Supplementary Material (ESI) for Journal of Materials Chemistry

Poly-L-lysine grafted onto silica Mesoporous (SBA 15) synthesized by NCA polymerization and click chemistry

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Supporting Information

Synthesis

Synthesis of α,ε-dicarbobenzoxy-l-Lysine:

This compound was synthesized according to earlier report.[1] 6 g (0.0328 mol) of Lysine di-hydrochloride was dissolved in 42 mL 2(N) NaOH, was taken into a 250 mL jacketed two necked flask and cooled to 0□C. 28.8 g (0.0846 mol) of carbobenzoxy chloride (50% toluene solution) containing 34 ml (4)N NaOH was taken into a dropping funnel and added drop wise into the two necked flask at 0^0C with stirring. After 12 h the resulting mixture was washed with diethyl ether (20mL x 2). The reaction mixture was acidified with aqueous potassium bisulfate and extracted with diethyl ether. For further purification aqueous potassium bicarbonate solution was added to the ether extract. Aqueous layer was taken and acidified with potassium bisulfate and the product was extracted with fresh diethyl ether. The combined organic layer was dried with anhydrous sodium sulfate and concentrated under reduced pressure to obtain *α,ε-dicarbobenzoxy-l-Lysine* as a colorless syrup. Yield: 12g

Synthesis of ε-carbobenzoxy- α -carboxyl-l-Lysine anhydride:

According to the previous report[1], 7 g (16.9 mmol) of crude *α,ε-dicarbobenzoxy-l-Lysine* was dissolved in 30 mL of dry diethyl ether, was taken into a 50 ml flame dried schelnk flask and cooled to 0^0C. Then 4.22 g (20.28 mmol) of powdered phosphorus pentachloride was added in it and stirred for about 30-45 min until dissolution of all the solid. After quick filtration, this solution was quickly concentrated under reduced pressure at 45^0C. The product was crystallized from a mixture of dry ethyl acetate and dry petroleum ether. For further purification the product was recrystallized thrice from dry ethyl acetate and dry

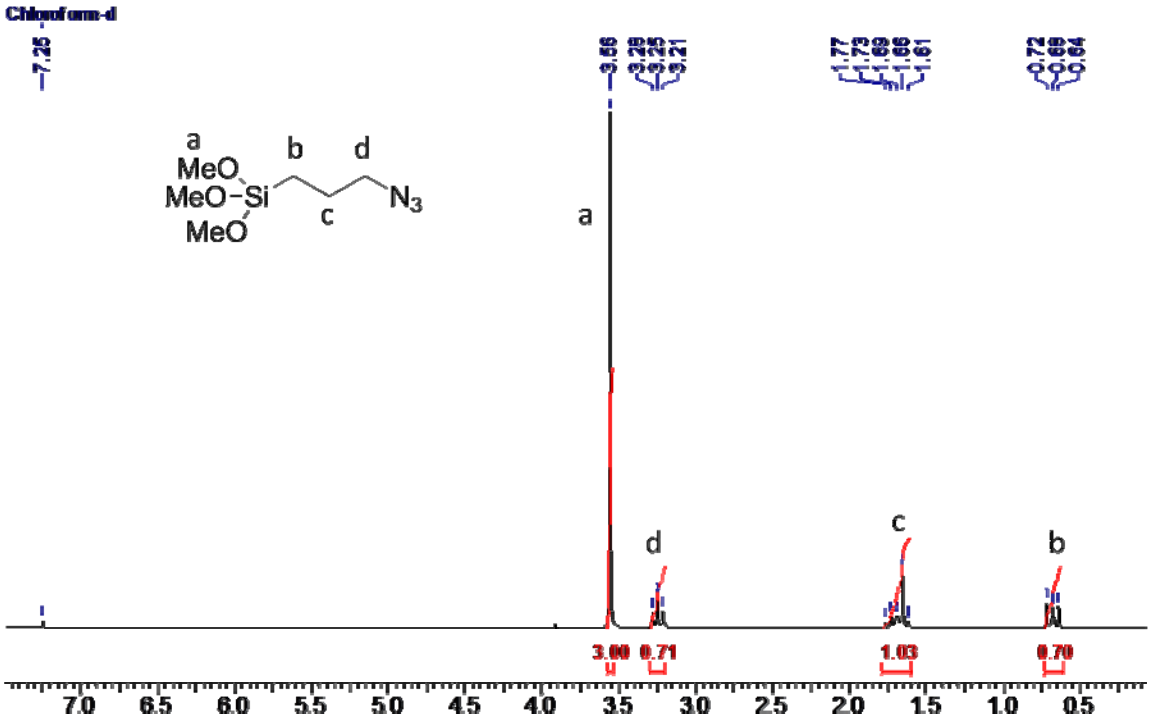
petroleum ether mixture. The product that obtained was dried under high vacuum at 50^0C and immediately transferred to the glove box. Yield: 4.5 g, (64%)

FT –IR: γ (Cm^{-1}, KBr): N-H (NCA, cbz) 3292; CH_2, 2936; CH_2, 2864; C=O (NCA)1814, 1692; C=O (cbz), 1652; N-H (cbz), 1534; C(=O)-O, 1257. ^{13}C NMR (100 MHz, $CDCl_3$) : δ 170.04, 156.93, 152.65, 128.61, 128.26, 128.04, 66.91, 57.47, 40.15, 30.83, 29.12, 21.35 ppm

Synthesis of Alkyne PEG 1000

Alkyne PEG is synthesized according to the earlier report.[2] In a typical reaction, NaH (0.255g, 6.4 mmol, 80% suspension in mineral oil) was added to a solution of monomethyl PEG-1000 (4g, 4 mmol) in dry THF (40) ml and the resultant mixture was allowed to stir for for 2 hours to afford a pale yellow solution. The reaction mixture was cooled to 0°C and propargyl bromide (1.43g, 12 mmol) was slowly added into the reaction mixture. The reaction mixture was stirred for an additional 24 h, diluted with ethyl acetate and concentrated in a vacuum. The final product was crystallized from hexane/ ethyl acetate mixture three times and directly used for CuAAC. Yield: 3.68 g

Figure S1: ^{1}H NMR and ^{13}C NMR of 3-azidopropyl trimethoxysilane



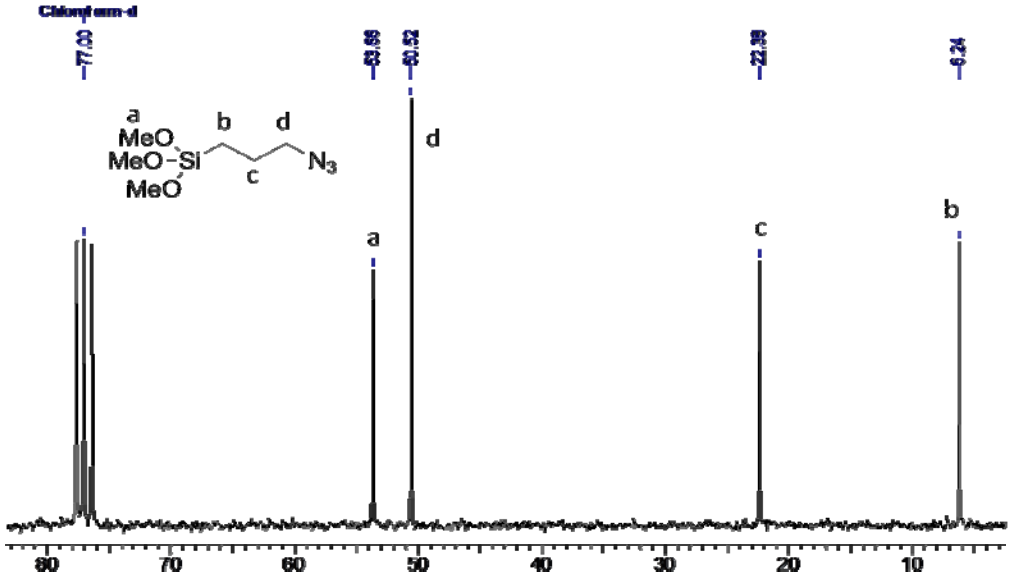


Figure S2: SEC trace in DMF in the presence of LiBr at 60 °C of alkyne-cbz-PLL-20

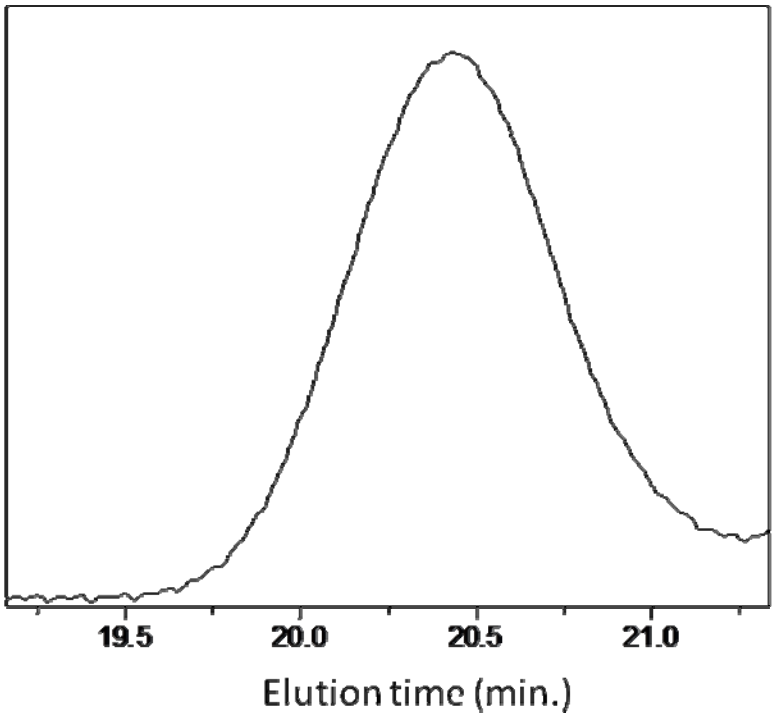


Figure S3: Infrared spectra of a) NCA cbz-lysine and b) alkyne-cbz-PLL in DMF.

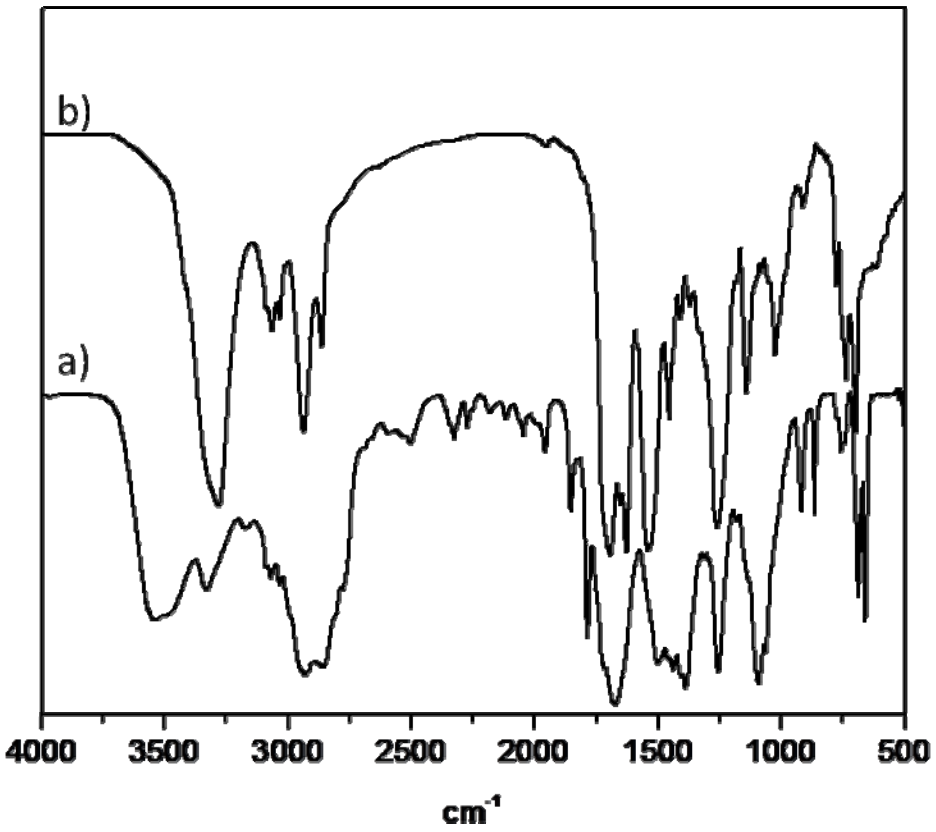
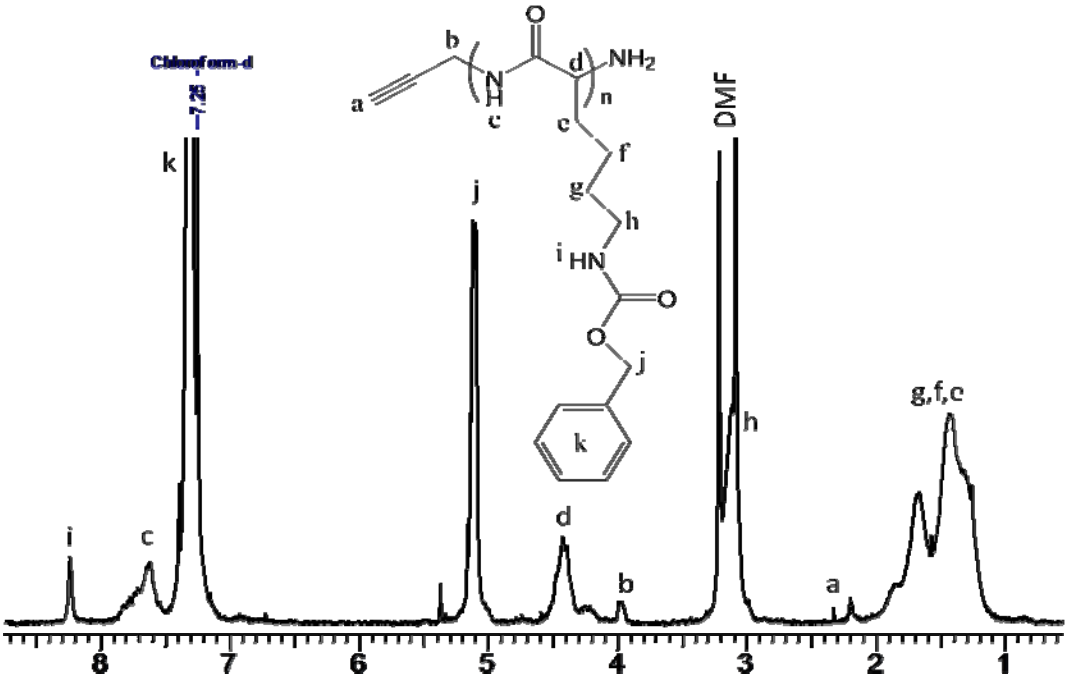


Figure S4 [1]*H NMR of alkyne-cbz- poly-L-lysine and alkyne-poly-L-lysine*



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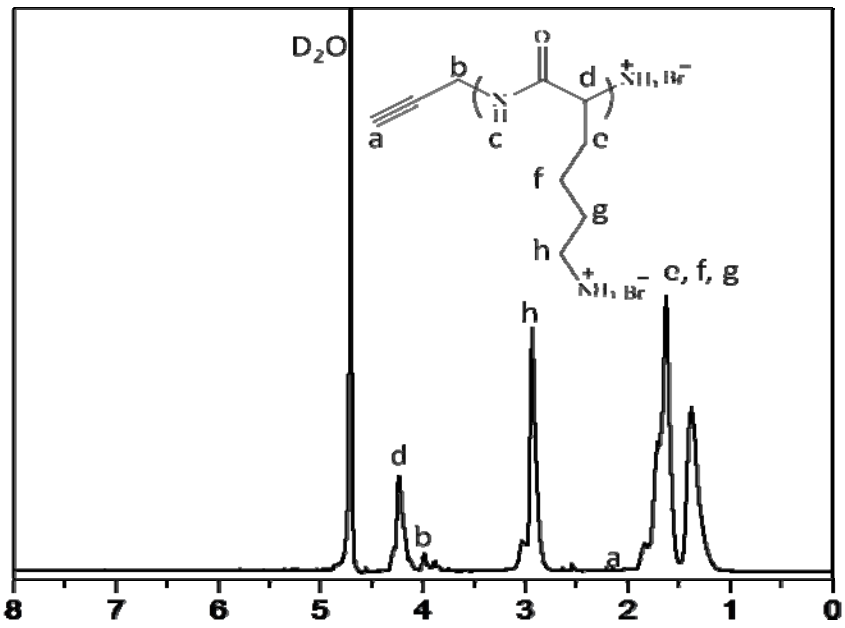

Reference

1. M. Bergmann, L. Zervas and W. F. Ross, *J. Biol. Chem.*, 1935, **111**, 245-260.
2. E. Delamarche, C. Donzel, F. S. Kamounah, H. Wolf, M. Geissler, R. Stutz, P. Schmidt-Winkel, B. Michel, H. J. r. Mathieu and K. Schaumburg, *Langmuir*, 2003, **19**, 8749-8758.