

# Chapter 2

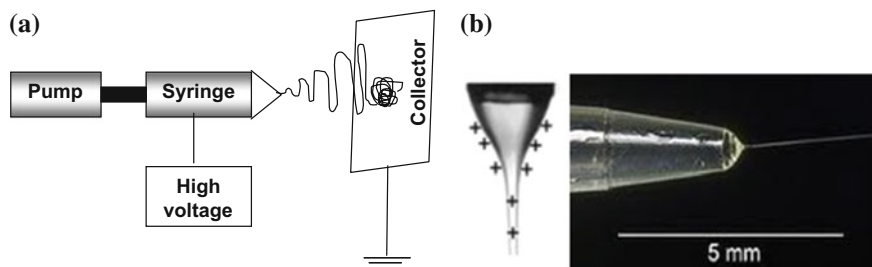
## Review of Literature: Melt Electrospinning

### 2.1 Introduction

Nanotechnology is an interdisciplinary area of science and technology dealing with the materials having dimensions in the range of 0.1–100 nanometres (nm). Recently, nanotechnology has been booming in many important areas such as medicine, engineering, electronics and textiles. In fibrous materials, it has predominantly come up in the form of electrospun nanofibres. The process widely used for the fabrication of nanofibres is electrospinning [1, 2] due to its simplicity and suitability for a variety of polymers. Other processes with the potential for the fabrication of nanofibres include flash spinning, bicomponent spinning, force spinning, meltblowing, phase-separation and drawing. In most of these processes, the fibres are collected as nonwoven random fibre mats known as nanowebs, consisting of fibres having diameters from a few nanometres to hundreds of nanometres.

### 2.2 Electrospinning

Electrospinning is the distortion of a droplet by the application of electric charge. It can be traced back to William Gilbert's published work in 1600 AD [3, 4]. Electrospinning is based on the principle of electrostatic instabilities in liquids or the distortion of a droplet by the application of electric charge [5]. The earliest and still the most widespread practical application of these instabilities is electro-spraying. Electrospinning is mainly employed for the fabrication of fibres with diameters ranging from tens of nanometres to a few micrometres from a number of polymers including both synthetic and natural as well as biopolymers [5–11].



**Fig. 2.1** Schematic diagram of **a** electrospinning device and **b** Taylor cone [12, 13]

The schematic of an electrospinning device is shown in Fig. 2.1a. Generally, in electrospinning, a liquid droplet is delivered to the tip of a capillary. When an electric field is applied, charges accumulate on the surface of the pendant droplet formed at the tip of the capillary and create an instability that deforms the hemispherical droplet into a conical shape, referred to as a Taylor cone [12, 13]. At this stage, there is a competition between the Coulombic repulsion of like charges favouring droplet distortion and surface tension opposing droplet division. When the applied electric field strength is sufficiently high, a liquid-jet is continuously ejected from the apex of the cone and travels towards the grounded plate as a barely visible nanoscale fibre (Fig. 2.1b). The high charge density on the surface of the fine jet leads to electrical instability, making it whip around rapidly. The jet diameter decreases due to stretching (whipping) and evaporation of the solvent.

### 2.2.1 Types of Electrospinning

Electrospinning can be classified into two groups: solution electrospinning and melt electrospinning, based on the nature of the polymer used [14, 15]. In the last two decades, research activities in electrospinning were mainly focused on solution electrospinning [16–19], whereas there are few works reported on melt electrospinning [20–22]. However, solution electrospinning of polyolefins, including polyethylene and polypropylene, has been limited due to high solvent resistance and high electrical resistivity [16].

#### 2.2.1.1 Solution Electrospinning

In solution electrospinning, the polymer is dissolved in a suitable solvent and the resultant solution is electrospun. Almost all the previous research focus on the fabrication of nanofibres by solution electrospinning. As this study was based on melt electrospinning, emphasis is given to describe the process in more detail than solution electrospinning. The readers willing to further investigate solution electrospinning can refer to the literature [10, 15].

### 2.2.1.2 Melt Electrospinning

In melt electrospinning, the polymer is heated to melt for electrospinning. Melt electrospinning has many advantages over solution electrospinning. Melt electrospinning is preferred where the problems of solvent recovery and toxicity are a concern. In melt electrospinning, the dissolution of polymers in organic solvents and the subsequent removal of the solvents are not required. Hence, it is free from the extra cost of solvents, and complex and expensive solvent-recycling equipment. While *in vitro* electrospinning directly onto cells is considered, solution electrospinning fails to meet the criteria. This is due to the toxicity of the solvent used in electrospinning, which needs to be fully removed prior to *in vitro* use [19]. Electrospinning directly onto cells is possible via melt electrospinning as the fibres contain no residual solvent and are free from the toxicity [23].

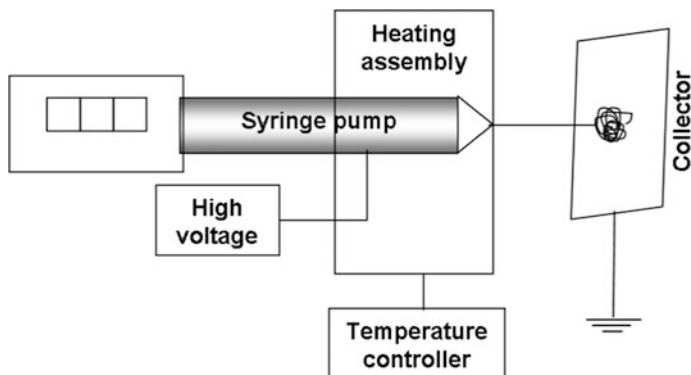
Melt electrospinning can commercially fabricate fibres of polymers with no proper solvent at room temperature such as polyethylene (PE), polypropylene (PP) and polyethylene terephthalate (PET). In the case of PP, one of the most widely used polymers in commercial spinning it is difficult to find a suitable solvent at room temperature. Hence, melt electrospinning provides an edge over solution electrospinning. Melt electrospinning also favours the production of multicomponent systems such as blends and composites, as in many cases no common solvent for all the components may exist [24]. Besides, the modelling of melt electrospinning is much easier as it is free from the complications associated with solvent evaporation.

In spite of these advantages, there has been limited work on melt electrospinning. This may be due to the limiting constraints associated with the process such as (i) the complex equipment used [25]; (ii) the electric discharge problem associated with the equipment design [26]; and (iii) the intrinsic difficulties associated with the polymer, such as high viscosity and low electrical conductivity [24]. The following section focuses on melt electrospinning (including the components, various configurations, polymers used and fibre diameters obtained).

The designing of equipment for melt electrospinning is complex compared to that for solution electrospinning. A melt electrospinning system is composed of the following components: high-voltage power supply, heating assembly, syringe pump, temperature controller and collector (Fig. 2.2).

#### 2.2.1.2.1 High-voltage supply

Generally, direct current (DC) is used as the source of power supply although it is feasible to use alternating current (AC). Very high voltage (usually in the range of 10–30 kV) is required for electrospinning. Lyons claimed that the polarity of the electrospinning system is arbitrary and can be reversed depending on the polymer type and final product [27]. The same production efficiency can be obtained without the damage to the extruder by changing the poles. If the spinneret is grounded and the collector is positively charged, the same electric field strength is created compared to the reverse approach.



**Fig. 2.2** Melt electrospinning equipment

#### 2.2.1.2.2 Heating assembly

The heating assembly is designed to melt the polymer to a suitable viscosity which can be electrospun easily. In all the heating assemblies, the major objective is to melt the polymer by providing heat from various sources such as heating element, heating gun, laser heating and ultrasound heating. The heating assembly works as a reservoir for the polymer melt. Some of the equipments used by various researchers for melt electrospinning are shown in Table 2.1. A spinneret is connected to the heating assembly at one end for the production of nanofibres.

#### 2.2.1.2.3 Syringe pump

During the melt electrospinning process, a syringe pump is used to pump the polymer melt at a constant and controllable rate through the spinneret.

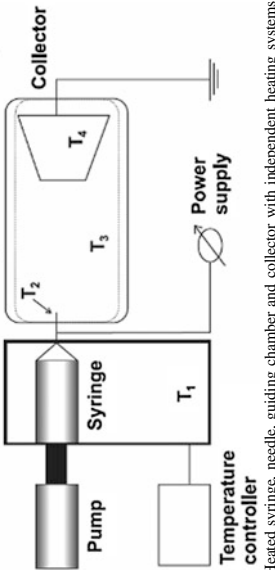
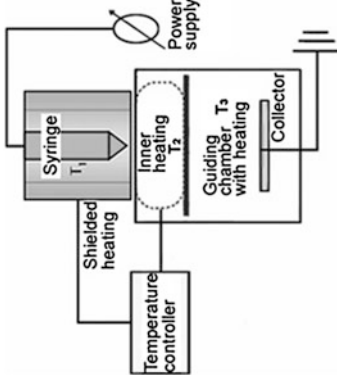
#### 2.2.1.2.4 Collector

The collector or the collecting surface is used to collect the electrospun fibres. Surfaces of different geometries and configurations are used as collectors. The collector is mounted on an insulating stand so that its electric potential can be controlled. In melt electrospinning devices, the high voltage is applied to the collector and the syringe is grounded.

### 2.2.2 Jet Trajectory

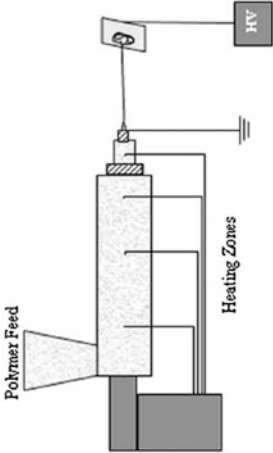
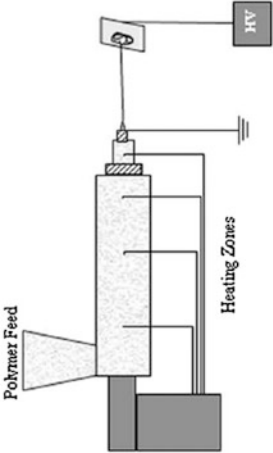
There is a remarkable difference between the trajectories of the jet in solution and melt electrospinning. In solution electrospinning, it has been established by numerous studies and models that the jet undergoes bending or whipping instability, which causes drastic stretching and thinning of the fibre [42]. However, in melt electrospinning the bending instability is either much weaker and localised

Table 2.1 Equipment and polymers used in melt electrospinning along with fibre diameters achieved

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
Low-density polyethylene (LDPE)	Heating rings around piston with temperature controller	MFI 2	315–355 °C	5–32	[28]
PP	 <p>Shielded heating Guiding chamber with heating Collector T<sub>1</sub> T<sub>2</sub> T<sub>3</sub> T<sub>4</sub> Pump Syringe Temperature controller Power supply</p> <p>Heated syringe, needle, guiding chamber and collector with independent heating systems</p>	Mw 195,100 T = 230 syringe T = 280–290 needle	15–40	[29]	
	 <p>Shielded heating Guiding chamber with heating Collector T<sub>1</sub> T<sub>2</sub> T<sub>3</sub> Syringe Temperature controller Power supply</p> <p>Ceramic circular heater for syringe and ring-shaped metal heater for part of spin line</p>	MFI 900	T = 330–410 °C	25–40	[30]

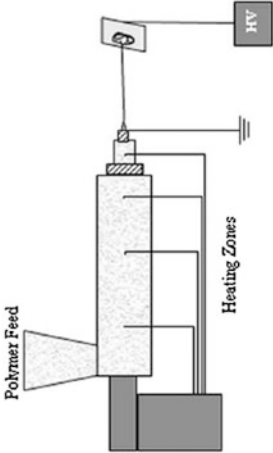
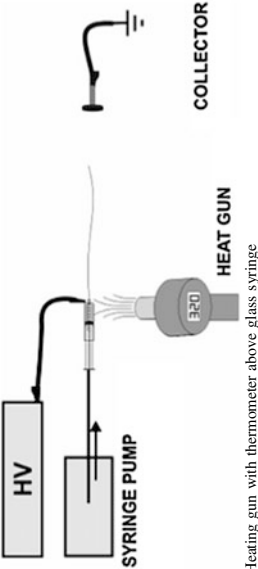
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Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
i-PP	 <p>Brabender table-top extruder with four heating zones</p>	Mw 190,000	T = 200	~ 10	[31]
	 <p>Brabender table-top extruder with four heating zones</p>	Mw 106,000	T = 200 °C	~ 6.9	[31]

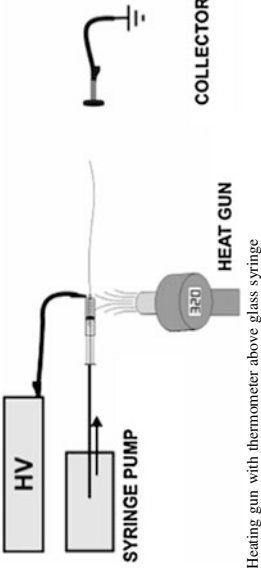
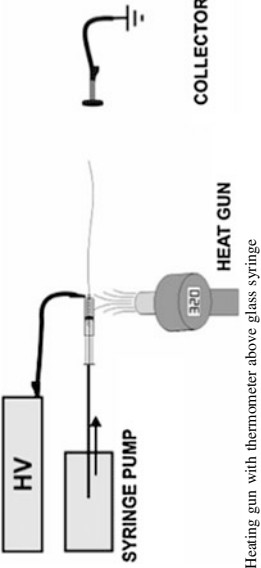
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Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
	 <p>Brabender table-top extruder with four heating zones SS cylindrical chamber with plunger and steel capillary</p>	Mw 12,000	T = 200 °C	~3.5	[31]
	 <p>Heating gun with thermometer above glass syringe</p>	Unknown $\rho$ (Pa s) 23 $\rho$ = zero-shear viscosity	T = 220–240 °C 320 °C	20–180 8.6 ± 1.0	[32] [22]

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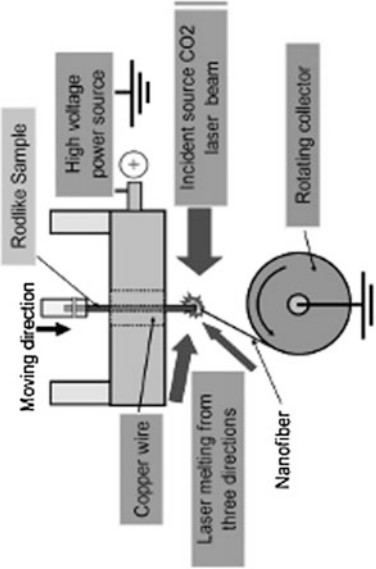
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Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
	 <p>Heating gun with thermometer above glass syringe</p>	$\rho$ (Pa s) 75	270 °C	$35.6 \pm 1.7$	[22]
i-PP-15 + 1.5 % Irgatec atactic PP	 <p>Heating gun with thermometer above glass syringe</p>	$\rho$ (Pa s) 33	270 °C	$0.84 \pm 0.19$	[22]
	Brabender table-top extruder with four heating zones	Mw 19,600	200 °C	~ 21	[31]
	Brabender table-top extruder with four heating zones	Mw 14,000	200 °C	~ 13	[31]

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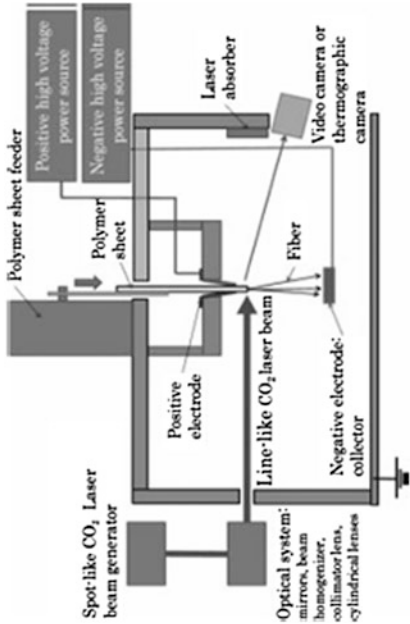


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Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
Polyethylene terephthalate (PET)	 <p>Rod-like polymer samples, CO<sub>2</sub> laser melting polymer from three directions, rotating collector</p>	Intrinsic viscosity (IV) = 0.512–0.706 dL/g	~260 °C	7	[33]

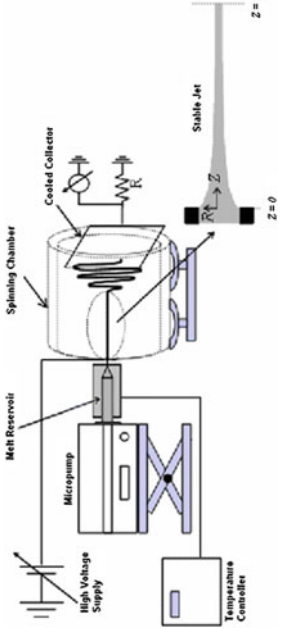
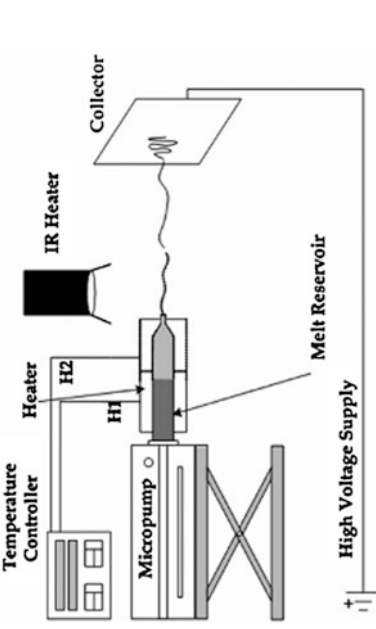
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Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
Polyethylene covinyl alcohol (EVOH)		MFI 4.4 at 190 °C	Laser power 45 W	0.8 ± 0.4	[34]
Poly lactide: Polyethylene covinyl alcohol (EVOH)	CO <sub>2</sub> line-like laser; sheets of polymer; multiple Taylor cones Rod-like polymer samples, CO <sub>2</sub> laser melting polymer from three directions, rotating collector, heated chamber.	poly(lactic acid (PLA) (Mn 96,080 MFI 76.9 at 210 °C) EVOH (MFI 1.6 at 190 °C)	Laser power 12 W	0.845 ± 0.5	[35]

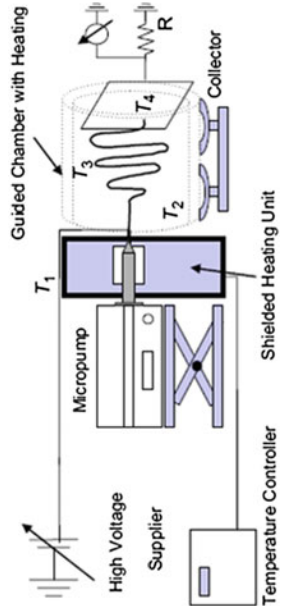
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Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
Nylon-6		MFI 3	250-300 °C	~1	[36]
Nylon-6	<p>Two heating zones around MACOR shielding, cooling collector</p> 	IV = 2.6l dL/g Mn = 5979	270-280 °C syringe 210-220 °C vicinity of spinneret; 29 kV	0.9 & 4	[37]
Nylon-6/12	<p>Electrical heating to syringe, IR heating to the vicinity of syringe CO<sub>2</sub> line-like laser; sheets of polymer; multiple Taylor cones</p>	ρ (g/mL) 1.3	230-300 °C	0.8-2.2	[34]

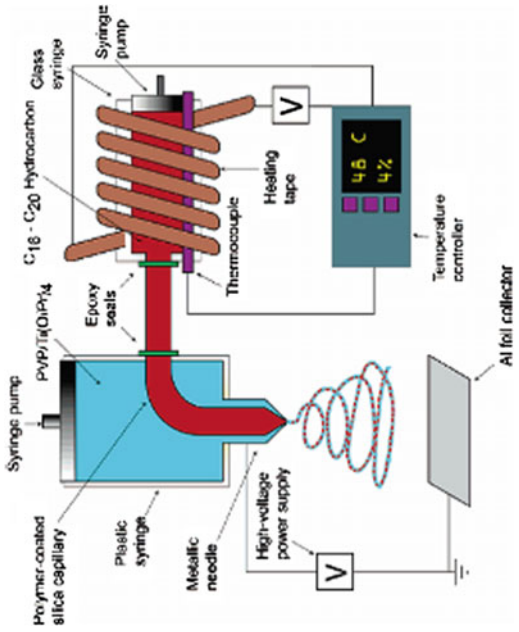
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Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
Poly(lactic acid)		Mw 186,000	200 °C	0.5–17	[24]
	Electrical heating, 4 zones heating equipment with cooled collector polymer resin				
	Same as Nylon 6	Mw 186,000	225 °C	10–25	[38]
	Same as PET	Mw 147,000	Laser: 6–20W	~ 1	[26]
	CO <sub>2</sub> laser melting polymer, rod-like samples with N <sub>2</sub> gas blow to melting area, rotating collector				
	Same as PET				
	CO <sub>2</sub> laser melting polymer, rod-like samples with N <sub>2</sub> gas blow to melting area, rotating collector	Mw 108,000	Laser: 6–8 W	1–2	[26]
Polyalrate	Same as PET				
	Rod-like polymer samples, CO <sub>2</sub> laser melting polymer from three directions, rotating collector	Apparent viscosity 3.358 Pa.S at 340 °C	~ 330 °C	2.5	[33]

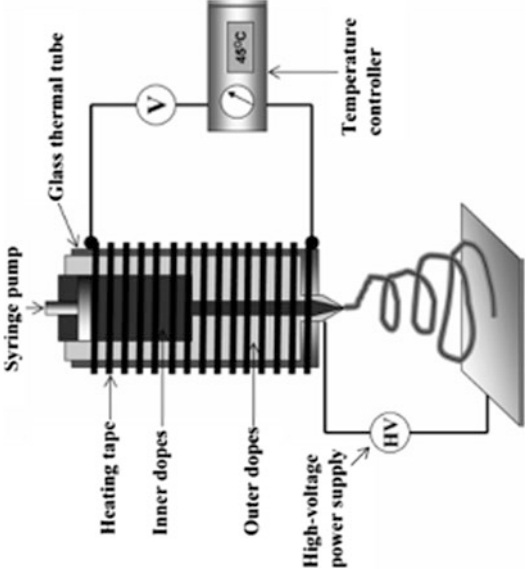
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Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
TiO <sub>2</sub> -PVP (Titanium dioxide-Polyvinylpyrrolidone) sheath loaded with octane		Unknown	68 °C	~0.15	[39]

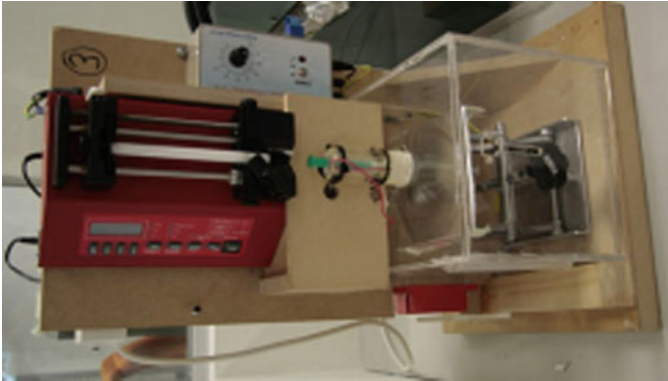
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Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
polymethyl methacrylate (PMMA) with CBT (CVL–bisphenol A–1-tetradeca-nol mixture system)		PMMA (Mw 120,000) bisphenol A, & CB-unknown	45 °C	Core 0.2–0.4 Shell 0.2–0.5	[40]
Polyhexamethylene dodecanediamide	Co-axial system, PMMA electrospun from solvent in outer syringe and CBT from melt in inner syringe heated by heating tape Same as EVOH CO <sub>2</sub> line-like laser; sheets of polymer; multiple Taylor cones	Unknown	Laser power 45 W	0.73 ± 0.2	[34]

(continued)

Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
20% PEG <sub>5000</sub> -block-PCL <sub>5000</sub> + 80% PCL (PEG: polyethylene glycol)		Mw ~ 65,960	90 °C	1.8 ± 0.8	[41]

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**Table 2.1** (continued)

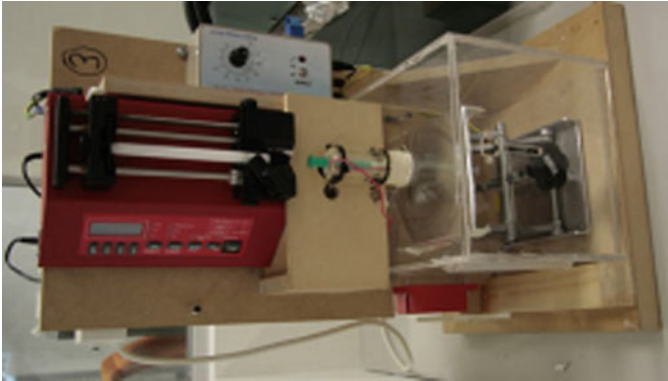
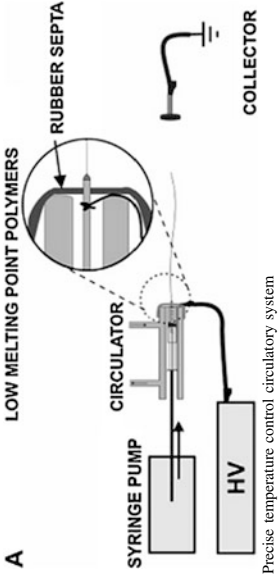
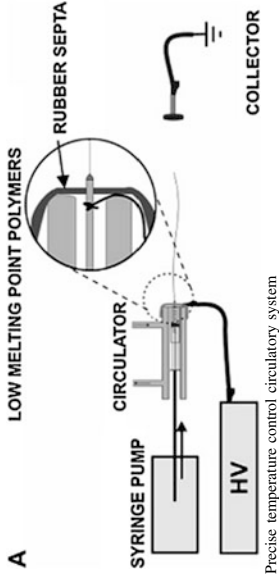
Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
10% PEG <sub>5000</sub> -block-PCL <sub>5000</sub> + 90% PCL		Mw ~ 72,980	90 °C	2.7 ± 2.0	[41]
Heated re-circulating water tank attached to glass jacket where syringe is seated					(continued)



Table 2.1 (continued)

Polymer	Equipment (type & features)	Material property (Mw, MFI, viscosity)(Mw: (Molecular weight); MFI: Melt flow index)	Process parameters	Fibre diameter (µm)	Ref.
PEG47-block-PCL95 + 30% PCL	<p><b>A</b></p>  <p><b>LOW MELTING POINT POLYMERS</b></p> <p><b>SYRINGE PUMP</b></p> <p><b>CIRCULATOR</b></p> <p><b>HV</b></p> <p><b>COLLECTOR</b></p> <p>Precise temperature control circulatory system</p>	$\rho = 49$	90 °C	$2.0 \pm 0.3$	[22]
PEG47-block-PCL95 + 30% PCL (dual collector)	<p><b>A</b></p>  <p><b>LOW MELTING POINT POLYMERS</b></p> <p><b>SYRINGE PUMP</b></p> <p><b>CIRCULATOR</b></p> <p><b>HV</b></p> <p><b>COLLECTOR</b></p> <p>Precise temperature control circulatory system</p>	$\rho = 49$	90 °C	$0.27 \pm 0.1$	[22]

[24] or not present at all [42]. The weak or lack of bending instability can be attributed to the high viscosity of the polymer melt [13]. In addition, the high surface charge density is also responsible for the occurrence of the bending instabilities in solution electrospinning. As in melt electrospinning, the majority of the polymers are electrically insulating, there is very little or no such instability [31].

Bending instabilities help in the reduction of the jet diameter to the order of nanometres, resulting in nanofibres. Suppression of these instabilities in melt electrospinning will appreciably prevent the stretching and hence result in a fibre diameter mainly in the micrometre range. When the electrospinning temperature is low, fast solidification of the jet is believed to suppress the instability in melt electrospinning [24]. It was found that in melt electrospinning with higher voltages, the bending instability was stronger and more irregular, similar to solution electrospinning [43]. In addition, at lower flow rates, the whipping motion became more vigorous.

## 2.3 Research on Melt Electrospinning

The initial work on melt electrospinning was presented in a patent approved in 1936 from Charles Norton from Massachusetts Institute of Technology where an air-blast was used to assist fibre formation [44]. The first scientific publication was reported by Larrondo and Manley [32, 45, 46] using thermoplastic polymers in 1981 as a three-paper series. Of the three papers, only the first one actually demonstrated the formation of fibres from a pendant molten droplet of PE and PP. In this work, they fabricated fibres of greater than 50  $\mu\text{m}$  in diameter from PP of 0.5–2 MFI at collector distances of 1–3 cm and applied voltages of 3–8 kV/cm.

The second paper was the modelling of the flow field in an electrostatically drawn jet using viscous silicone oil in the place of polymer melts. The third paper illustrated the deformation effects of an electrostatic field on a molten polymer droplet of PE or Nylon-12 suspended in hexane. Until these works, it was believed that only fluids and polymer solutions could form such jets under the effect of electrostatic field. This work established the fact that a molten polymer can be drawn electrostatically and was in compliance with Taylor's formulae for other fluids [13]. Subsequently, several researchers worked on melt electrospinning with several configurations using different polymers, which are highlighted in the following section.

### 2.3.1 *Equipment Used*

A variety of equipment has been used by researchers which may differ by the modes of heating, heating system, material feed and collector design [47]. When designing melt electrospinning equipment, the important thing is the selection of a

suitable heating system. A variety of equipment with different means of heating, such as electrical heating by heating elements [24, 28, 31]; laser heating devices [26, 33]; and heating by circulating fluids (i.e. heated air by heat guns [22, 48], circulating oil), have been used. Among these, electrical heating is widely adopted for melt electrospinning as it is easier to design and use [49]. The electrical heating system should be isolated from the high-voltage power supply in order to prevent potential disruptions with the electric field. Therefore, in many experiments high voltage is applied to the collector and the spinneret is grounded. However, a system has been described by Joo and Zhou [50] which overcomes the issue of isolation from the high-voltage source.

Electrospinning by laser or laser electrospinning has been used by several researchers [42]. A solid polymer sample in the form of a rod or sheet is fed to the laser melting zone, where it is melted. The lasers uniformly and locally heat the sample at the interaction point with the polymer. The polymer is heated instantly and locally in the laser melting system and therefore can avoid thermal degradation. The electric discharge problem associated with the conventional melt electrospinning can be prevented in laser electrospinning as heating is performed from a distance. The other advantage of laser melting is that the fibres can be fabricated from polymers containing impurities, as nozzles are not used in this equipment. In laser electrospinning, there is no report regarding the temperature of the heating zone, but the power intensity has been reported.

Another approach is heating by a heat gun, which is a simple, safe and inexpensive approach for melt electrospinning. The polymer in powder form is put into a syringe and heated to the appropriate temperature directly by a heat gun. This can prevent the accidental surface contamination of biomaterials with high-temperature circulating fluids. The issues of overheating and undesirable degradation of the polymers with low melting point can be avoided by the use of circulating air from heat guns. The equipment used by researchers in melt electrospinning experiments along with their features is summarised in Table 2.1.

### 2.3.2 *Polymers Used*

Melt electrospinning can be applied to a wide range of polymers. The first published work on melt electrospinning of PP was by Larrondo and Manley [32] two decades ago. In subsequent work, researchers diversified the materials that can be used in melt electrospinning [42]. The fabrication of micro- and nanofibres of various polymers such as polylactic acid (PLA) [24, 26], PP [14, 22, 31, 48], low-density PE (LDPE) [28], PET [42], polyalirite [33], polycaprolactone (PCL) [51] and PEG<sub>47</sub>-*b*-PCL<sub>95</sub> [22, 51] by melt electrospinning has been reported by several researchers. The list of polymers used in melt electrospinning has been summarised in Table 2.1.

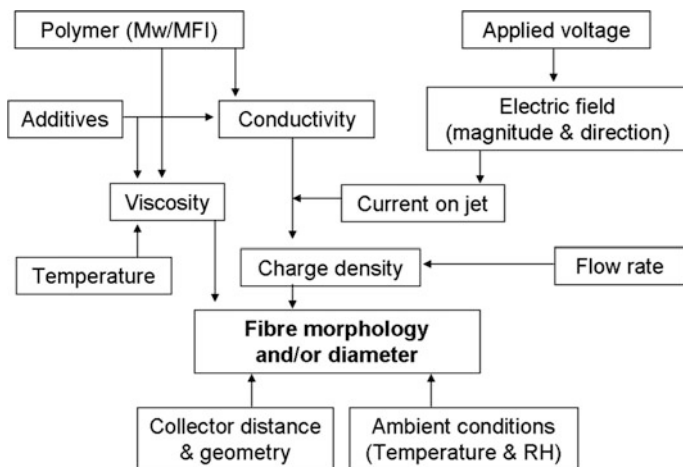
## 2.4 Fibre Diameter and Influencing Factors

In melt electrospinning, a range of obtainable fibre diameters have been explained for a variety of polymers. In the majority of recent work, the melt electrospun fibres are in both nanometre and micrometre scales (Table 2.1). The fibre diameter can be controlled by different parameters related to the polymer (i.e. molecular weight [31], MFI [23] and stereoregularity [31]); and the process (applied voltage, collector distance, nozzle diameter, flow rate [31] and temperatures of the spinneret and the spinning region [24]). Apart from these, the ambient conditions such as the temperature and relative humidity (RH) also affect the fibre diameter. The list of factors influencing fibre diameter is shown in Fig. 2.3.

In melt electrospinning, accurate dispensing of the polymer to the spinneret and maintaining the homogeneity of the polymer melt are important to achieve consistent fibre diameter [42]. Liu et al. [14] performed an orthogonal design study to find the dominant factors (such as electric field, collector distance, temperature and polymer MFI) affecting the fibre diameter in melt electrospinning. It was found that the MFI of the polymer was the most influencing parameter affecting average fibre diameter and standard deviation (SD). The importance of the parameters was listed in the following order:

For average fibre diameter : MFI > applied voltage > collector distance > temperature

For SD : MFI > temperature > collector distance > applied voltage.



**Fig. 2.3** Schematic of the factors influencing fibre diameter and morphology in melt electrospinning

### ***2.4.1 Effects of Molecular Weight/MFI***

Until now it has been found that molecular weight is the most important parameter governing the fibre diameter in melt electrospinning [31]. Higher molecular weight polymers possess higher degrees of chain entanglement and hence lower MFI [52]. As a result, coarser fibre diameters will be formed from these polymers. It has been shown that high-quality fibres can be best made using molecular weights between 40,000 and 80,000 g/mol, much lower than those of the polymers used for engineering applications [49]. Lyons et al. [31] showed that as the molecular weight decreased continually, the fibre diameter was decreased. In addition, the stereoregularity of the polymeric chain affects the fibre diameter. For example, isotactic polymers of PP (iPP) produced finer fibres than atactic polymers [31]. This was because in atactic PP the methyl group is randomly positioned with respect to the main backbone and therefore it hardly crystallises. The inability to closely pack due to the steric hindrances may result in large fibre diameters.

### ***2.4.2 Effect of Viscosity***

The viscosity of the polymer melt is at least one order higher than the viscosity of polymer solution [17, 18]. Higher temperature reduces the viscosity, which results in the fabrication of finer fibres [53]. There are several ways to reduce the viscosity of polymers, such as increasing the process temperature and addition of additives such as rheology modifiers [54, 55]. Due to the high viscosity of the polymer melt, sufficient time is required for stabilisation of the flow rate, especially when a small diameter and/or lengthy spinneret are used. It was found that the higher polymer MFI produced smaller diameter fibre due to the low viscosity [26]. Dang et al. used LDPE with a very low MFI (i.e. 2), which was challenging for melt processing and employed very high temperatures for melt electrospinning.

### ***2.4.3 Effect of Applied Voltage***

The external electric field is a fundamental requirement for electrospinning and an important factor governing the fibre diameter. In melt electrospinning, the only force which is responsible for fibre stretching is the electrostatic force, as there is no solvent evaporation. Melt electrospinning involves the stretching of predominantly non-conducting liquid under the influence of an applied electric field. Several studies have shown that the increase in the applied voltage decreases the fibre diameter [24, 31]. However, a few others have shown a different trend, of a decrease followed by an increase in fibre diameter [56]. The increase in applied

voltage reduces the fibre diameter in melt electrospinning as the polymer is subjected to higher dragging force [31, 57, 58]. However, after a threshold value of the applied voltage, the size of the Taylor cone decreases and the fibre diameter starts increasing. This is because after a threshold value, the applied voltage pulls the polymer fluid faster than the flow rate can replace it and hence the size of the Taylor cone diminishes.

#### ***2.4.4 Effect of Collector Distance and Type***

Change in the collector distance influences the fibre diameter. In some experiments, collector distances of <10 cm [22, 59, 60] were used, whereas others were >10 cm [23, 30]. It was found that at shorter collector distances the fibre diameter was coarser, as the jet is not sufficiently thinned before being collected. In addition, a small effect of collector distance on fibre diameter was observed [26, 35]. Interestingly, a trend of decrease followed by an increase in the fibre diameter was observed by increasing the collector distance [28]. As cooling of the fibre is essential for solidification, shorter collector distances can result in slightly molten fibres being deposited, with fusion of the fibres at the crossover point.

Various types of collectors are being used in melt electrospinning. For example, a scanning electron microscopy (SEM) aluminium stub covered with aluminium foil and Petri dishes were used as collectors and placed at variable distances of 5–30 cm [23]. It was found that the quality of the fibres collected onto the aluminium stub was much better than the fibres collected on Petri dishes. In another approach, glass microscope slides were used as the collector with or without reactive molecules on their surface. The surface modification affected the manner in which the fibres were collected. In addition, collectors such as rotating drums or plates can be used to achieve various patterned structures.

#### ***2.4.5 Effect of Flow Rate***

Polymer flow rate indicates the amount of polymer melt flowing per unit time. It was found that increase in the flow rate caused more polymer melt being drawn from the nozzle and hence produced coarser fibres [42]. The flow rate used in the melt electrospinning is significantly lower than that of solution electrospinning. A low flow rate is essential for getting good quality fibres with fine diameter in melt electrospinning [61]. This may be due to the increased charge density at lower flow rates lowers the fibre diameter. The effect of spinning volume per unit time was studied by placing the polymer chip directly on the spinneret orifice during melt electrospinning [31]. As no flow rate was applied, the volume of polymer was continuously reduced, as the fibres were continually fabricated. This resulted in a

decrease in the size of the Taylor cone continuously, leading to a decrease in the fibre diameter.

#### ***2.4.6 Effect of Spinneret Size***

The diameter of the spinneret used for melt electrospinning is higher compared to that for solution electrospinning, as the flow of high viscous melt through a smaller diameter orifice is challenging. There is a strong correlation between the fibre diameter and the spinneret size [24]. However, it was observed that the average fibre diameter decreased initially as the capillary diameter decreased. Once the diameter reached a critical value, the fibre diameter started increasing significantly [28]. This is because the extremely small orifice resulted in high pressure being required to form the Taylor cone, which in turn led to increased flow of the polymer melt to the apex of the capillary, resulting in thicker fibres.

#### ***2.4.7 Effect of Temperature***

The nozzle temperature is one of the most important parameters affecting fibre diameter. It was found that as nozzle temperature increased, both the average fibre diameter and the SD decreased [24, 28]. This is because the increase in the melt temperature decreases the viscoelasticity and thus the final jet gets thinner. Nozzle temperatures above certain values are not applicable, as there are chances of thermal degradation and the elongational viscosity of the melt is not high enough to maintain a continuous jet.

#### ***2.4.8 Effect of Ambient Parameters***

The fabrication of electrospun fibres from polymer melt can be influenced by ambient conditions such as relative humidity (RH), vacuum conditions, surrounding gases and temperature. There have been very few studies on the effect of ambient parameters on the fibre diameter in melt electrospinning. The ambient conditions from the tip of the syringe to the collector can also influence the morphology and diameter of electrospun fibres [37]. It was found that high ambient temperatures in the vicinity of the spinneret made the electrospinning process quicker and produced more uniform fibre [24].

### ***2.4.9 Effect of Electrical Conductivity***

The electrical conductivity of a polymer is an important parameter which determines the charge density and is closely related to the attenuating force during electrospinning. The net charge density is mainly affected by the applied electrostatic field and the electrical conductivity of the polymer solution or melt. Almost all the polymers used in solution electrospinning are conductive except few dielectrics. However, the majority of the polymers used in melt electrospinning are not electrically conductive, which makes the process difficult for fibre fabrication [49]. For example, the solution of PP in Decalin had an extremely low electrical conductivity compared to other polar solvents, which made the initiation of electrospinning difficult [62].

### ***2.4.10 Other Parameters***

Kilic et al. [63] studied the effect of polarity on the melt electrospinning process. It was found that inverting the polarity (when the collector is charged and needle grounded) lowers the production rate and results in coarser and less homogenous surfaces. Kessick et al. [64] performed electrospinning by using both AC and DC potential and found that AC potential can reduce the amount of fibre whipping associated with DC potential. In a laser-based melt electrospinning device, the laser power affects the fibre diameter. It has been found that the fibre diameter is large at lower laser power, but it decreases and then becomes constant with increasing laser power [26].

## **2.5 Surface Morphology**

Surface morphology of the fibres influences the performance of the fibres. Melt electrospun fibres generally appear smooth and cylindrical, with thread-like or ribbon-like structure [14, 29]. There is no solvent evaporation in melt electrospinning, which may lead to inconsistencies on the fibre surface. The smooth surface is due to the partial solidification which occurs rapidly as the jet leaves the spinneret. The material properties affecting the fibre surface morphology are the polymer molecular weight/MFI and the tacticity. Theoretically, the factors affecting melt electrospinning process can also affect fibre morphology. A minute change in the operating parameters can lead to considerable change in the fibre morphology. The surface morphology of the fibres is also affected by the environmental conditions such as RH and temperature.



Melt electrospun fibres can be deposited either randomly or in an oriented manner to give an aligned nanofibre array. Patterning of the fibres for specific applications can be achieved by altering the collector geometry. Until now only a limited amount of work has been done on the patterning of melt electrospun fibres [20, 51]. In some cases, the fibres can consist of two components to form side-by-side or bicomponent structures. Melt electrospinning is poor in generating oriented fibres over significant gaps, probably due to the focused nature of the deposition and elongated jet [22]. The formation of islands of fibres was a common phenomenon while melt electrospinning directly onto metallic collectors, but it did not appear during in vitro investigations [23].

## 2.6 Properties of Melt Electrospun Fibres

### 2.6.1 Mechanical Properties

Nanofibres are used for a wide range of applications such as filtration, tissue engineering, reinforcement composites and micro/nano-electro-mechanical systems (MEMS/NEMS) [65]. During the service life of these products, various forces are exerted on the fibres, which may result in deformation and even lead to permanent failure. Therefore, the mechanical characterisation of nanofibres is essential to assure the product's serviceability. As nanofibres are very fine in diameter, commercial equipment cannot be directly used for mechanical testing due to the following reasons: (a) difficulty in manipulation (due to their smaller size, handling of ultrafine fibres without causing any damage is extremely difficult); (b) non-availability of a suitable observation mode; (c) non-availability of an accurate and sensitive force transducer (as the force required to break the nanofibres is in the nano-Newton range, commercial equipment are not suitable for nanofibres); and (d) difficulty in the preparation of single-strand nanofibers [66].

A variety of techniques are available for the mechanical characterisation of nanofibres such as the tensile test, three-point bending test [42], resonance frequency measurements [67], atomic force microscopy (AFM), based cantilever bending experiments [68, 69] and nano-indentation [70, 71]. Commercial mechanical grippers are not suitable for nanofibres; due to the small size of nanofibres, it is difficult to avoid the slippage or breakage of the fibre at the grips [72]. Another problem associated with tensile testing is misalignment of the sample axis with respect to the load direction, which may lead to premature sample failure. The most convenient method of avoiding these problems is to collect the fabricated fibres on a frame or paper that can be directly mounted on the tensile tester. Tensile testing of nanofibres can be carried out using the Nano Bionix System (MTS, USA) [73, 74].

### 2.6.2 Thermal Properties

The thermal properties of melt electrospun fibres have been investigated by several researchers [21, 24]. Differential scanning calorimetry (DSC) studies have shown that the degree of crystallinity of PLA fibres produced by melt electrospinning is very low [24]. Thermal investigations of PEG/cellulose acetate composite have shown that the fibres have good thermal stability and reliability [21]. The fibres imparted balanced thermal storage and release properties and the thermal properties were reproducible after 100 heating–cooling cycles.

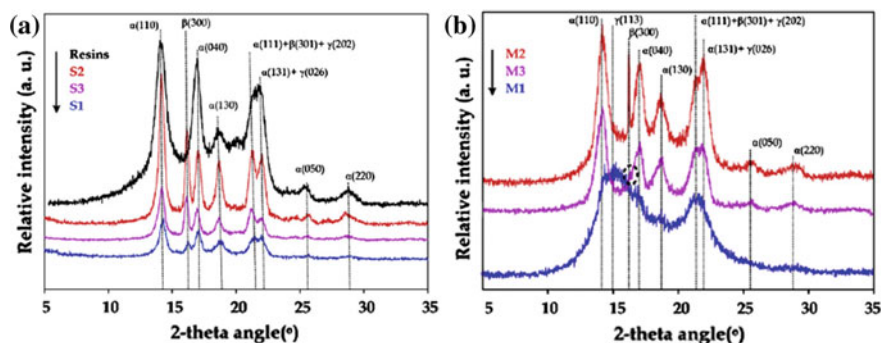
### 2.6.3 Crystalline Properties

Crystalline properties are one of the most important factors determining the mechanical properties of the fibre. In general, the elastic modulus of the fibres increases with the degree of crystallinity and results in better mechanical properties. The solvent evaporation in the case of solution electrospinning leads to crystal formation by the solidification of aligned polymer chains [75, 76]. However, the melt electrospun fibres are reported to possess a very low degree of crystallinity and less stable crystal structure due to rapid quenching by electrohydrodynamically driven air flow near the polymer jet [42]. In solution electrospinning, the solidification rate of solvent evaporation and in melt electrospinning the cooling rate of the jet influence the crystalline properties of the fibres [77].

X-Ray diffraction (XRD) studies have shown that as-spun PLA melt electrospun fibres with 10  $\mu\text{m}$  diameter are mostly amorphous in nature with no distinct crystal peaks [24]. On annealing, the peak strength gradually increased and the peak shifted towards higher temperature depending on the degree of annealing. In another study, high crystallinity values have been reported for LDPE [28].

It has been found that melt electrospun PP fibres cannot maintain oriented structures at room temperature. Therefore, in order to keep the aligned structures at room temperature, a collector cooled below the  $T_g$  of PP was used [24]. In another approach, the results from DSC and XRD of PP showed  $\beta$ -form crystals present in solution electrospun fibres, whereas  $\alpha$ -form crystals were prevalent in melt electrospun fibres (Fig. 2.4) [37]. The fibres changed their morphology to  $\alpha$  and  $\gamma$ -form crystals after annealing.

Cho et al. [37] fabricated Nylon 6 fibres via solution (with formic acid) electrospinning at room temperature and melt electrospinning at elevated temperature. The crystal structure and effect of annealing on the structure were studied by various techniques. The results obtained from DSC, XRD and Fourier Transform Infrared (FTIR) indicated that the melt electrospun Nylon 6 fibres predominantly exhibited the metastable  $\gamma$ -crystalline forms and low molecular orientation; whereas the solution electrospun fibres showed both  $\alpha$  and  $\gamma$ -form crystals and higher molecular orientation. When the annealing temperature was high, the metastable



**Fig. 2.4** Figure XRD reflection patterns for PP polymers and PP fibres obtained by **a** solution electrospinning and **b** melt electrospinning

$\gamma$ -crystals in melt electrospun fibres easily transformed into stable  $\alpha$ -form crystals and the crystals in solution electrospun fibres exhibited higher thermal stability.

### 2.6.4 Other Properties

The PP webs prepared both from solution and melt electrospinning exhibited superhydrophobicity with a water contact angle of  $151^\circ$ , which is substantially higher than those of a commercial PP nonwoven web and a compression moulded PP film [37]. The enhanced hydrophobicity can contribute to excellent barrier performance without losing permeability when applied to protective clothing.

A number rheology modifier has been successfully used for reducing the viscosity of polymer melt. This included metallic stearates, boron nitrides, fluoropolymers, siloxanes and liquid crystal polymers [78]. The commercially available siloxanes and fluoropolymers have high molecular weights. Low molecular weight additives include fatty acids, silicon oils, mineral oils and wax, which are well established in the polymer processing field. It has been found that the low molecular weight additives can provide good results in extrusion at a much lower level of addition than their high molecular weight counterparts. Although the mechanism of viscosity reduction is still under debate, the external lubrication effects of the blend during die flow are generally agreed on [78, 79]. In the case of polyolefins, the main additives used for reducing the viscosity are sodium oleate (SO) [80, 81], polyethylene glycol (PEG) [82, 83] and polydimethylsiloxane (PDMS) [84, 85].

Although the rheological properties of the polymer melt strongly influence the melt electrospinning, a limited number of studies have give attention to the study of rheological properties. It was found that the average viscosity of PLA melt was one to two orders of magnitude higher than that of PLA/chloroform solution [24]. PLA did not exhibit strong shear thinning over a wide range of shear rates.

## 2.7 Conclusion

This chapter described the electrospinning process, which is widely used for the fabrication of nanofibres. Although, other processes such as flash spinning, bicomponent spinning, force spinning, meltblowing, phase-separation and drawing with the potential for the fabrication of nanofibres exist, electrospinning is widely accepted due to its simplicity and suitability for a variety of polymers. Nanotechnology has become a major area of research in many scientific organisations due to its wider applications in medicine, engineering, electronics and textiles. Different types of electrospinning such as solution and melt electrospinning have been discussed in this chapter. A special emphasis has been given to melt electrospinning, as this research is based on it. Furthermore, the characterisation of melt electrospun nanofibres has also been discussed. This chapter is essential for the readers to understand the principles of melt electrospinning.

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