

Selection of optimum electrospinning parameters

Several experiments were performed to optimize the electrospinning process for producing PVDF nanofibers. Various parameters such as solvent, tip to collector distance (TCD), and flow rate to get bead-free, smooth and uniform fibers with smaller average fiber diameter (AFD). In general, N,N-dimethylformamide (DMF), dimethyl sulfoxide (DMSO) and triethylphosphate (TEP) are considered as good solvents for PVDF as they have high dielectric constants to match with that of PVDF. DMSO plays a vital role in increasing the electrical properties and dielectric constant in PVDF compared to DMF and TEP [1].

TCD, flow rate and type of solvents has played a vital role in obtaining the desired nanofiber morphology. The fibers produced from DMF solution were brittle and difficult to handle. However, fibers produced using DMSO were ductile with improved morphology and higher production rate (**Fig.1**). This may be attributed to the high boiling point and high dielectric constant of DMSO [2]. The critical entanglement concentration for PVDF in DMSO was found to be around 18 wt./v%. Increase in flow rate resulted in increased AFD (**Fig.2**). At a TCD of 15 cm fibers were randomly oriented and partially wet. However, at a TCD of 20cm fibers were smooth, uniform and bead-free with improved orientation. A further increase in TCD leads to decrease in collection of fibers with bead formation (**Fig. 3**).

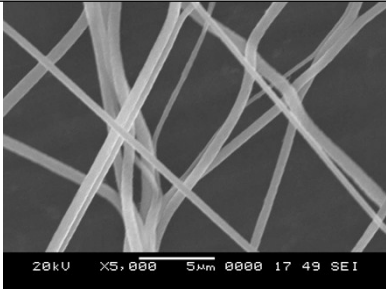
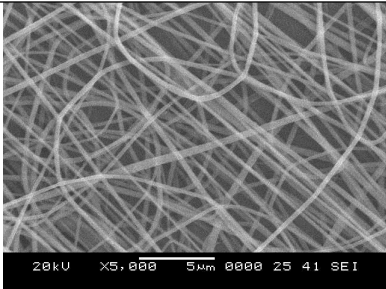
Parameters	Solvent	SEM Micrographs	Remarks
Flow rate :0.5 mL/hr Tip to collector Distance (TCD) : 20 cm Concentration : 18 wt./v% Applied Voltage : 20 kV	DMF		Fibers were smooth and bead free. Fibers were brittle and the AFD was 790 nm. Production rate of fibers was low.
AFD ± SD (nm)		790 ± 143	
Flow rate :0.5 mL/hr Tip to collector Distance (TCD) : 20 cm Concentration : 18 wt./v% Applied Voltage : 20 kV	DMSO		Smooth, uniform and bead free fibers. Fibers were collected at larger rate compared to DMF solvent.
AFD ± SD (nm)		302±85	

Fig.1 SEM images (magnification 5,000× and scale bar = 5 μm) of PVDF nanofibers electrospun from DMF and DMSO.

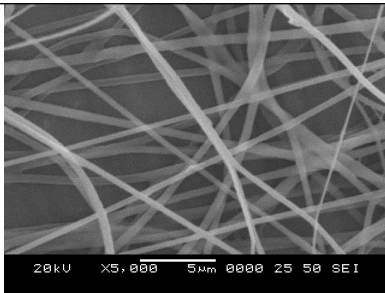
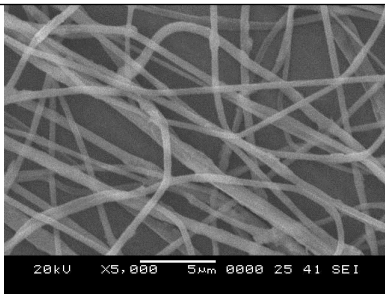
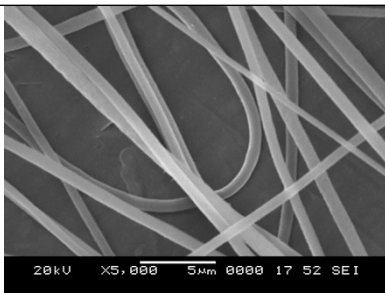
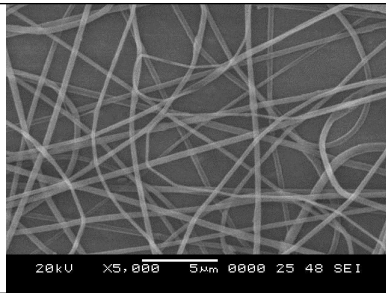
Parameters	Flow rate (mL/hr)	SEM micrographs	Remarks
Solvent : DMSO Tip to collector Distance (TCD) : 20 cm Concentration : 18 wt./v% Applied Voltage : 20 kV	0.5		Smooth, Uniform and bead free fibers were produced.
AFD ± SD (nm)		302±85	
Solvent : DMSO Tip to collector Distance (TCD) : 20 cm Concentration : 18 wt./v% Applied Voltage : 20 kV	0.8		Fibers were randomly oriented and also fibers were not uniformly distributed.
AFD ± SD (nm)		464±92	
Solvent : DMSO Tip to collector Distance (TCD) : 20 cm Concentration : 18 wt./v% Applied Voltage : 20 kV	1.0		Fiber diameter increased drastically. Fibers had a ribbon-like morphology.
AFD ± SD (nm)		632±93	

Fig.2 SEM images (magnification 5,000× and scale bar = 5 μm) of PVDF nanofibers electrospun at different flow rates.

Parameters	Tip to Collector Distance (TCD), cm	SEM micrographs	Remarks
Solvent : DMSO Flow rate :0.5 mL/hr Concentration : 18 wt./v% Applied Voltage : 20 KV	15		Fibers were randomly oriented. Partially dried.
AFD ± SD (nm)		358 ± 87	

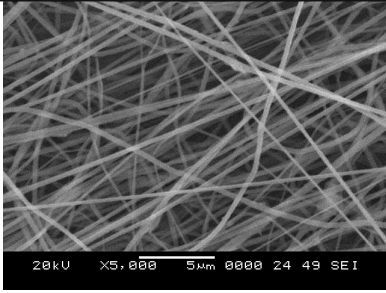
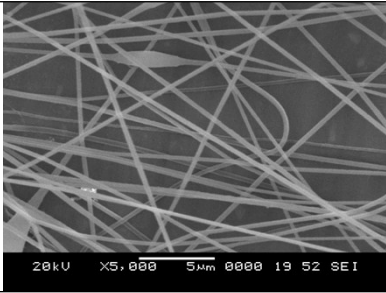
Solvent : DMSO Flow rate :0.5 mL/hr Concentration : 18 wt./v% Applied Voltage : 20 KV	20		Nanofibers produced were smooth, uniform and bead free. Proper drying and collection of fibers.
AFD \pm SD (nm)		302 \pm 85	
Solvent : DMSO Flow rate :0.5 mL/hr Concentration : 18 wt./v% Applied Voltage : 20 KV	25		AFD of fibers decreased. Beads were observed. Collection of fibers was too low.
AFD \pm SD (nm)		289 \pm 53	

Fig.3 SEM images (magnification 5,000 \times and scale bar = 5 μ m) of PVDF nanofibers electrospun at different tip to collector distances (TCD).

References

1. R. Gonçalves, A. C. Lopes, G. Botelho, I. C. Neves and S. Lanceros-Mendez, *J. Polym. Res.*, 2013, **20**.
2. S. Janakiraman, A. Surendran, S. Ghosh, S. Anandhan and A. Venimadhav, *Solid State Ionics*, 2016, **292**, 130–135.