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SUPPLEMENTARY INFORMATION

Heat-chill Method of Preparation for Self Assembled Amphiphilic Poly(ε-caprolactone)-Poly(ethylene glycol) Block Copolymer based Nanoparticles for Drug Delivery

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Experimental

1. FT-IR spectra of all the samples were recorded using KBr pellets by spectrophotometer (Perkin Elmer 550, FT-IR spectrometer, USA) as shown in fig. 1. In FT-IR spectra of PECE triblock copolymer, a strong C=O stretching band appeared at 1721 cm^{-1}, which was attributed to the ester group. There is no absorption in 2250–2270 cm^{-1}, which indicates that the –NCO groups of HMDI disappeared completely due to coupling reaction between –NCO and –OH groups. The absorption band at 1528 cm^{-1} was attributed to the N–H bending vibrations, which confirmed the formation of PECE triblock copolymers. Gong et al. also reported similar results for solution polymerized PECE triblock copolymer.[1]

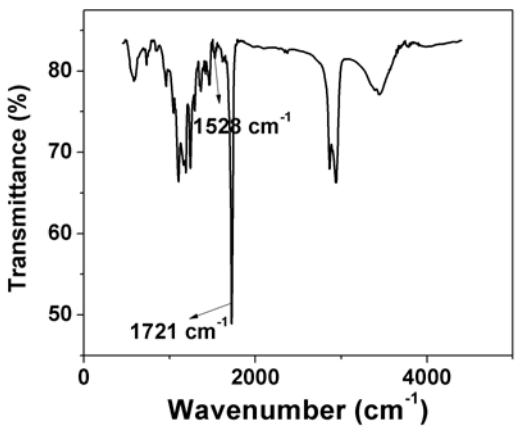

Fig.1 FTIR spectrum of PECE

2. NMR Spectrometer (BrukerAvance-II, 400 MHz) was used for ^1H NMR spectrometric analysis. $CDCl_3$ was used as solvent using TMS as an internal standard for this study. Fig. 2 shows the ^1H NMR spectrum of the PECE. The peak for chemical shift at 3.36 ppm corresponds to methylene proton of –O**CH3**. Peak at 4.06 correspond to –O$\mathbf{CH_2}CH_2CH_2CH_2CH_2CO$- in the PCL block. Weak peaks at 3.13 and 1.47 ppm belong to methylene proton of linker HMDI. The ethylene peak of the ethylene glycol ($\mathbf{CH_2CH_2}O$) unit (at 3.6 ppm) and the methylene peak of the caprolactone ($CO\mathbf{CH_2}CH_2CH_2CH_2CH_2O$) unit (at 2.2 ppm) in ^1H NMR spectra were used for the determination of number average molecular weight.[1-2]

3. Molecular weights and molecular weight distribution were measured at ambient temperature using a size exclusion chromatography [3] (Water, model 510 HPLC pump (Viscotek, USA), equipped with a Waters series *R*-400 differential refractometer and Waters Ultrastyragel columns of 10,000, 1000, 500 Å pore size which were preceded by a prefilter. HPLC grade tetrahydrofuran (THF) (Spectrochem, India) was used as the eluent at a flow rate of 1 mL/min. Before injecting into the GPC system, polymer solutions were filtered through a prefilter-filter combination system compatible with organic solvents. Polystyrene of narrow polydispersity was used as a calibration standard. The molecular weight of the polymer was calculated using polystyrene calibration curve.

4. Calibration graph was prepared to calculate encapsulation efficiency. Celecoxib was dissolved with varying concentration in 4ml tetrahydrofuran containing 100 µl of PBS(pH 7.4). Absorbance's at 256 nm were determined using UV-Vis spectrophotometer and plotted against concentration. Equation I shows the relationship between the absorbance and drug concentration. Where, y is the concentration of the drug (µg/ml) and x is the absorbance at 256 nm. R^2 corresponding to linear fit is 0.998.

$$Y = 24.08x - 1.55 \qquad \text{(I)}$$

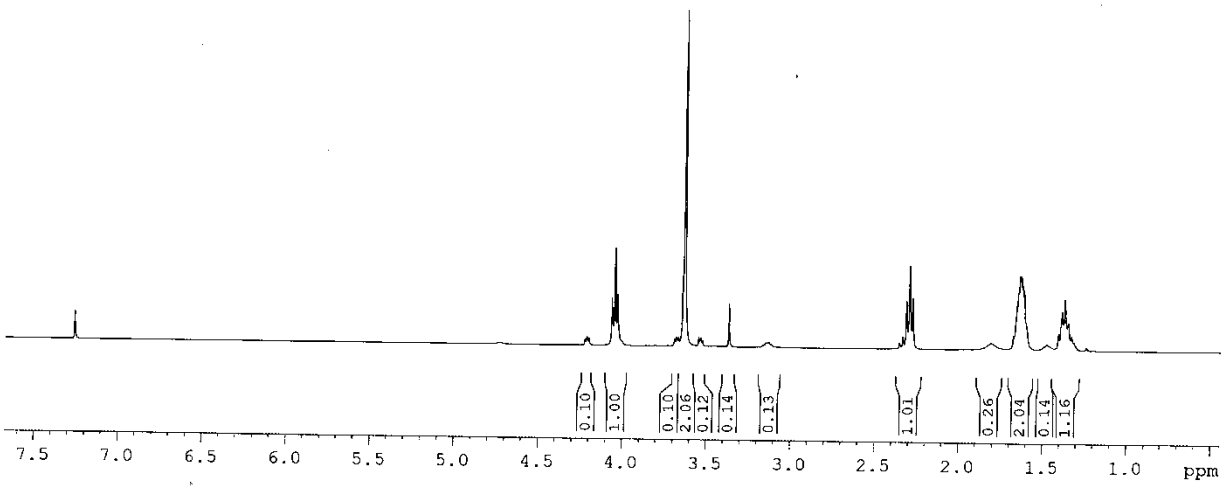

Fig. 2 ^{1}H NMR spectrum of PECE

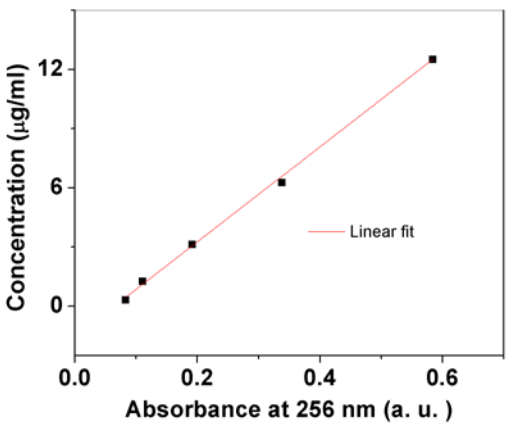

Fig. 3 Calibration graph showing relationship between celecoxib concentration and absorbance at 256 nm in tetrahydrofuran.

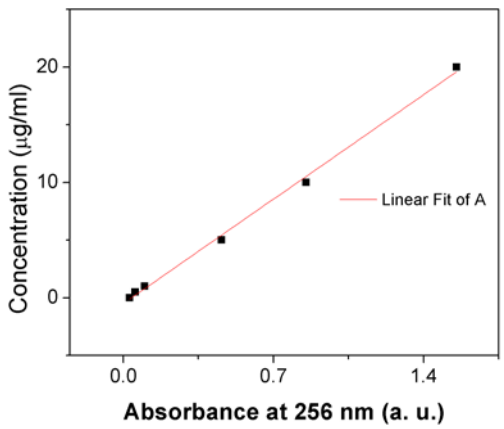

Fig. 4 Calibration graph showing relationship between celecoxib concentration and absorbance at 256 nm in PBS.

5. Calibration graphs were also prepared to determine the drug release. Celecoxib was dissolved with varying concentration in PBS (pH 7.4) containing 3% Tween 20. Absorbance's at 256 nm were determined using UV-Vis spectrophotometer and plotted against concentration. Equation II shows the relationship between the absorbance and drug concentration. Where, y is the concentration of the drug(µg/ml) and x is the absorbance at 256 nm. R^2 correspond to linear fit is 0.996.

$$Y = 12.932x - 4.91 \qquad \text{(II)}$$

Notes and references

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