

# The effect of temperature and humidity on electrospinning

S. De Vrieze · T. Van Camp · A. Nelvig · B. Hagström ·  
P. Westbroek · K. De Clerck

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**Abstract** Electrospinning is a process that generates nanofibres. Temperature and humidity affect this process. In this article the influence of humidity and temperature on the formation and the properties of nanofibres are studied using cellulose acetate (CA) and poly(vinylpyrrolidone) (PVP) as target materials. The experiments indicate that two major parameters are dependent of temperature and have their influence on the average fibre diameter. A first parameter is the solvent evaporation rate that increases with increasing temperature. The second parameter is the viscosity of the polymer solution that decreases with increasing temperature. The trend in variation of the average nanofibre diameter as a function of humidity is different for CA and PVP, which can be explained by variations in chemical and molecular interaction and its influence on the solvent evaporation rate. As the humidity increases, the average fibre diameter of the CA nanofibres increases, whilst for PVP the average diameter decreases. The average diameter of nanofibres made by electrospinning change significantly through variation of temperature and humidity.

## Introduction

Electrospinning is a relatively simple fibre-forming process (Fig. 1) and offers a unique method to produce nanofibres

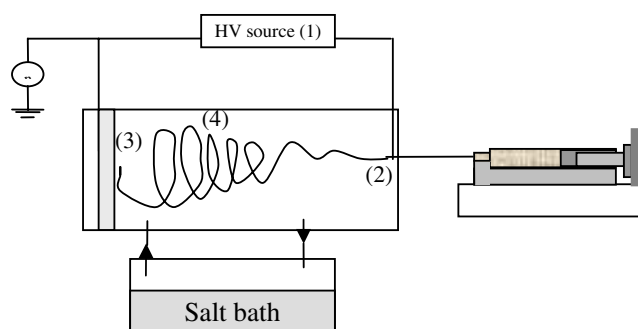
[1–4] from polymer solutions or melts. Electrospinning relies on electrostatic forces obtained by applying an electrical field by means of a DC voltage source (1) between the tip of a nozzle (2) and a collector (3). Once the electrostatic forces overcome the surface tension of the polymer solution at the nozzle tip a jet stream (4) is drawn from the tip of the nozzle. The jet elongates while solvent is evaporating and the so produced nanofibres are deposited on the collector in the form of a random nonwoven structure. Electrospun nanofibres have interesting properties such as high-specific surface area, high porosity and high-absorption capacity, which are useful properties for applications in filtration [5], sensors [6] and (bio)medicine [7].

Despite the apparent simplicity of the electrospinning principle, the process itself is quite complicated because many parameters influence the properties of the obtained nanofibrous structures. These parameters can be merged in three groups, i.e. solution parameters, processing parameters and ambient parameters. Most of the research work published so far has focussed on parameters belonging to the first two groups. However, ambient parameters such as humidity, temperature and ambient gas composition influence the process and the outcome of the process. This was shown in recent studies [8, 9]. In these studies the role of humidity is investigated as a function of nanofibre diameter. With increasing ambient humidity the diameter of polyethylene oxide (PEO) decreases and finally results in beaded fibres. Although, the effect of the temperature of the polymer solution to be electrospun is studied [10, 11], no studies about the influence of surrounding temperature on fibre diameter and nanofibrous structure properties can be found. A higher solution temperature is known to generate thinner nanofibres because of the decrease in viscosity [11]. Recently, a model for electrospinning was developed [12]. This model was developed for aqueous

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S. De Vrieze (✉) · T. Van Camp · P. Westbroek · K. De Clerck  
Department of Textiles, Ghent University, Technologiepark  
907 9052 Ghent, Belgium  
e-mail: sander.devrieze@Ugent.be

A. Nelvig · B. Hagström  
Swerea IVF, 104, SE-43122 Molndal, Sweden



**Fig. 1** Schematic drawing of the special designed electrospinning setup

polymer solutions and showed that relative humidity (RH) had a minor effect on the fibre diameter.

Poly(vinylpyrrolidone) (PVP) and cellulose acetate (CA) are two polymers that have already been the subject of many studies in electrospinning [13, 14]. Both polymers have been electrospun in different solvent systems. For PVP, ethanol [13], while for CA, a mixture of acetone and N,N-dimethylacetamide (DMAc) is commonly used [14] as a solvent.

Controlling the diameter of the fibres is important because of the influence on the properties of the resulting nonwoven. Humidity and temperature play an important role in this aspect [15]. The objective of the present study is to provide experimental observations of the influence of the ambient conditions temperature and humidity on electrospinning of PVP and CA.

## Experimental section

### Materials

Cellulose acetate (Mn 30.000) was obtained from Sigma Aldrich. PVP K90 (Mr 360.000) was purchased from Fluka. Ethanol with a purity of 99.5% was obtained from Swednet. Acetone was of an analytical grade, obtained from Scharlau. DMAc was 99.5% pure and was purchased from Merck. Potassium acetate (Merckx, extra pure), magnesium chloride (Sigma Aldrich 20833-7), magnesium nitrate hexahydrate (Fluka 63084), sodium chloride (Sigma Aldrich S98888) and lithium chloride (Kebolab, purum) were used in salt baths, at their specific saturation concentration.

### Preparation of the solutions and the salt baths

Poly(vinylpyrrolidone) solutions of 7 and 10 wt% were prepared in ethanol. CA solution of 17 wt% in 2:1 v/v acetone:DMAc solvent system was prepared. All solutions were

**Table 1** Saturation concentration at 293 K in water [16]

Lithium chloride	61.2 g/100 mL
Potassium acetate	200 g/100 mL
Magnesium chloride	54.2 g/100 mL
Magnesium nitrate hexahydrate	125 g/100 mL
Sodium chloride	35.9 g/100 mL

**Table 2** The ambient relative humidity for saturated salt baths (% RH) [16]

	283 K	293 K	303 K
Lithium chloride	11.29 ± 0.41	11.31 ± 0.31	11.28 ± 0.24
Potassium acetate	23.28 ± 0.53	23.11 ± 0.25	21.61 ± 0.53
Magnesium chloride	33.47 ± 0.24	33.07 ± 0.18	32.44 ± 0.14
Magnesium nitrate hexahydrate	57.36 ± 0.33	54.38 ± 0.23	51.40 ± 0.24
Sodium chloride	75.67 ± 0.22	75.47 ± 0.14	75.09 ± 0.11

magnetically stirred under room temperature in a closed system for 4 h to allow complete dissolution of the polymers. A number of saturated salt solutions were prepared. The proper amount of salt was dissolved in 500 mL of demineralised water (Table 1). Each saturated salt solution possesses its own specific RH in the surrounding atmosphere. The equilibrium RH of the salt baths at the three different working temperatures can be found in Table 2.

### Equipment

#### Electrospinning

The solutions were electrospun in a closed environment. An electrospinning setup, specific for this purpose was constructed. It is shown in Fig. 1. The setup consists of an infusion pump (New Era Pump Systems Inc) and a high-voltage source (Gamma High Voltage Research, ES50P-10 W/DAM). A grounded aluminium foil was used as collector. There is an electrospinning chamber (a polymethylmethacrylate cylinder), which is connected to a saturated salt bath. A fan inside the connection tubes enables the closed system to have a homogeneous RH and temperature in the closed system. During electrospinning, the fan is shut down to avoid that airflow disturbing the formation and flight path of the nanofibres. The whole setup stands in a temperature controlled room. Humidity and temperature are measured by a transducer, which is connected to a computer (Easyview 5.0.8 software).

#### SEM analysis

The morphology of the electrospun nanofibres was examined using a scanning electron microscope (Jeol JSM-5300)

at an accelerating voltage of 15 kV and software Jeol Semafore 4.02. Prior to SEM analysis, the sample was coated with gold using a sputter coater (Jeol Ion sputter JFC-1100). The average diameter was determined by taking 50 measurements for one set of parameters.

*Nanofibres formation experiments*

Each experiment had a deposition time of 2 min. It was checked that the RH inside the chamber was not significantly changed during this time. The process parameters were set constant and can be found in Table 3. For example, all the experiments executed using 7 wt% PVP solutions at a tip-to-collector distance (TCD) of 12 cm were performed with a flow rate of 3 mL h<sup>-1</sup> and an applied voltage of 10 kV.

The humidity and temperature were selected and held constant during the experiments. For each temperature (283, 293 and 303 K ± 0,2), the different RH (20-30-45-60% ± 1%) was investigated. No investigation was done for CA at RH 20%, because of negative results at higher RH. Prior to electrospinning experiments the polymer

solutions were equilibrated at the same temperature as the electrospinning chamber. After electrospinning, the non-wovens were first evaluated visually for being intact, prior to SEM analysis.

**Results and discussion**

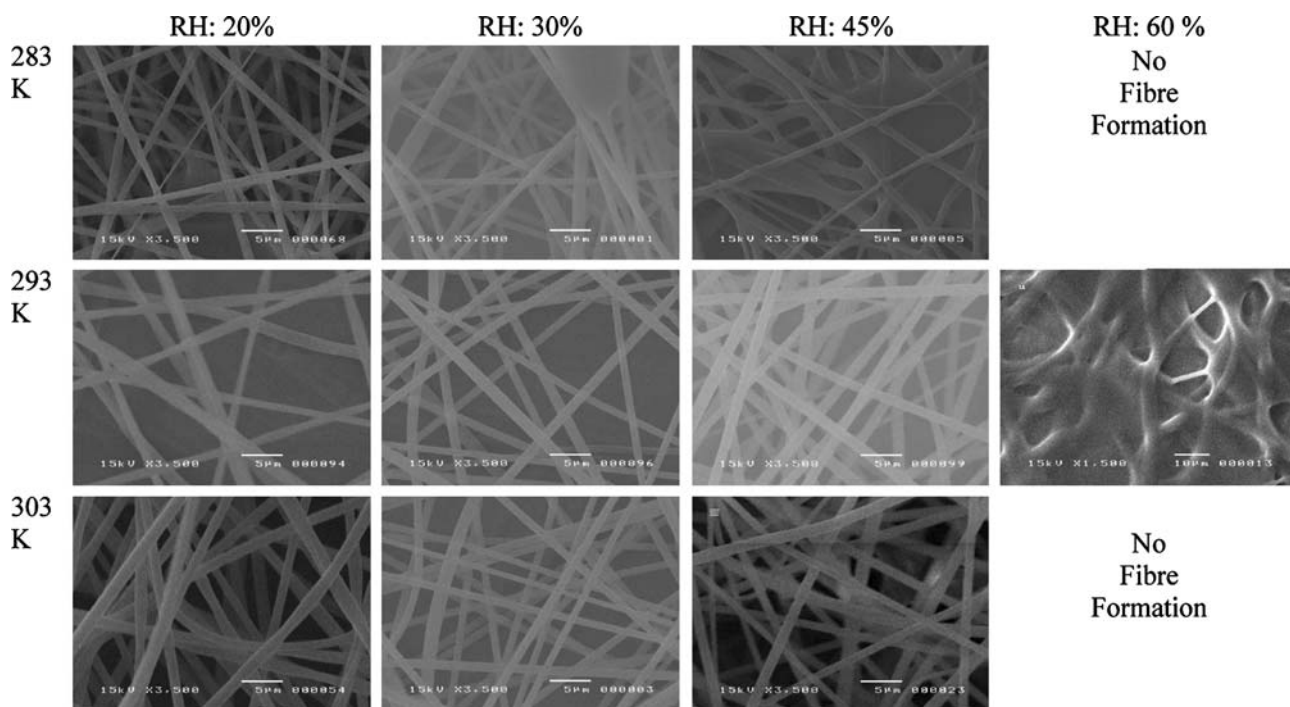
Effects of humidity and temperature on the electrospinning of PVP

A set of nanofibrous structures was prepared from electrospinning using solutions of 7 and 10 wt% of PVP in ethanol. The formation of nanofibres and the morphological properties of these fibres were studied as a function of ambient temperature (283, 293 and 303 K), RH (20, 30, 45 and 60%) and TCD (12 and 18 cm). As an example Fig. 2 shows SEM images of nanofibres obtained from electrospinning experiments using a 10 wt% solution of PVP and a TCD of 12 cm as a function of temperature and RH. Similar results were obtained for a TCD of 18 cm and for the experiments performed with solutions of 7 wt% PVP.

A number of interesting characteristics and relations can be found from the data given in Fig. 2. First it can be seen that it is not possible to obtain well-defined nanofibres at high RH (60%). The main reason is that the PVP-ethanol solution absorbs ambient water during electrospinning. The absorption of water is favoured by a higher humidity because of the higher partial pressure of water in the

**Table 3** The constant set process parameters

	PVP 7 wt%	PVP 10 wt%	CA 17 wt%
TCD 12 cm	3 mL/h–10 kV	3 mL/h–10 kV	1 mL/h–15 kV
TCD 18 cm	3 mL/h–15 kV	3 mL/h–15 kV	1 mL/h–18 kV



**Fig. 2** Resulting fibres for PVP 10 wt%–12 cm TCD

atmosphere. This causes the deposit of a transparent film formed by “fused” fibres on the collector because at higher humidity the absorption of water does not allow to complete the drying process during the time of flight of the polymer solution jet. On a macroscopic level, one can observe a transparent instead of a white PVP deposition. A similar effect of water absorption was already seen in a polystyrene tetrahydrofuran mixture. This resulted in nanofibres with pores [17].

Second, for smaller PVP concentrations (7%) also no fibres were obtained at 45% RH. At lower concentrations more solvent needs to be evaporated during electrospinning, thus the fibre formation process requires more time. However, the time of flight of the jet remains constant thus the fibre formation process cannot be completed due to incomplete evaporation of the solvent. Taking into account that the PVP-ethanol mixture easily absorbs water explains why the fibre formation at lower RH is incomplete. In addition, at the lowest temperature (283 K) no fibres were formed starting from 30% RH. Again the same reasons as above cause this result. At lower temperatures the solvent evaporation is slower and thus the fibre formation process cannot be completed within the time of flight period of the jet.

In electrospinning, a charged jet comes out of a Taylor cone. The charged jet elongates and moves towards a collector in a straight line for a certain distance. Then it begins to bend and develop a spiral path. The repulsive force between charges carried by the jet causes the jet to elongate and become thinner. The elongation and thinning of the charged jet continues until solidification takes place [9]. By solidification is here meant that the solvent is evaporated to such an extent that the inter molecular forces between the polymer molecules are becoming so high that further deformation (stretching) is impossible. The solidification should preferably happen before the nanofibres are deposited on the collector in order to avoid them to fuse together into a more or less continuous film due to the plasticizing effect of remaining solvent.

First described by Dalton [18], the evaporation rate for a pure liquid from a free surface is proportional to the difference of the saturated vapour pressure and the vapour pressure in the surrounding air of the solvent. The saturated vapour pressure of the used solvents and water can be found in Table 4.

**Table 4** Saturated vapour pressure of used solvents (Pa) [19]

Temperature (K)	Ethanol	Acetone	DMAc	Water
283	3,137	15,408	67	1,205
293	5,869	24,609	176	2,313
303	10,467	37,882	365	4,219

The average diameter of the nanofibres decreases at higher humidity (Fig. 3). This was already observed in [9] for PEO nanofibres. But, different to [9], water is not used as a solvent in this study. The evaporation rate of ethanol stays the same at different RH at the same temperature. What differs is the absorption of the surrounding water. This causes the nanofibres to solidify slower. The elongation of the jet can continue longer, resulting in thinner nanofibres.

The experiments show that the PVP nanofibres become thinner at higher humidity. This mechanism stops at a certain higher humidity. At this higher humidity, the nanofibres are ‘fusing’. This can be seen in Fig. 2 at 60 RH. The nanofibres look like as if they flow out, this results of course in a larger diameter of the ‘nanofibres’.

The temperature has an interesting effect on the average diameter of the PVP nanofibres (Fig. 3). At 283 and 303 K, the average diameter of the nanofibres is lower than at 293 K. There are two main and opposing effects as a function of temperature.

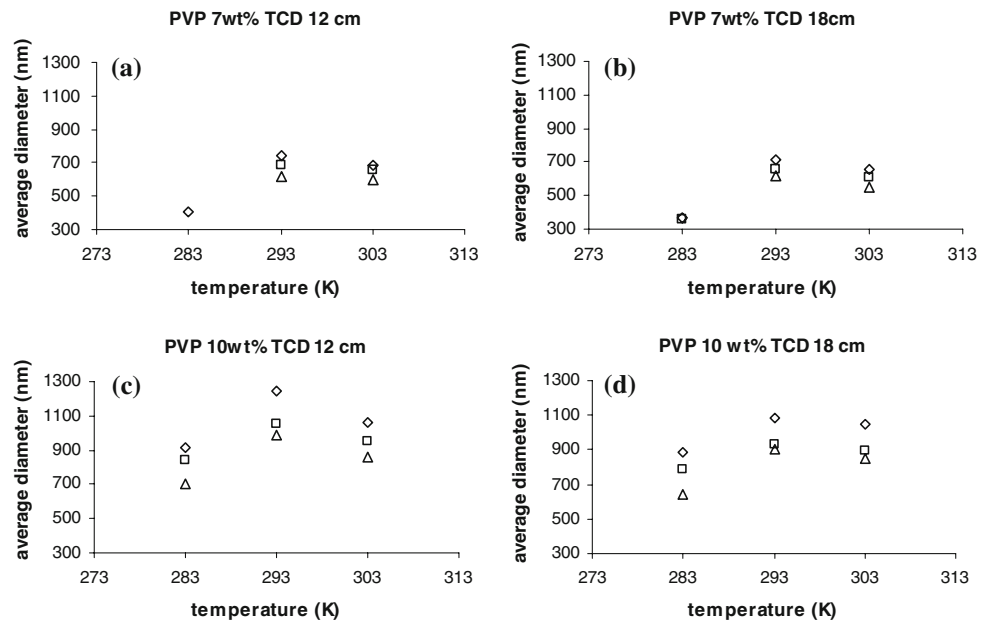
The first effect is the evaporation rate of the solvent, in this case ethanol. This rate decreases exponentially with decreasing temperature. Thus, it takes a longer time for the jet to solidify, continuing the elongation of the jet. The second effect is the rigidity of the polymer chains. The polymer chains have more freedom to move at higher temperatures, resulting in lower solution viscosity. The electric force applied during electrospinning is responsible for the stretching of the polymer solution jet. The stretching is opposed by viscous forces and surface tension (stretching results in creation of new surface area). The lower solution viscosity due to a higher temperature will thus produce higher stretching rate and thinner fibres. This effect was described in [11], where not only smaller diameters were reported, but also a higher chain orientation.

At the lowest temperature (283 K) probably the first effect is dominant over the second effect due to the exponential variation of the solvent evaporation rate with temperature. At the higher temperatures the second effect is dominant because of the exponential decrease of the viscosity as a function of increasing temperature. Both effects result in a decrease of the fibre diameter, thus a smaller diameter of the fibres is obtained at low temperatures due to the first effect and a smaller diameter is also obtained at high temperatures due to the second effect. Intermediate the fibre diameter goes through a maximum.

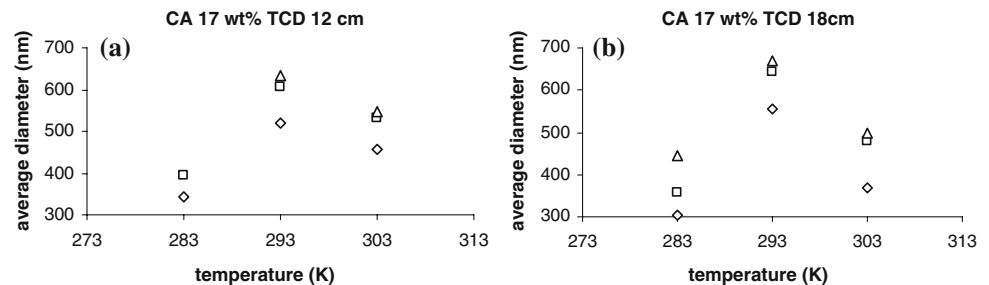
Effects of humidity and temperature on the electrospinning of cellulose acetate

No experiments were performed with the CA solution at a RH of 20%. This was because of the good results

**Fig. 3** The average diameters of the PVP nanofibres. **a** 7 wt% TCD 12 cm **b** 7 wt% TCD 18 cm **c** 10 wt% TCD 12 cm **d** 10 wt% TCD 18 cm  $\diamond$ : RH 20%,  $\square$ : RH 30%,  $\Delta$ :RH 45%



**Fig. 4** The average diameters of the CA nanofibres. **a** TCD 12 cm **b** TCD 18 cm.  $\diamond$ : RH 30%,  $\square$ : RH 45%,  $\Delta$ : RH 60%



already at higher RH (except for TCD 12 cm at 283 K). The average diameter of the obtained CA nanofibres changes similar as the PVP nanofibres (Fig. 4) when changing the temperature. Both mechanisms of evaporation and viscosity result in the decreasing of the average diameter at 283 and 303 K. The formation of a maximum in the fibre diameter as a function of temperatures can be explained in the same way as for PVP in the previous section.

According to Fig. 4 there is a difference with the observations made for PVP. For PVP (and similar, the PEO in [9]), the average diameter decreases when the humidity increases. At higher humidity, these solutions solidify slower, resulting in a decrease of the average fibre diameter. For CA, the average diameter increases with increasing RH (Fig. 4).

When looking at the solution of CA dissolved in acetone:DMAc, adding water results in a fast precipitation of the polymer. If the ambient humidity is high, more water will be absorbed or make contact with the jet during the experiment. It may be speculated that this precipitates the nanofibres in the jet faster, and prohibits the elongation of the jet, resulting in larger average fibre diameter.

An interesting remark can be made about the resulting nonwovens. Only at RH 45 and 60% at 303 K a complete nonwoven was formed. At all the other conditions, there was always a wet spot in the middle. Similar observations were made in normal lab experiments, where CA only formed plain nonwovens when the weather outside (and the lab conditions) was humid. We believe that this effect is also caused by the chemical nature of CA. The higher the humidity, the more water absorption and precipitation of CA will occur.

**Conclusions**

Temperature and RH are two ambient parameters that strongly affect electrospinning. RH makes the nanofibres thicker or thinner, depending on the chemical nature of the polymer. The change in temperature causes two main and opposing effects to change the average diameter. Evaporation rate of the solvent and viscosity of the solution are two opposing mechanism that have an effect on the mean fibre diameter. Other polymer systems will also be affected by temperature and RH.

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