FABRICATION OF POLYCAPROLACTONE SCAFFOLDS USING AN E-JET 3D PRINTING SYSTEM

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ABSTRACT: The electrohydrodynamic jetting (or E-jetting) 3D printing system developed in-house is used as a fiber-based fabrication approach which applies electrical voltage between a nozzle and a substrate to deposit fibers onto the substrate layer by layer. PCL (polycaprolactone) is chosen as biomaterial of scaffolds because of its bio-compatibility and bio-degradability. This study focused on the fiber characteristics impacted by two main parameters, solution dispensing feed rate and plotting speed, to optimize filament diameter, filament formation and stability. Scaffolds fabricated with 70wt% PCL with size of 30×30mm and pore size of 300×300μm were investigated and characterized.

KEY WORDS:E-jetting, polycaprolactone (PCL), 3D printing.

INTRODUCTION

In recent decades, the E-jetttingtechnology, such as electrospinning and electrospraying, based on principle of the dynamics of electrically charged fluids, have attracted a lot of interests and rapidly developed. Electrospinning is a directand flexible process to produce polymeric fibers range from 100nm to 5μm, and at least one or two orders of magnitude less than fibers fabricated by conventional approaches such as micro-extrusion or solution spinning. The nanoscale architecture of the electrospun scaffolds exhibit a high surface area-to-volume ratio, high porosity(Chiu et al., 2005). Hence, the electrospinning technology is widely used in biomedical devices, like drug delivery(Liao, Chew, & Leong, 2006), tissue engineering(Tamayol et al., 2013) and so on. Although the electrospinning is relative simpler and varied setups, such as rotational mandrel(Moffat et al., 2008), separate plates(Xie et al., 2010), have been developed to collect different kinds of fibers (random and aligned), there still exists some inherent disadvantages in

Proc. of the Intl. Conf. on Progress in Additive Manufacturing
Edited by Chua Chee Kai, Yeong Wai Yee, Tan Ming Jen and Liu Erjia

ISBN: 978-981-09-0446-3 :: doi:10.3850/978-981-09-0446-3_058

electrospinning technique, e.g.small pore size (usually <10μm), difficulty for building three dimension structure, difficulty for post mechanical handling, etc., which limit the applications of electrospinning(Hong & Madihally, 2010).

Researchers in our group have developed a novel technology named electrohydrodynamic jet printing (E-jetting) system based on polycaprolactone biomaterial(Li et al., 2013). PCL is a kind of biocompatible and biodegradable polymers, which has been widely used in scaffolds design in tissue engineering (SHOUFENG YANG, 2001). Fibers in micron scale were deposited by applying high voltage between the nozzle and the substrate. With the movement of the collector, highly aligned and controllable fibers can be achieved repeatedly.To further optimize the parameters of the E-Jetting process to obtain stable fiber characteristics, this study focuses on the fiber characteristics impacted by two main parameters, namely solution feed rate and plotting speed.

METHODS AND MATERIALS

1. Experiment setup

The experiment setup consists of four maincomponents: syringe pump, nozzle, high voltage amplifier and translational stage (fig.1). A syringe with solution inside was put on the syringe pump to inject solution at a certain feed rate. The movement of the 4-axis translational stage wasmodulated precisely by the controller. This high voltage difference generated an electrical field between nozzle and substrate.The mutual columbic repulsion between the ions induces a tangential stress on the liquid surface, thereby deforming the liquid at nozzle tip into a conical shape, known as Taylor cone (Park et al., 2007). When the electrical force between the solution tip and the substrate overcome the surface tension within the molecules, the solution surface broke and came down.

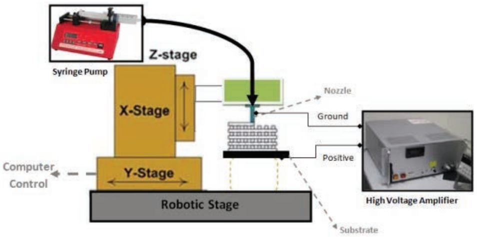


Figure 1 Schematic of E-Jet Printing System Setup

2. Materials preparation and fabrication

The solution was prepared as published before(Li, et al., 2013), briefly, solutions of 70% w/t were prepared by dissolving PCL (80 kDa) in acetic acid(99.7% purity) and stirred continuously for 4 hours at room temperature to obtain homogenous PCL solution. Each layer of scaffold was fabricated using an orthogonal configuration with the movement of the stage and the pore size was designed to be 300μm×300μm. Multiple layers are deposited repeatedly layer by layer.

3. Test Range of Parameters

The quality of E-Jetted fibers is mainly determined by the processing parameters, i.e.solution concentration, high voltage, dispensing feed rate and plotting speed. The weight volume ratio of PCL solution used in this project was 70%, which was proved by previous studies that this concentration was able to plot good quality filaments(Li, et al., 2013). The flow rate which was controlled by syringe pump ranged from 2.0μl/min to 6.0μl/min. The electrical voltage is 3kV. Plotting speed varied from 15mm/s to 60mm/s. The gapdistance between nozzle tip and substrate was set to 3mm.

4. Mechanical testing

Mechanical testing was conducted to investigate the tensile properties of the scaffolds determined by the variables by using InstronMicrotester. In order to clamp the scaffolds firmly, paper with size of 30mm×5mm was glued to the top and bottom of the 30×30mm scaffold at both sides to prevent it from slippery. Each scaffold was placed in a flat and vertical manner to make sure that the results were accurate. Uniaxial tensile force was applied to the scaffold.

RESULTS AND DISCUSSIONS

1. Effects of Mass Flow Rate on scaffold fabrication

Fig. 2 shows how the diameters of the scaffoldsdetermined by the dispensing feed rate, and fibers fabricated with five plotting speeds (20, 30, 40, 50, 60mm/s) were investigated and compared. From the figure, it can be found that with the increase of the dispensing feed rate, initially, the fiber diameters all increased; this is because more solution dispensed out of the nozzle and thickened the fiber diameter. However, when the feed rate was further increased (to 6μl/min), the fiber diameter was decreased reversely. The phenomenon occurred because the high dispensing speed of the solution corrupted the balance of the voltage and plotting speed, and not all the solution pushed out can be dragged onto the substrate; hence, some solution accumulated on the nozzle tip and solidified quickly because of the high solution viscosity, and in turn change the distance between the nozzle and the substrate.

2. Effects of Plotting Speed on scaffold fabrication

In this experiment, ten plotting speed range from 15mm/s to 60mm/s with 5mm/s interval were investigated and the fiber diameters were measured. It is observed that plotting speed has significant effects on filament diameter. Filament diameter decreases significantly with the increase of plotting speed (Fig.3). With the increase of the plotting speed from 15mm/s to 60mm/s, fiber diameter decreased from 125.81±10.55μm to 74.20±7.09μm.

Fig. 4 indicated the impact of plotting speed to the scaffold morphology. Based on the observation of the experiment, the jet of PCL solution became more stable with the increase of plotting speed. When plotting speed decreased, filament diameter increased significantly. This made it harder for the filaments to solidify and thus the junctions of the filaments immerged together between layers.This can result in decrease of the interconnectivity of scaffolds. Additionally, even though scaffolds with large filament diameter and connected joints have better mechanical strength, itis highly possible that scaffolds would be fabricated with flaws. During solidification process, softfilaments with large diameter can easily disorder at the middle point due to gravity when it is only supported at two joints. As shown in Fig.4(A), because of the solidification problem result from the thick fibers, the middle portion of the fiber is much thinner than the fibers in junction and fibers immerges in junctions, which causes the non-uniformity of fibers, while gradually increasing the plotting speed, these phenomena reduced and morphology became uniform (Fig.4(B-C)).

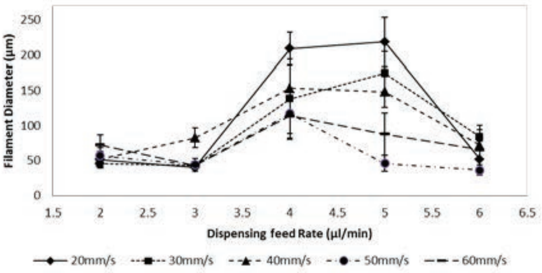


Figure 2 Effects of dispensing flow rate on filament diameter

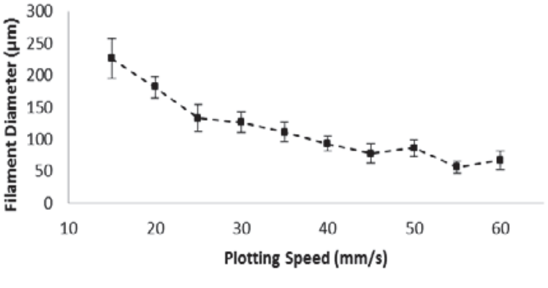


Figure 3 Effects of plotting speed on filament diameter

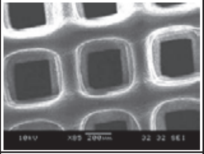
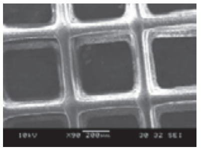
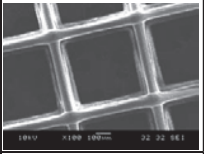
A	B	C
		
15mm/s	30mm/s	45mm/s
125.81±10.55µm	86.69±12.52µm	68.23±7.19µm

Figure 1 SEM images of scaffolds with different plotting speed

3. Mechanical testing

The samples for testing mechanical properties were fabricated using the parameters of dispensing feed rate 2.0µl/min, plotting speed 25mm/s and high voltage 3.0V, whose processes was proved to be repeatable and stable. Fig. 5(A) demonstrates the Instron mechanical testing machine, (B) shows the clamping setup as described in method and material section. After clamping, the gauge length was 20mm, and the test was carried on with tensile speed of 10mm/min and five samples were duplicated. Fig. 5(C) indicates the typical stress-strain curve of the as-fabricated scaffolds. The Young's modulus was 34.86±9.39Mpa and ultimate stress was 3.01±0.83Mpa. Compared with the mechanical properties of the conventional electrospun PCL random fibers (young's modulus 7.12±0.8Mpa), even the aligned fibers (young's modulus between 11.93±1.22Mpa and 33.2±1.98Mpa)(Thomas, et al., 2006), the E-Jetted fibers has better mechanical properties; however compared with extruded PCL fibers (young's modulus 190±6Mpa)(Averous, Moro, Dole, & Fringant, 2000), the mechanical strength of the E-Jetted PCL fibers are less strong. The reason for the difference in mechanical properties among distinct fabrication processes is the difference in

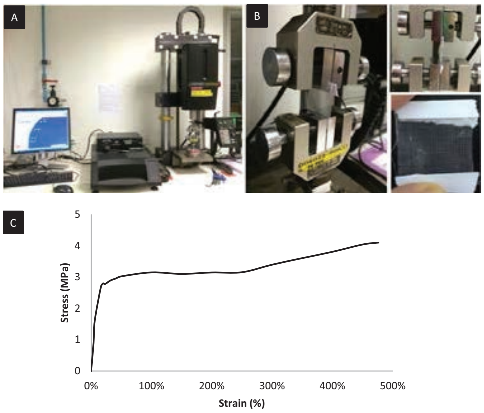


Figure 5 (A) Overview of Instron mechanical testing system; (B) Clamping setup for scaffold testing; (C) Typical stress-strain curve of scaffold

fiber diameters. As to electrospun fibers, they are usually nano/submicron scale; for extruded process, fibers are usually several hundred microns. Hence, it is assumed that with the increase of the fiber diameters, the mechanical strength will be reinforced.

CONCLUSION

In this study, by using 70% w/t PCL solution, the experimental parameters were further investigated and optimized. It can be concluded that diameters of the E-Jetted fibers can be controlled by adjusting the dispensing flow rate and plotting speed; however, for the sake of process stability, there is upper limitation for the feed rate (6μl/min in this work). In addition, the morphology (fiber uniformity, junction pattern, etc.) of the as-fabricated scaffolds are determined by the fiber diameters. In terms of mechanical properties, the E-Jetted fibers are proved to possess superior mechanical strength than the conventional electrospun fibers, although it should be further enhanced for different scaffoldingapplications.

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