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Experimental investigation on process parameters of near-field deposition of electrospinning-based rapid prototyping

Deepak Parajuli[a], Pisut Koomsap[a], Alok A. Parkhi[a] and Pitt Supaphol[b]

[a]Industrial and Manufacturing Engineering, School of Engineering and Technology, Asian Institute of Technology, Pathumthani, Thailand; [b]Petroleum and Petrochemical College, Chulalongkorn University, Bangkok, Thailand

ABSTRACT
Near-field electrospinning (NFES) with its capability to produce a straight fine fibre has been integrated into additive manufacturing for the fabrication of scaffolds with controllable pattern structures. However, building the third dimension with NFES is not easy due to the unsolidified fibre while being deposited. Presented in this paper is an investigation on the influence of process parameters on achieving a small cylindrical fibre from the near-field fibre deposition of an electrospinning-based rapid prototyping. A set of experiments have been conducted on solutions of polycaprolactone (PCL) in N,N-dimethylformamide (DMF). Parameters of interest are voltage, standoff distance, polymer concentration, environmental condition and needle size. From the experimental results, polymer concentration, environmental condition and needle size had influence on achieving a small cylindrical fibre. Under near-field deposition, the concentration should be high, the needle should be small and the temperature should be maintained during the process.

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1. Introduction

Near-field electrospinning (NFES) that stemmed from conventional far-field electrospinning (FFES) (Sun *et al.* 2006) has received much attention in recent years because it allows a deposition in a control fashion of a continuous fine fibre produced effortlessly from a stable polymer jet that is electrically drawn from a liquid polymer or polymer melt. The ability to control the pattern of the fibre deposition has been of researcher interest because it can help improve the performance of the conventional electrospinning that deposits the fibre randomly on a collector. For instance, in tissue engineering on which more than 40% of research publications on advanced applications of nanofibres are (Thavasi *et al.* 2008), electrospun fibres are used to construct scaffolds required to support cell attachment, proliferation and differentiation (Tellis *et al.* 2008). To perform their functions effectively, the fabricated scaffolds must meet not only mechanical and biological requirements but also structural requirements (Hasan *et al.* 2014). Porosity which is a space per a whole volume ratio should be high to contribute space for cells to attach and regenerate in all direction (Loh and Choong 2013) while a large pore size accommodates and delivers the cells for tissue regeneration (Too *et al.* 2002). Besides, pore interconnectivity is needed for the exchange of nutrients and gas, and for the cells to penetrate into the scaffold structure. The random deposition of FFES, however, limits the ability to control the construction of internal channels within the scaffolds (Lam *et al.* 2002). As a result, the cells tend to grow on the surface instead of penetrating inside the scaffolds (Li *et al.* 2014).

Three-dimensional construction with controllable architecture by using electrospinning has been attractive for tissue engineering because electrospun fibres can mimic the fibre structure of natural extracellular matrix (Cai *et al.* 2013). Electrospinning has been applied with additive manufacturing for scaffold fabrication where electrospinning was used to create fine fibres while additive manufacturing techniques were used to control the architecture (Park *et al.* 2008, Chen *et al.* 2009, Owida *et al.* 2011). With direct writing capability of NFES, researchers have tried recently to fabricate 3D scaffolds directly from electrospinning. They are known under the names of electrohydrodynamic jet (EHD-jet) (Wei and Dong 2013), E-jetting (Li *et al.* 2014), electrospinning-based rapid prototyping (ESRP) (Chanthakulchan *et al.* 2015a) and electrohydrodynamic direct writing (EDW) (Zheng *et al.* 2016). These techniques are similar that they all follow fused deposition modelling process for a layer construction but instead of using extrusion process for fibre creation, NFES is applied to generate electrostatic force to draw a continuous fibre from a polymer solution or melt.

CONTACT Pisut Koomsap ✉ pisut@ait.asia

Table 1. Parameters classified based on influencing level on fibre diameter (Thompson *et al*., 2007).

Major influence parameters	Moderate influence parameters	Minor influence parameters
Initial jet radius Volumetric charge density Standoff distance Initial elongational viscosity Relaxation time	Initial polymer concentration Perturbation frequency Solvent vapour pressure Solution density Electrical potential	Vapour diffusivity Relative humidity Surface tension

Table 2. Average diameters of polymer jets and dry fibres at different supplied voltages.

Applied voltage level (kV)	Jet diameter (μm)	SD (μm)	Fibre diameter (μm)	SD (μm)	TR
3.2	57.15	0.69	162.54	2.80	2.84
3.5	53.08	1.20	168.14	2.80	3.17
3.7	55.24	1.31	159.74	5.60	2.89
3.8	57.55	0.69	162.54	5.60	2.82

Table 3. Average diameters of jets and dry fibres at different standoff distances.

Standoff distance (mm)	Jet diameter (μm)	SD (μm)	Fibre diameter (μm)	SD (μm)	TR
5	57.15	0.70	162.54	2.80	2.84
8	51.09	1.12	156.94	4.85	3.07
10	44.31	1.08	148.53	2.80	3.35
12	41.93	1.13	130.78	4.28	3.12
14	41.59	0.55	129.85	4.28	3.12

The set-up of an equipment for NFES is similar to the conventional FFES, except that the standoff distance (SOD) is much shorter and the pattern is created by controlling the movement of the collector. When NFES was first introduced, the formation of a polymer droplet was done by dipping a wire tip in a solution (Kameoka *et al*. 2003, Sun *et al*. 2006). Later on a syringe needle has been applied for fast continuous deposition of a fibre for a large area (Chang *et al*. 2008, Bisht *et al*. 2011, Bu *et al*. 2012). The fibre stretching is created by the speed of the collector instead of the bending instability (Zheng *et al*. 2016). The faster the collector moves, the smaller the fibre diameter will be (Auyson *et al*. 2013). Chanthakulchan *et al*. (2015a) conducted experiments by using a solution of 10% weight of polycaprolactone (PCL) in N-dimethylformamide, and supplying 3.2 kV between a 20G needle with an inner diameter of 610 μm and a collector that were 5 mm apart. The experimental results showed the repeatability and reproducibility of the technique. However, deposited fibres remained in liquid state. The liquid fibres settled down to form a flat ribbon shape instead of a cylindrical shape. In case of multilayered fibre pattern, new layer was deposited on the previous liquid layer. Both the layers being in liquid state combined together to form one larger layer of scaffolds with lattice pores. Li *et al*. (2014) experienced similar results and reported much better control of the height of scaffolds at high PCL concentration in acetic acid (>70%w/v, PCL: acetic acid) when 2.2 kV was supplied between a 200 μm inner diameter nozzle and a collector that were 2 mm apart.

NFES benefits from the continuity of conventional electrospinning and superior location control to produce nanofibre patterns over larger areas. However, because the SOD is short, large amount of the solvent remains in the jet and majority of solidification process happens on the collector. Researchers have tried to solve this problem, for example, by increasing a flight time (i.e. to extend the straight part) by lowering voltage, and increasing viscoelasticity of the polymer solution (Bisht *et al*. 2011). Compared to the works on the conventional electrospinning, the process investigation has been much less. Presented in this paper is a study to have better understanding of near-field deposition in ESRP. A set of experiments were conducted to investigate the influence of process parameters on achieving small cylindrical fibres. The experiments were conducted in a particular sequence starting from voltage to SOD, polymer concentration, environmental condition and needle size. Next section reviews the parameters affecting the electrospinning process. Section 3 explains experimental set-up used for carrying out the experiments. Section 4 presents the experiments and results. The conclusions are addressed in the final section.

2. Literature review

The characteristics of electrospun fibres are influenced by several parameters that include solution properties (e.g. viscosity, conductivity, etc.), process parameters (e.g. electric potential, SOD, needle diameter, polymer concentration, etc.) and ambient conditions (e.g. temperature, humidity) (Doshi and Reneker 1995). For conventional electrospinning, significant change in fibre diameter occurs during bending instability when the fibre undergoes stretching and elongation (Rogina 2014). Intensive investigations have been conducted theoretically as well as experimentally on various types of materials. Thompson *et al*. (2007) for instance studied 13 parameters in an electrospinning theoretical model and determined their different effects on the fibre diameter as illustrated in Table 1. Majority of these parameters have an influence on enlarging the fibre diameter. Only SOD, elongational viscosity and solution density have an influence on reducing the diameter.

In case of NFES, there are few reports on the influence of parameters on the fibre diameter. Similar to the results from FFES (Katti *et al*. 2004, Gu *et al*. 2005), the influence