

Supporting Information

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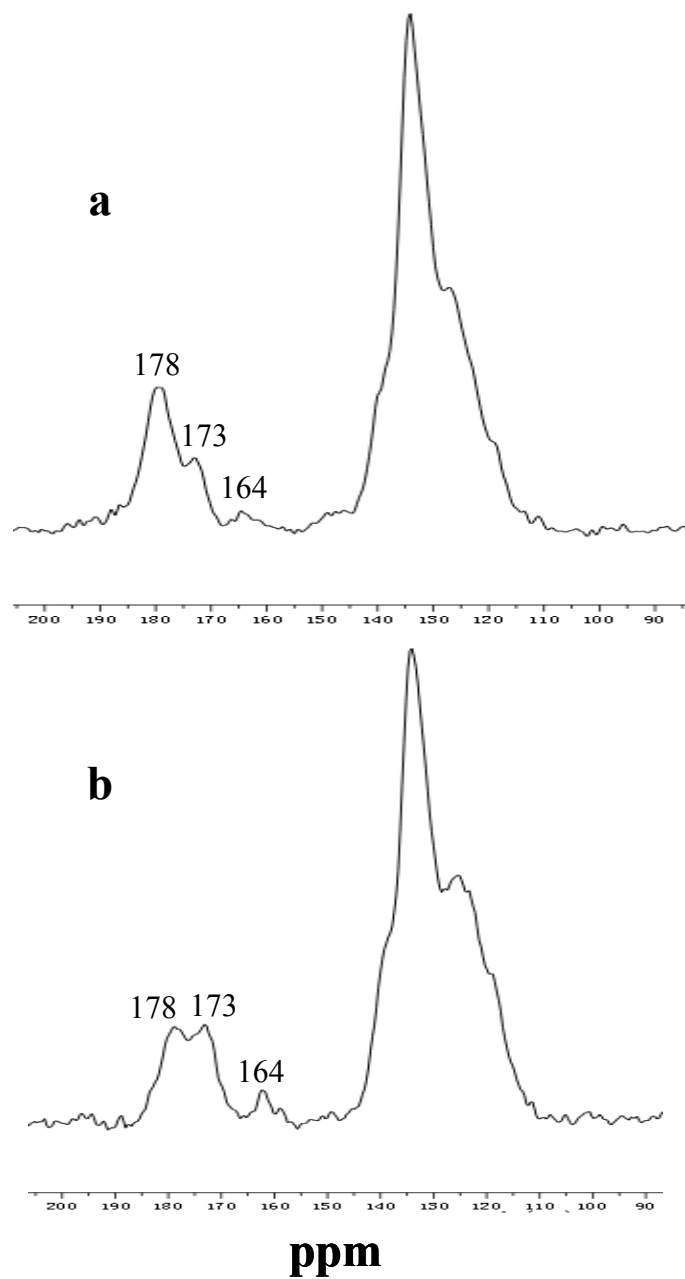
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The reaction precipitate between L-Leu-OtBu and CPAPhS in different synthesis condition was also examined by ¹³C CP/MAS NMR spectra. SIF 1a (Supporting Information Figure 1a) showed the ¹³C CP/MAS NMR spectrum of L-Leu-OtBu-APAPhS, which was synthesized by mixing 0.2g CPAPhS and 0.4g L-Leu-OtBu in anhydrous DMF and the suspension was stirred at 50 °C for 3h. The resonance peak at 173 ppm, which assigned to carbon species of carbonyl in carboxylic acid -C(=O)OH, decreased compared with ¹³C CP/MAS NMR spectrum of CPAPhS (Figure 2a). The ¹³C CP/MAS NMR spectrum of L-Leu-APAPhS showed that the resonance peak at 173 ppm increased compared with L-Leu-OtBu-APAPhS after released with dilute trifluoroacetic acid (TFA) (50% TFA in CH₂Cl₂) (SIF 1b). A resonance peak at 164 ppm, which assigned to carbon species of carbonyl in amide formed between carboxyl groups in CPAPhS and NH₂ groups in L-Leucine, was also observed in ¹³C CP/MAS NMR spectra of L-Leu-OtBu-APAPhS and L-Leu-APAPhS (SIF 1). Apparently, grafting yield of L-Leu-OtBu in the reaction precipitate of heated for 3h (SIF 1) was lower than in the reaction precipitate of heated for 6h (Figure 2). But, the same behaviors of ¹³C CP/MAS NMR spectra were observed in L-Leu-OtBu-APAPhS and L-Leu-APAPhS of different synthesis condition. The ¹³C CP/MAS NMR spectra of L-Leu-OtBu-APAPhS and L-Leu-APAPhS in different synthesis condition further indicated that the two-dimensional molecular space with

L-Leucine (layered L-Leu-APAPhS) was successfully synthesized by grafting L-Leucine in layered CPAPhS.



SIF 1. ^{13}C CP/MAS NMR spectra of (a) L-Leu-OtBu-APAPhS formed at 50 °C for 3h, (b) L-Leu-APAPhS (cleavage precipitates from a)