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# Regulating poly-caprolactone fiber characteristics *in-situ* during one-step coaxial electrospinning *via* enveloping liquids



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## ABSTRACT

Fiber modulation *in-situ* during the electrospinning process *via* enveloping liquid manipulation is a relatively un-explored process parameter. In this study, one-step control of fiber diameter and morphology are demonstrated using three liquids (ethanol, dimethyl silicone oil and mineral oil) serving as the outer-flowing medium during the co-axial process. Poly-caprolactone (PCL) fibers with various surface morphologies (porous, rough and smooth) and mean diameters (2–8 μm) were prepared. The results clearly show the modified enveloping liquid coaxial electrospinning approach permits fiber size regulation, hydrophobic enhancement and surface topography variation. This development has potential to meet requirements of polymeric fibers with micro and nano-patterned surface topographies, for biomedical and pharmaceutical applications, in a single step.

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## 1. Introduction

The utilization of polymeric fibers for biomedical and pharmaceutical applications has seen considerable growth over the last decade [1,2]. To date, several methods have been developed to prepare polymeric fibers with well-defined structures such as gas foaming, rapid prototyping and phase separation [3,4]. In addition, the electrospinning (ES) method, which utilizes an electrostatic field to engineer fibers, has attracted wide attention because of several key advantages over other existing methods.

Although conventional ES operation parameters permit size variation, the possibility to modulate size and surface properties in a single step has been limited. The modulation of fiber morphology during the ES process is usually coupled with several peripheral components, such as collection substrate [5,6]. However, this is not always ideal as the deposition distance also needs to be investigated (in tandem with collection medium) and in terms of active loaded fibers, could result in leaching of encapsulated drug (or biomolecules) before fiber solidification which impacts encapsulation and process efficiency. Therefore, enabling *in-situ* size and surface morphology (and therefore inherent hydrophobicity) control in one-step coaxial ES without altering the collecting

medium is extremely valuable.

Poly-caprolactone (PCL) is a synthetic biocompatible polymer with desirable biodegradability, mechanical properties and is also hydrophobic. For these reasons its use has been explored in several biomedical fields [7,8]. In this study, a coaxial ES approach using outer nozzle (enveloping) liquids to tailor fiber characteristics was explored. Optimum processing parameters such as polymer concentration and collector distance were first investigated. Subsequently, the impact of three enveloping liquids (ethanol, mineral oil and dimethyl silicone oil) was demonstrated. Resulting fibers were systematically investigated for their surface topography, hydrophobicity and mean diameter size distribution.

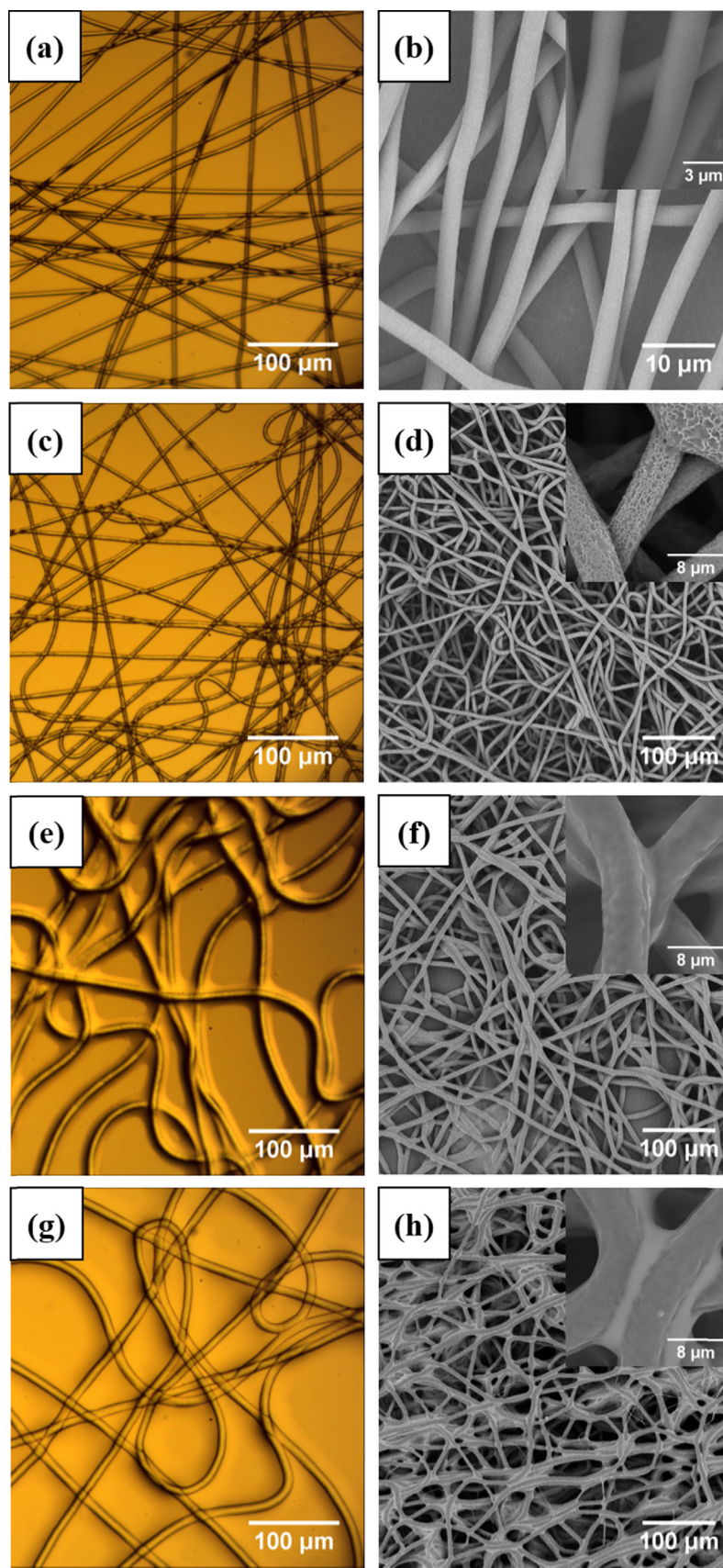
## 2. Materials and methods

### 2.1. Materials

Poly-caprolactone (PCL,  $M_w$ : 80,000 g/mol) and mineral oil were obtained from Sigma-Aldrich (St. Louis, USA). Dichloromethane (DCM) and ethanol were purchased from Sino-pharm Chemical Reagent Co., Ltd (Shanghai, China). Dimethyl silicone oil was obtained from Aladdin Chemistry Co., Ltd (Shanghai, China). Deionized water (DI water) was produced with a Millipore Milli-Q Reference ultra-pure water purifier (USA). All chemicals were analytical grade and were utilized without additional purification.

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**Fig. 1.** Optical micrographs and SEM of electrospun fibers without and with enveloping liquids. (a)-(b) PCL fibers without outer liquid. PCL fibers formed using the following enveloping liquids; (c)-(d) ethanol, (e)-(f) mineral oil and (g)-(h) dimethyl silicone oil. Insets at top right corners of electron micrographs are high-magnification images.

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