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Synthesis and meticulous molecular, morphological and thermal characterization of linear and star-shaped polycaprolactones

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Architecture of polymers is an important parameter that influences their physical properties and final applications. Polymers with similar molar mass but varying architecture (such as linear to stars with different number of arms) can have very different physical properties. Therefore, characterization of polymers with regard to their architecture is imperative and at the same time very challenging. This is not achievable by conventional techniques. Modern chromatographic techniques can be the answer to such complex problems. In this study, linear and star-shaped polycaprolactones with varying number of arms are synthesized by ring opening of ε-caprolactone. The polymers were characterized in detail to obtain information of by-products in the samples. Size exclusion chromatography could not differentiate products with similar molar mass but different architecture and if there are any by-products in the sample. Liquid chromatography at critical conditions on NP and RP columns provided more insight into the products with regard to number of arms and possible by-products. MALDI-TOF MS analysis confirmed the structures and successful separation of targeted product from unwanted by-product. 2D-LC by coupling of LCCC to SEC in the second dimension demonstrated the similar size of the products that are separated successfully by LCCC. The purity and analysis of star-shaped precursor is imperative for further synthesis of star block copolymers that is not given much attention in recent literature. Thermal and morphological properties of the polycaprolactones with similar molar mass but different architectures are evaluated with DSC, TGA and AFM.

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Chromatographic resolution of angiotensin II receptor antagonists (sartans)

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First time a simple, sensitive and unified quantification method has been developed to analyze the complete class of angiotensin II receptor antagonists which are used in the treatment of hypertension either alone or in combination with some other drugs. The most important advantage of developed method was that the eight separate drugs can be determined on a single chromatographic system without modifications in detection wavelength and mobile phase. The drugs were separated on a Purospher Star 4.6 mmx25 cm, 5 µm, C18 column maintained at 40°C with 1 mL min−1 flow rate using UV detection at 254 nm. Good separation (Rs>2.0) was achieved in a short analysis allowing simultaneous determination of all eight sartans. The effect of variation in flow rate, detection wavelength and column oven temperature was also studied. The proposed method was statistically validated in terms of precision, accuracy, linearity, specificity and robustness. The newly developed method proved to be specific, robust and accurate for the quantification of eight sartans in commercial pharmaceutical formulations.

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