$ | q <i>bulk</i> (±0.5) | Δq (±0.5) |
|-----------------------|-------------------------|-----------------------------|----------------------|-----------|
| 12 | 0.79 | 3.05 | 7.35 | 3.86 |



Figure S5. Picture of the gel formed by NP@LCE.

Characterization of the mesophase by small-angle XRD



Figure S6. X-ray diffraction pattern of NP@LCE oriented by shearing. The direction of shearing is parallel to the meridian.



Figure S7. Radial profile over 360° of the pattern of NP@LCE. Black labels: reflections of the smectic layering (d = 27.2 Å); red label: periodicity within the siloxane sublayer ($h_{sil} \sim$

6.24 Å, $\xi \sim 17$ Å [Scherrer], $\sigma_{sil} = (h_{sil}/0.9763)^2 \sim 40.8$ Å²); green label: periodicity within the aliphatic sublayer ($h_{ch} \sim 4.48$ Å, $\xi \sim 18$ Å [Scherrer], $\sigma_{ch} = (h_{ch}/0.9763)^2 \sim 21.0$ Å²).



Figure S8. Azimuthal profiles of the pattern of NP@LCE, $\psi = 0^{\circ}$, $\pm 180^{\circ}$ corresponding to the direction of shearing. Black line: 1st order smectic layer reflection ($\psi = 0^{\circ}$, FWHM = 35°); red line: periodicity within the siloxane sublayer ($\psi = 100 \pm 10^{\circ}$, FWHM = 110°); green line: periodicity within the aliphatic sublayer ($\psi = 50 \pm 10^{\circ}$, $\psi = 130 \pm 10^{\circ}$, FWHM = 80°).

Magnetic Properties



Figure S9. Magnetic susceptibility χ' component vs. temperature for 3.3 nm iron oxide NP@OA.



Figure S10. Magnetic susceptibility χ '' component vs. temperature for 3.3 nm iron oxide NP@OA.



Figure S11. $ln(\tau)$ vs. $1/T_w$ with the fit performed from the Néel-Brown model for 3.3 nm iron oxide NP@OA.



Figure S12. Magnetic susceptibility χ ' component vs. temperature for the NP@LCE.



Figure S13. Magnetic susceptibility χ '' component vs. temperature for NP@LCE.

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Figure S14. $ln(\tau)$ vs. $1/T_w$ with the fit performed from the Néel-Brown model for NP@LCE.

Synthetic part

Materials

4-benzyloxybenzoic acid (99 %), methyl 4-hydroxybenzoate, 4-benzyloxyphenol (99 %), oleic acid (99%), 4-dimethylaminopyridine (DMAP, 99 % pure) were purchased from Alfa-Aesar. Undecenol, 4-vinyloxybutanol (99 %), triphenylphosphine (PPh₃, 99 % pure), diisopropyl azodicarboxylate (DIAD, 94 % pure) were obtained from Acros. Octyl ether (97%, b.p. 287°C) purchased from Fluka. 1-ethyl-3-(3was dimethylaminopropyl)carbodiimide hydrochloride (EDCI), FeCl₃.6H₂O (97%) and PtCODCl₂ (98 % pure) were purchased from Aldrich. Pd/C catalyst was obtained from Lancaster (99 % pure). 1,1,3,3,5,5-hexamethyltrisiloxane (HTMS) was purchased from ABCR. Sodium oleate (97%) was purchased from TCI.

Dimethylformamide (DMF) was obtained from Normapur (99.8 % pure). Hexane and cyclohexane were obtained from Carlo-Erba (>99.9 % pure). Methanol (MeOH), obtained from Carlo Erba (>99.9 % pure), was dried over magnesium turnings and iodine before distillation. Dichloromethane (CH₂Cl₂), obtained from Carlo Erba (stabilized with amylene, 99.8 % pure), was distilled over calcium chloride prior to chemical reactions and N₂-chromatography columns. Tetrahydofuran (THF), obtained from Ridel-de-Haën (> 99 % pure), was dried over potassium hydroxide, and distilled over sodium wire and before use. Toluene, purchased from Alfa Aesar (99.5 % pure), was first extracted with concentrated sulfuric acid until the acidic phase was colorless, then washed with brine and dried over sodium sulfate and finally distilled over sodium wire and stored over 3Å molecular sieves. HCl was obtained from Normapur (37 % pure). All other chemicals were used without purification.

Synthesis

4-(Benzyloxy)phenyl 4-(benzyloxy)benzoate



In a three necked round bottom flask, 3.00 g of 4-benzyloxybenzoic acid, 0.826 g of 4benzyloxyphenol and 0.214 g de DMAP were dried in vacuum with a thermal gun and then dissolved in 100 mL of dry dichloromethane. Argon was added and the emulsion cooled to 0°C. 3.850 g EDCI were added and the reaction mixture was magnetically stirred for 5h. The resulting solution was extracted with brine (2x200 mL). The organic phase was then recovered and concentrated in vacuum. The residue was purified by silica column (dichloromethane) to yield 2.9 g of a white solid (67 %).

¹H-NMR (300 MHz, CDCl₃): δ (ppm): 5.07(2H, s, PhC<u>H</u>₂), 5.17 (2H, s, PhC<u>H</u>₂), 7.00 (2H, d, *m*-Ar, *J*=8.5 Hz), 7.05 (2H, d, *m*-Ar', *J*=8.9 Hz), 7.12 (2H, d, *o*-Ar', *J*=8.9 Hz), 7.39 (10H, m, <u>Ar</u>CH₂), 8.15 (2H, d, *o*-Ar, *J*=8.5 Hz). ¹³C-NMR (75 MHz, acetone-*d*₆): δ (ppm): 70.2 (Ph<u>C</u>H₂), 164.7 (COOAr), 163.0 (ArOBn), 159.7 (Ar'OBn), 145.3 (Ar'OCO), 134.0 (2C, *o*-ArH), 123.7 (2C, *o*-Ar'H), 122.0 (ArCOO), 118.6 (2C, *m*-Ar'H), 115.9 (*m*-Ar'H) ppm.

4-Hydroxyphenyl 4-hydroxybenzoate



In a 500 mL two necked round bottom flask, 2.00 g of 4-(benzyloxy)phenyl 4-(benzyloxy)benzoate were dissolved in 250 mL of dry THF and the solution flushed with argon. 25 mg of palladium in charcoal (10%) were slightly added and placed in a specially designed hydrogen system. The mixture was stirred at room temperature until the starting product was consumed. The black suspension was filtered on Celite \mathbb{R} and the solvent evaporated in vacuum to yield 1.2 g of a white solid (90%).

¹H-NMR (300 MHz, acetone-*d*₆): δ (ppm): 6.93 (2H, d, *m*-Ar, *J*=8.5 Hz), 7.07 (2H, d, *m*-Ar', *J*=8.9 Hz), 7.23 (2H, d, *o*-Ar', *J*=8.9 Hz), 7.95 (2H, s, ArOH), 8.03 (2H, d, *o*-Ar, *J*=8.5 Hz). ¹³C-NMR (75 MHz, acetone-*d*₆): δ (ppm): 115.9 (2C, *m*-Ar'H), 118.6 (2C, *m*-Ar'H), 118.9 (ArCOO), 123.7 (2C, *o*-Ar'H), 134.0 (2C, *o*-ArH), 145.3 (Ar'OCO), 155.8 (Ar'OH), 164.1 (ArOH), 165.0 (COOAr).

4-(4-(Vinyloxy)butoxy)phenyl 4-(4-(vinyloxy)butoxy)benzoate



In a three-necked round bottom flask with a stirring bar, 1.00 g (4.35 mmol) of 4-

hydroxyphenyl 4-hydroxybenzoate and 2.74 g (10.43 mmol) of triphenylphosphine were mixed. An argon flush was performed and the solids were dissolved in 150 mL of anhydrous THF. The mixture was stirred and cooled down to 0°C. Then 1.90 mL (10.43 mmol) of 4immediately, 1.40 mL vinyloxybutanol were added and (9.13)mmol) of diethylazodicarboxilate (DIAD) were added dropwise. The reaction mixture was kept to 0°C for 1 h and left to warm up to room temperature for 8 h. The resulting solution was concentrated to dryness and redissolved in a minimum quantity of a mixture of dichloromethane-cyclohexane (1:3) and purified by triethylamine-neutralized silica gel. The crude product was finally recrystalized from anhydrous methanol to yield white crystals (80 %). Cr 72 (38.2) SmC 87.0 (1.3) I.

Elemental analysis for C₂₅H₃₀O₆ (MW = 426.5 g.mol⁻¹): Calcd: C% 70.40, H% 7.09; Found: C% 70.42, H% 6.91. ¹H-RMN (300 MHz, CDCl₃), δ (ppm): 1.88 (8H, m, C<u>H</u>₂-CH₂O), 3.77 (4H, t, ³*J*=5.9 Hz, CH₂O), 4.02 (2H, dd, ^{2gem}*J*=2.0 Hz; ^{3cis}*J*=6.0 Hz, C<u>H</u>₂=CH-O), 4.10 (4H, t, ³*J*=6.1 Hz, CH₂OAr), 4.20 (2H, dd, ^{2gem}*J*=1.3 Hz; ^{3trans}*J*=14.5 Hz, C<u>H</u>₂=CH-O), 6.50 (2H, dd, ^{3cis}*J*=6.8 Hz; ^{3trans}*J*=14.3 Hz, CH₂=C<u>H</u>-O), 6.92 (2H, d, ^{HH}*J*=6.8 Hz, *m*-Ar), 6.99 (2H, d, ^{HH}*J*=6.8 Hz, *o*-Ar), 8.14 (2H, d, ^{HH}*J*=7.8 Hz, *m*-Ar'), 8.17 (2H, d, ^{HH}*J*=7.9 Hz, *o*-Ar'). ¹³C-NMR (75 MHz, CDCl₃) δ (ppm): 25.6 (CH₂), 25.7 (CH₂), 25.8 (CH₂), 25.9 (CH₂), 67.4 (<u>CH₂OCH=CH₂), 67.5 (<u>CH₂OCH=CH₂), 67.7 (CH₂OAr'), 67.8 (CH₂OAr), 86.4 (CH=<u>C</u>H₂), 86.5 (CH=<u>C</u>H₂), 114.2 (*m*-ArH), 115.0 (*m*-Ar'H), 121.8 (ArCOO), 122.5 (*o*-Ar'H), 132.2 (*o*-ArH), 144.4 (Ar'OCO), 151.8 (<u>C</u>H=CH₂), 156.6 (Ar'OCH₂), 163.2 (ArOCH₂), 165.2 (COOAr).</u></u>

Methyl 4-(undec-10-enyloxy)benzoate



In a 500 mL round-bottom flask, 10.00 g (65.7 mmol) of methyl 4-hydroxybenzoate, 22.41 g (85.4 mmol) of PPh₃ and 13.43 g (78.9 mmol) of undecenol were dissolved in 250 mL of anhydrous THF. The mixture was magnetically stirred and cooled down to 0° C for 15 minutes and then 14.6 mL (65.7 mmol) of DIAD were added dropwise. After the addition, the mixture was left to react for 12h. The solvent was dried and the remaining solid was purified by column chromatography (SiO₂, dichloromethane-cyclohexane 60:40) to yield white crystals, 15.2 g (76 %).

¹H-RMN (300 MHz, CDCl₃) δ (ppm): 1.31 (12H, m, CH₂), 1.80 (2H, m, ³*J*=6.3 Hz, CH₂),

2.04 (2H, q, ³*J*=6.2 Hz, CH₂) 3.89 (3H, s, CH₃O), 4.00 (2H, t, ³*J*=6.4 Hz, CH₂O) 4.96 (2H, m, CH₂=CH), 5.82 (1H, m, CH₂=C<u>H</u>), 6.90 (2H, d, ^{HH}*J*=8.8 Hz, *m*-Ar), 7.98 (2H, d, ^{HH}*J*=8.8 Hz, *o*-Ar).

4-(Undec-10-enylbenzoic) acid (CL)



In a 500 mL round-bottom flask, 10.4 g (34.1 mmol) of the methyl ester were dissolved in 300 mL of ethanol. After total solubilization, 2.7 g (48.1 mmol) of potassium hydroxide dissolved in 40 mL of water were added. The reaction mixture was set at reflux for 15 h. After reaction completion, the solvent was evaporated and the remaining solid redissolved in 500 mL of water. The resulting solution was acidified until reaching pH=2 to obtain a white solid which was filtered and then crystallized in methanol (7.80 g, 79%).

¹H-RMN (300 MHz, CDCl₃) δ (ppm): 1.35 (12H, m, CH₂), 1.80 (2H, m, ³*J*=6.3 Hz, CH₂), 2.06 (2H, q, ³*J*=6.2 Hz, CH₂), 4.02 (2H, t, ³*J*=6.2 Hz, CH₂O) 5.03 (2H, m, C<u>H₂</u>=CH), 5.83 (1H, m, CH₂=C<u>H</u>), 6.93 (2H, d, ^{HH}*J*=8.8 Hz, *m*-Ar), 7.94 (2H, d, ^{HH}*J*=8.8 Hz, *o*-Ar).

Iron oleate complex (Fe(OA)₃)

7.37 g (27.33 mmol) of FeCl₃.6H₂O were dissolved in 60 mL of H₂O (Milli-Q) and 80 mL ethanol. Then, 25 g (82 mmol) of sodium oleate dissolved in 140 ml hexane were mixed with the iron (III) solution. The resulting biphasic solution was refluxed at 70°C for 4 h and kept under stirring. When the reaction was completed, the solution was cooled and then the organic phase containing the iron oleate complex was separated (separatory funnel), washed three times with 30 mL distilled water to extract salts, dried using MgSO₄, and finally hexane was evaporated. The resulting iron oleate complex was a reddish-brown viscous solution. The final product has been stored at a temperature of 4°C.

Iron oxide nanocrystals, NP@OA

The synthesis of iron oxide nanocrystals has been carried out by modification of synthetic parameters of published procedures. 2 g $(2.2 \times 10^{-3} \text{ mol})$ of Fe(OA)₃ and 0.155 g $(0.55 \times 10^{-3} \text{ mol})$ of oleic acid were added to 20 ml octyl ether. The mixture was kept under vigorous stirring for 1 h to dissolve the reactants. The solution was heated to 290 °C with a heating rate of 5 °C/min. without stirring and was refluxed for 60 min at this temperature under air. The resultant black solution was then cooled down to room temperature and the nanocrystals were

washed 3 times by addition of ethanol and centrifugation (4000 rpm, 10 min.). This step allowed us to discard the supernatant containing excess oleic acid and reaction by-products. The nanocrystals could be easily suspended in various organic solvents (toluene, hexane, dichloromethane).

Iron oxide nanocrystals, NP@CL

50 mg of **NP@OA** and 100 mg (0.34 mmol) of 4-(undec-10-enylbenzoic) acid were dissolved in dry dichloromethane and refluxed for 3 days. The resulting dispersion was first cooled down at 0 °C and then the solvent was evaporated in vacuum. The residue was redissolved in dry dichloromethane and passed through a Biorad SX1 column using CH_2Cl_2 as eluent. The functionalized nanocrystals were easily suspended in toluene and dichloromethane.

MC-LCE containing ferrite nanoparticles as crosslinking agents, NP@LCE

In a 3 mL vial, 390 mg (0.9 mmol) of 4-(4-(vinyloxy)butoxy)phenyl 4-(4-(vinyloxy)butoxy)benzoate, 208.5 mg (1.00 mmol) of 1,1,3,3,5,5-hexamethyltrisiloxane and 10 mg (~ $3.4x10^{-3}$ mmol) of functionalized ferrite nanoparticles (**NP@CL**) were dissolved in 1.00 mL of thiophene-free toluene. The mixture was put in an ultrasonic bath for 5 min and then heated until a homogeneous, orange-brown solution was obtained (at 40°C). Then, 20 µL of Karstedt catalyst (2 % wt in xylenes) were added and the reaction mixture was magnetically stirred at 300 rpm until a gel was obtained (24 h). The gel was put out of the vial and washed with a Soxhlet apparatus with cyclohexane for 72h. The washings were concentrated obtaining a percentage of soluble content of 12 %.