

# Electrospinning of Alginate and Polyethylene Oxide Blends Using Pulsed Electric Fields to Fabricate Chopped Nanofibres

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**Abstract—** For Drug delivery systems using biopolymer nanofibres, chopped nanofibres are required because the amount of drug to be released depends on the length of the fibre. Electrospinning is a simple method used in fabricating polymer nanofibres. In this work the non-continuous-mode electrospinning technique has been successfully used to fabricate and chop nanofibres. Results obtained with samples of a synthetic polymer, polyethylene oxide (PEO) and blends of the natural biopolymer alginate with PEO are presented.

## I. INTRODUCTION

Nanoparticles have already found important uses in drug release applications and diagnostics because the nanoscale is reflected in the biological scale, for example, in sizes ranging from that of enzymes and viruses up to bacteria [1]. It is believed that nanotechnological approaches in medicine will have a major impact in the next decades. Lately, controlled drug delivery systems have gained significant attention, especially with respect to the use of polymer nanofibres. For drug delivery systems, the ability to control the length of the nanofibre is very important since the amount of drug released depends on the length of the fibre. Techniques for fabricating and chopping nanofibres that will provide the required control must therefore be developed.

Electrospinning, the simplest and most cost-effective method of fabricating polymer nanofibres, has been researched worldwide. To date, over 50 synthetic or natural polymers and polymer blends have been successfully spun using the electrospinning process, for the most part in research laboratories [2]. The current research initially employed polyethylene oxide (PEO), which is a synthetic polymer, several blends of which have been used for diverse applications in recent studies [3]. PEO is easy to spin; hence, it

helps to evaluate the system of electrospinning. For biomedical applications such as drug delivery, the materials used must be biocompatible, and natural polymers are therefore preferred [3, 4].

The method of electrospinning produces polymer nanofibres using electrostatic force. In the process, a high voltage is used to create an electrically charged jet of polymer solution, which elongates, and finally the solvent is evaporated to leave the nanofibres on the collector. Thus, an electrified jet ejects from a droplet when the spinneret is kept at higher a potential than the threshold voltage. On the other hand, the jet does not eject when the spinneret is kept at lower than the threshold voltage. Therefore, it is possible to chop nanofibres by accurately controlling the values of the applied voltages using a specialized high voltage pulsed power supply for non-continuous-mode electrospinning process.

## II. EXPERIMENTAL

### A. Material preparation

PEO with a molecular weight of 600,000, purchased from Aldrich®, was chosen for the preparation of the solution for electrospinning. The PEO fibres were electrospun using a 5% (w/w) concentration of PEO in distilled water. Sodium alginate (SA), a polysaccharide derived from brown seaweed [5], a biodegradable polymer, was also studied. However, electrospinning of the SA from aqueous solutions seems difficult due to its high electrical conductivity. In fact SA has been successfully spun in polymer blends formed by blending SA with a non-toxic, non-ionic, and biocompatible synthetic polymers, such as PEO. Based on the above discussion, SA from brown algae (2500 cps and a 2% solution at 25 °C) and PEO were chosen for the preparation of the blended solution. SA and PEO solutions with a 3% (w/w) concentration in distilled water were first prepared separately. Then, the SA and PEO solutions were blended together.

### B. Experimental setup

Fig. 1 illustrates a schematic of the experimental setup used to perform the non-continuous-mode electrospinning experiments. It consists of a high-voltage power supply, a pulse circuit, a wave shaping resistor, a syringe pump, syringe with metallic needle, and a grounded collector. A custom built IGBT-based pulsed power supply has been used for non-continuous-mode electrospinning. The pulse width is down to a few hundred microseconds and the voltage amplitude is up to 10kV. A wave shaping resistor was connected to the electrospinning setup in parallel. The pulse voltage was applied between the needle and the collector plate. The polymer solution was forced through a syringe needle at a constant rate of 0.04 ml/min by the syringe pump, resulting in the formation of a droplet of polymer solution at the syringe tip. An AWG 24 blunt needle was used with the syringe that was placed vertically above the collector plate during all the experiments. The gap distance between the needle and the collector is 150mm. The electrospun fibres were collected on a grounded collector that is a thin round plate made of aluminum, with a diameter of 300 mm. A Leo 1530 Gemini scanning electron microscope (SEM) was used to see the morphology of the electrospun fibres.

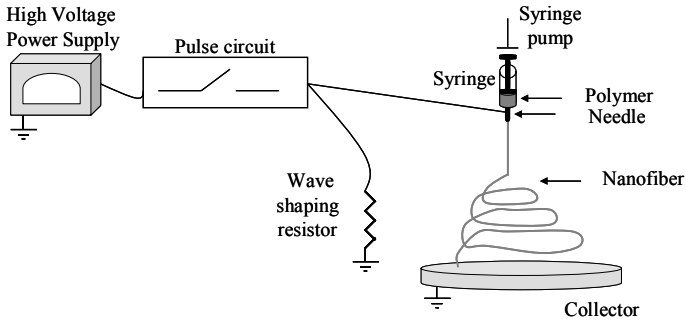


Fig. 1. Schematic of the experimental setup used to perform the non-continuous-mode electrospinning experiments.

### III. RESULTS AND DISCUSSIONS

#### A. The pulse-on and pulse-off voltages for non-continuous-mode electrospinning

For non-continuous-mode electrospinning, determining the pulse-on and pulse-off voltages that are applied is necessary. To start a jet, the pulse-on voltage must be above the threshold voltage. The pulse-on voltage also influences the morphology of the nanofibers. To stop a jet, the pulse-off voltage must be below the threshold voltage. To find the threshold voltage, different magnitudes of DC voltage were applied to the electrospinning apparatus using the PEO solution as a test sample. By controlling the magnitude of the applied DC voltage to the electrospinning apparatus, different types of droplets and jets from the needle were observed corresponding to different levels of DC voltages, as shown in Fig. 2. From this result, the pulse-on voltage of 10 kV and the pulse-off voltage of 2.7 kV were determined, and selected as test voltages to be applied for the electrospinning setup.

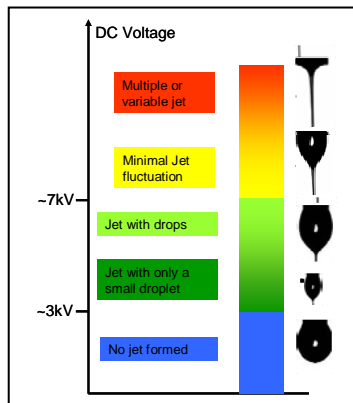


Fig. 2. Formation of different types of droplets and jets as a function of applied DC voltage.

### B. Non-continuous-mode electrospinning with a PEO solution

Fig. 3 shows the changes in the droplet and jet formations. Fig. 3 (a) shows the droplet that was accumulated during the pulse-off voltage. The pulse-on voltage was applied to the needle where the droplets were to be prepared. When the voltage is applied, the meniscus of the liquid deforms into a Taylor cone, and a jet of the liquid is then ejected from

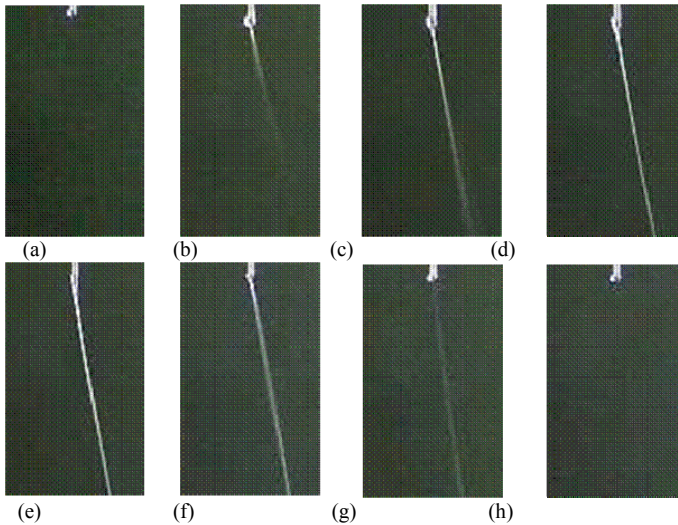


Fig. 3. Droplet and jet formations produced by the pulse voltage,  $T=500$  ms,  $D=40\%$ : (a) During the pulse-off voltage (b)  $T = T_{\text{form}} = 0$  s (c)  $T = 33$  ms (d)  $T = 66$  ms (e)  $T = 100$  ms (f)  $T = 200$  ms: the pulse-off voltage has begun to be applied (g)  $T = 233$  ms (h)  $T = 266$  ms.

the cone as shown in Fig. 3 (b). The time for the Taylor cone to form and then for a jet to eject is denoted as  $T_{\text{form}}$ . To set a standard, the time for the event shown in Fig. 3 (b) was determined to be  $T = T_{\text{form}} = 0$  s. Fig. 3 (c) shows the jet before it became stable. As can be seen from Fig. 3 (d) and (e), the stable jet was observed during  $T = 66$ - $200$  ms. At  $T = 200$  ms in Fig. 3 (f), the voltage was turned off. Sixty-six ms after the voltage was turned off, the jet completely disappeared, and the cone returned to the initial meniscus, as can be seen in Fig. 3 (h).

A field emission scanning electron microscope (FE-SEM) was used to examine the diameter and the morphology of the fibres, and the small droplets on the collector. Fig. 4 shows the SEM images of the nanofibres. It can be seen that the diameters of the most fibres in all cases are in the range of 100-400 nm with rounded beads.

From Figs. 3 and 4, it can be said that nanofibres were successfully fabricated and chopped with the PEO solution using the pulsed power supply.

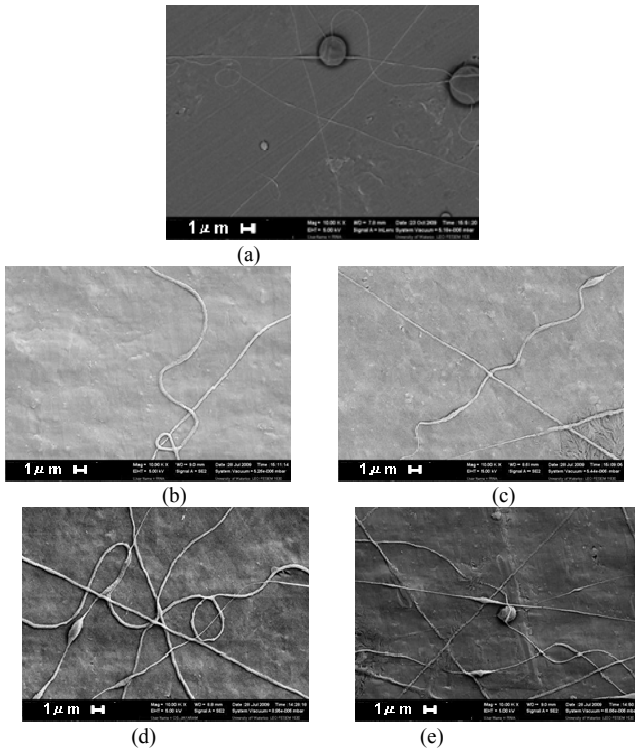


Fig. 4. SEM images of the samples produced by the pulse voltages: (a) T=200 ms, D=40 % (b) T=500 ms, D=20 % (c) T=500 ms, D=40 % (d) T=1,000 ms, D=20 % (e) T=1,000 ms, D=40 %

### C. Non-continuous-mode electrospinning with SA/PEO blended solution

The conductivities of the blend solutions are listed in Table 1, measured with Con11/110 Conductivity/TDS handheld meter. Fig. 5 shows the changes in the droplet and jet formation for sample B from Table 1. The explanations of Fig. 5 are the same as those of Fig. 3 except for Fig. 3 (b). Fig. 5 (b) shows that a jet is ejected and then breaks up into small droplets when the voltage is applied. This effect might occur because the surface tension of the droplet is too strong to initiate a jet. In fact, the size of the droplet shown in Fig. 5 (a) is larger than that shown in Fig. 3 (a).

In the cases when the volume ratios of the SA and the PEO are 20:80, 40:60, and 60:40, the jet is always ejected during the turn-on voltage; however, droplets were occasionally observed. When the ratio is 80:20, no jet is observed. As can be seen from Table 1, a higher proportion of SA creates a higher conductivity in the solution because SA is a polyelectrolyte, which has a high conductivity. In the case of an 80:20 volume ratio of SA to PEO, no jet is observed. Similar observations have been made with high conductivity solutions as it is hard to attain the required tangential electric field for jet formation. The strong repulsive force among the polycations could also be a key factor that hinders electrospinning [6]. In the cases of 20:80, 40:60, and 60:40 volume ratios, the jet is always ejected during the turn-on voltage; however, the droplet occasionally falls downward.

TABLE 1: CONDUCTIVITIES OF THE SA/PEO BLENDED SOLUTIONS

Sample ID	Volume ratio SA (3%) : PEO (3%) in water	Conductivity [mS/m]
A	20:80	83.2
B	40:60	156.6
C	60:40	398.0
D	80:20	612.0

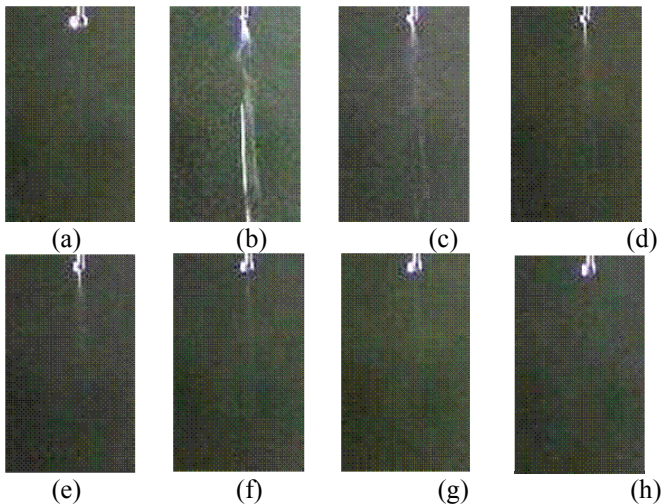


Fig. 5. Droplet and jet formations of the SA/PEO solution 40:60: (a) During the pulse-off voltage (b)  $T = 0$  s (c)  $T = 33$  ms (d)  $T = 66$  ms (e)  $T = 100$  ms (f)  $T = 200$  ms: the pulse-off voltage, (g)  $T = 233$  ms (h)  $T = 266$  ms.

Fig. 6 shows SEM images for samples A, B, and C in Table 1. It shows that the diameters of most of the fibres for all volume ratios are in the range of 30-40 nm. The diameters in Fig. 6 are thinner than those in Fig. 4, because the conductivities of the SA/PEO solutions are higher than that of the PEO solution, 7.6 mS/m. Electrospinning involves the stretching of the solution, which is caused by the repulsion of the charges on its surface. Therefore, if the conductivity of the solution is increased, more charges can be carried by the electrospinning jet, thus resulting in the stretching of the solution. The increase in the stretching of the solution tends to yield fibres with smaller diameters. The beads also become smaller and more spindle-like. However, if the conductivity is too high, the recombination of charges may hinder any jet formation.

From Figs. 5 and 6, it can be said that nanofibres were successfully fabricated and chopped using the blends of SA and PEO solutions using the pulsed power.

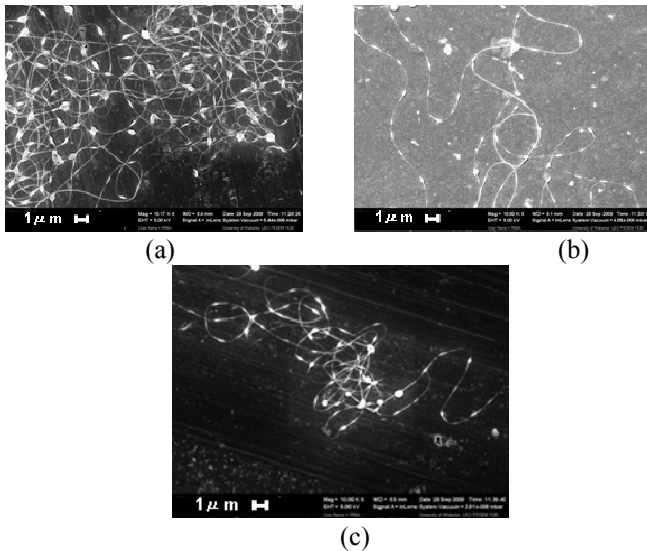


Fig. 6. SEM images of the samples produced by the pulse voltage using the SA/PEO solutions: (a) 20:80 (b) 40:60 (c) 60:40.

#### IV. CONCLUSIONS

Nanofibres were successfully fabricated and chopped with the PEO blended solutions using the pulsed power application. The diameters of most of the fibres using the PEO solution are 100-400 nm with rounded beads. In the case of the 80:20 volume ratio of SA to PEO, no jet was observed. In the cases of volume ratios of 20:80, 40:60, and 60:40, the jet was always ejected during the turn-on voltage, and the diameters of most of the fibres are in the range of 30-40 nm. The diameters of the fibres obtained using SA/PEO blended solutions are thinner than those obtained from PEO solution which could be related to the higher conductivities of the SA/PEO solutions compared to PEO alone.

#### V. ACKNOWLEDGEMENTS

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