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PREPARATION AND CHARACTERIZATION OF HYDROPHOBICALLY MODIFIED HYDROXYPROPYLCELLULOSE

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The preparation of hydrophobically modified water-soluble polymers is an interesting subject for different applications (1,2). That modification can be attained by the introduction of long alkyl side chains, leading to particular cases of comb-type polymers (3). This kind of polymers is known to be able to give side-chain crystallization when the length of the side chains exceeds about eight carbon atoms.

In this work we report the preparation and characterization of hydroxypropyl-cellulose, HPC, modified with palmitoyl chloride, CH_3-$(CH_2)_{14}$-COCl. The degree of modification was determined by ^{1}H-NMR. Two of the spectra are shown in figure 1 and the results are presented in Table I. The different samples were characterized by DSC and X-ray diffraction, employing synchrotron radiation (at HASYLAB, Hamburg, Germany). The DSC results are also shown in Table I.

Table I. Degree of modification and thermal data of the different samples

Sample	% modification	T_m (°C)	Total)H (J/g)
HPC	0	-	-
PLHPC1	11	-	-
PLHPC2	26	-1	30
PLHPC3	50	29	61

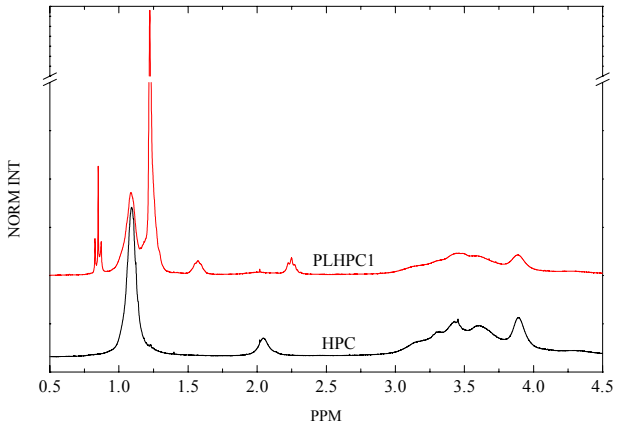

Fig. 1: ^{1}H-NMR spectra of the indicated samples.

Figure 2 shows the DSC curves (second melting) of the different samples. The melting peak corresponding to the side-chain crystallization is clearly observed already in sample PLHPC2, although it melts below room temperature. Regarding sample PLHPC3, with the highest degree of modification, its peak melting temperature is 29°C, of the order of that found for atactic poly-1-hexadecene, PHD (4). It has to be considered that the side chains are of comparable length in the two cases.

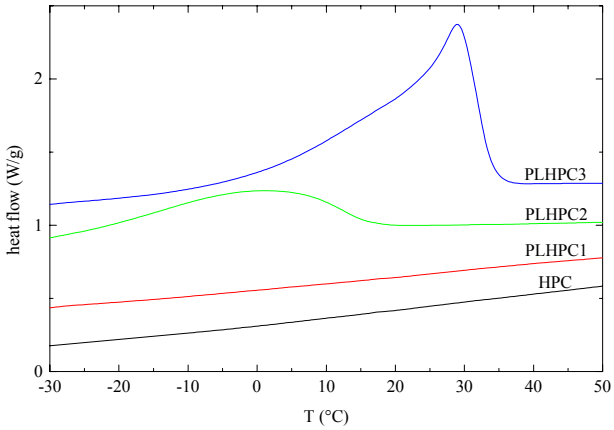

Fig. 2: DSC curves (second melting) of the different samples.

The synchrotron profiles at both middle- (MAXS) and wide-angle X-ray scattering (WAXS) corresponding to sample PLHPC3 in a melting experiment are shown in figure 3. A small amount of crystallinity, around 20%, is deduced from the WAXS diffractogram at room temperature. The MAXS region reveals a diffraction at 3.07nm (and its second order at 1.538nm) again very close to the values found for PHD (5). By rising the temperature, the sample is completely molten at 31°C (in perfect agreement with the DSC results), leaving the two wide peaks (in the WAXS and MAXS regions) characteristic of molten comb-like polymers (6).

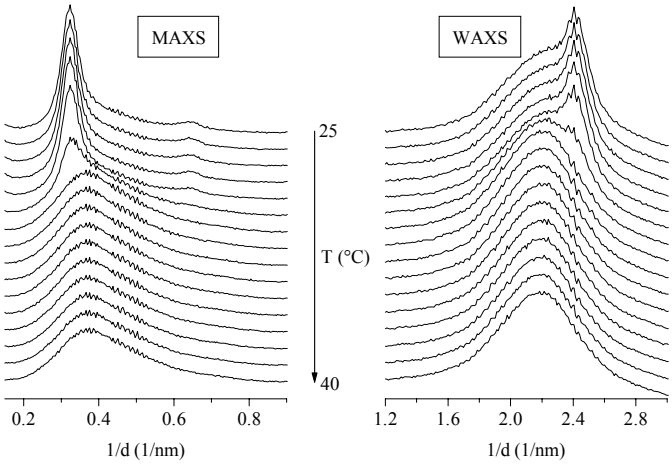

Fig. 3: MAXS/WAXS synchrotron profiles corresponding to sample PLHPC3 in a melting experiment.

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References:

1. W. Marconi, S. Cordelli, A. Napoli, A. Piozzi, *Macromol. Chem. Phys.* 200, 1191 (1999)
2. O. Marstokk, J. Roots, *Polymer Bull.* 42, 527 (1999).
3. N.A. Platé, U.P. Shibaev, "*Comb-shaped polymers and liquid crystals*", J.M.G. Cowie ed., Plenum Press, New York, 1987.
4. B. Peña, J.A. Delgado, E. Pérez, A. Bello, *Makromol. Chem., Rapid Commun.* 13, 447 (1992).
5. B. Peña, J.A. Delgado, A. Bello, E. Pérez, *Polymer* 35, 3039 (1994).
6. A. Turner-Jones, *Makromol. Chem.* 71, 1 (1964).