Electronic Supplementary Material (ESI) for RSC Advances.

Phosphorus pentoxide as a cost-effective, metal-free catalyst for ring opening polymerization of ε-caprolactone

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Supporting Information available

1.NMR analysis

a- Conversion calculations

The 1H NMR analysis of the crude samples in $CDCl_3$ allows to evaluate the conversion by comparing the signals of protons –CH2 ε ' of ε-CL and those ε of PCL; according to the formula

$$\text{Conv}\ (\%) = [A\varepsilon / A\ (\varepsilon + \varepsilon')] * 100.$$

In a second step, the products purified by precipitation in cold methanol and dried at room temperature in the vacuum oven were analyzed by 1 H NMR in $CDCl_3$ and analysed by SEC in THF to obtain their mass distribution.

b- 13C NMR Spectra of PCL

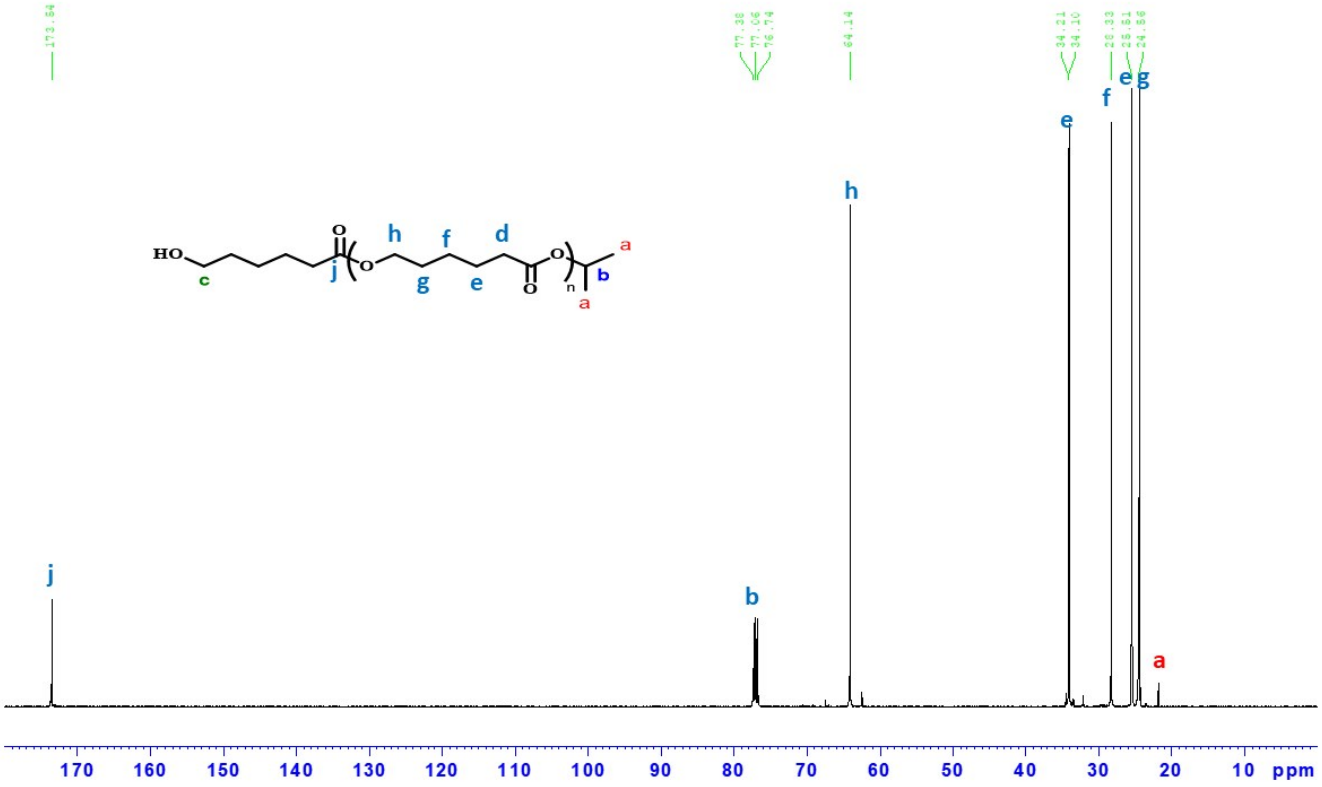

Figure 1. ^{13}C NMR spectra of the obtained PCL in $CDCl_3$ solvent

c- 1H NMR spectras of chain extension experiment of CL polymerization

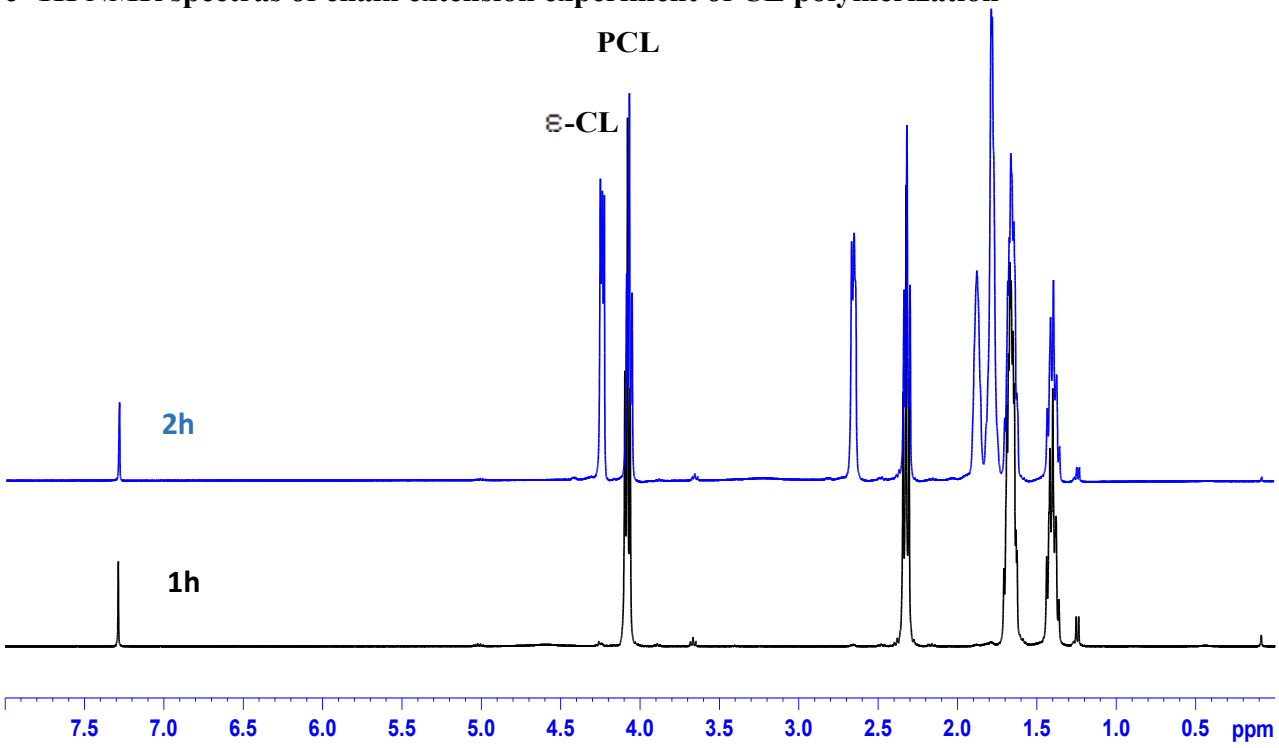

Figure 2: 1H NMR spectrums of PCL in $CDCl_3$ after 100% conversion of $[\varepsilon\text{-CL}]_0/[P_2O_5]_0/[\text{iPrOH}]_0$ = 50/1/1 (black line) and after 50% conversion of 100 equivalents of ε–CL compared to isopropanol and P_2O_5 (blue line)

2. SEC Analysis of chain extension experiment of CL polymerization

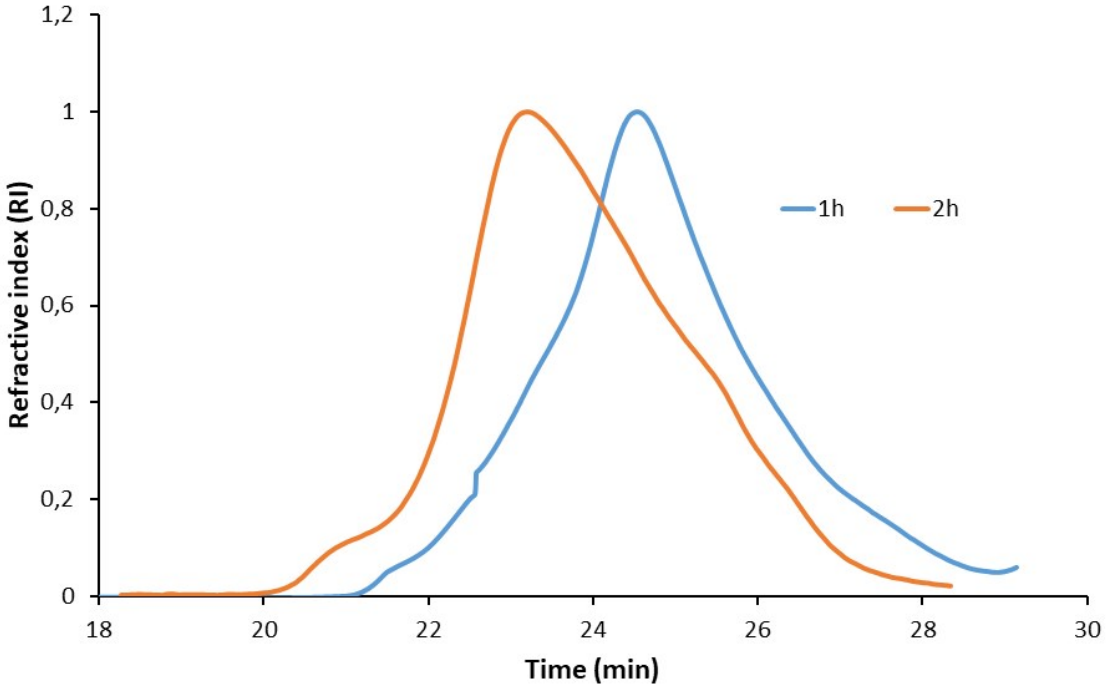

Figure 3: SEC curves of PCL made with P_2O_5/iPrOH , orange line final product (*Mn* = 11800 g mol^{-1}) and blue line prepolymer (Mn = 7505 $g.mol^{-1}$)

3. Thermal properties

a- Differential Scanning Calorimetry (DSC) Analysis:

The tests were carried out using a Perkin Elmer Jade DSC under a stream of Gas Nitrogen purge flow: 20.0 ml/min, with a temperature ramp of 10 °C/min. The samples used have a mass of between 7 -10 mg analysed in a temperature range of -60°C to 100 °C.

b- Thermogravimetric analysis (TGA):

The TGA was performed to determine the thermal stability of polymer materials. TGA was performed by using the TA Instruments Q500 analyser at heating rate of 10 °C min^{-1} under nitrogen atmosphere (80 cm^3 min^{-1}).

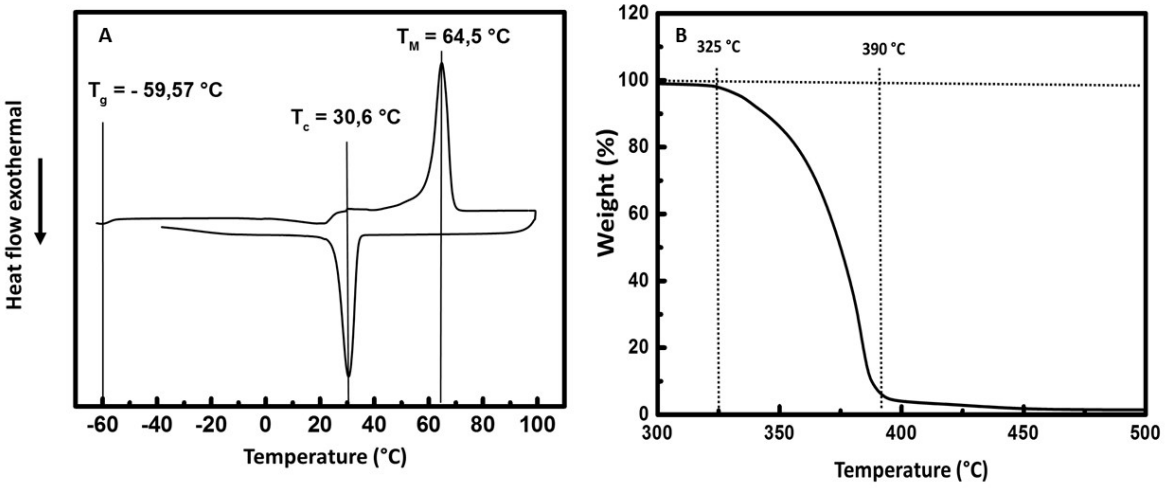

Figure 4: A: DSC curve of PCL prepared by ROP of ε-CL (at 100 °C in bulk, $[CL]_0/[P_2O_5]/[iPrOH]$=200:1:1) with the conv. of 98% and (b) TGA thermograms of PCL