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A Review on Electrospun Short Fiber Production

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Abstract: Nanotechnology has become the interest of researchers in recent years for their unique properties of submicron scale materials. Nanotechnology also consists of nanofibers made from natural or synthetic polymers which can be electrospun into ultra-thin continuous fibers. These nanofibers are versatile as it can be found in various applications such as in filtration, affinity membranes, tissue engineering, biosensors, scaffolds, drug delivery and fiber reinforcement. Over the years, many researchers have reported various methods used to produce short electrospun fiber by means of ultrasonication, mechanical cutting, UV cutting, precipitation method, microtome cutting, cryo-microcutting, cryogenic milling, ball milling, and razor blade cutting under liquid nitrogen. The aim of this paper is to provide a review on electrospun short fiber production which elaborates more on the scission methods of the continuous as-spun fibers. The literature shows that several methods have been proposed and utilized, with varying degrees of success. Overall, it can be concluded that further research is needed to fully understand the complexities of this area and to develop a more effective approach.

Keywords: Electrospinning, short fiber, scission, nanofiber

1. Introduction

Short polymer fibers with a diameter and length of submicron are intriguing the research community due to its unique attributes which provides opportunities for a various application. It can be used as reinforcements for brittle materials by altering the mechanical propeties of a composite material as well as ease of molding. Short fibers or more specifically, short nanofibers, can be applied even in the biomedical field such as drug delivery capsules, tissue engineering scaffolds, filtration devices, membranes, sensors[1].

Among various methods for fiber production, electrospinning is a relatively efficient way of producing continuous as-spun ultra-thin fibers with diameters down to submicron scales. The lengthy fibers requires a the secondary process to convert it into short fibers which is neither totally simple nor cost-effective and the electrospun ultrafine fibers' relatively low tensile strength[2] makes it very difficult to produce fibers with less than 200 aspect ratio by means of physical alteration [3]. Electrospinning can be used to produce fibers using natural or synthetic polymer such as collagen [4], gelatin [5], chitosan [6], poly(l-lactic acid) (PLLA) [7], poly(glycolic acid) [8], poly-ε-caprolactone (PCL) [9]. Electrospinning method requires an electric field with a high voltage to electrically charge viscous polymer solution in order to be jetted by electrical forces where the polymer will melt and the solvent molecules will evaporate prior to reaching the collector [10]. As it is then deposited on a collector, it is collected as a thin sheet of fiber mat. Further scissioning of these long thin fibers are then carried out by various methods but more predominantly, ultrasonication[11]–[16]. Among various methods available, some which are discussed in this literature include mechanical cutting [17], Ultra-Violet (UV) cutting [18], microtome cutting [19], micro cutting under liquid nitrogen [30], cryogenic milling [20], ball milling[21] and razor blade cutting under liquid nitrogen.

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There are a limited number of papers available regarding electrospun short fiber production and its applications using various popular methods as highlighted in this paper. Other review papers concentrate more on the parameter of electrospinning and its effects on the production of electrospun fiber along with its purposes [22]. Therefore, the objective of this paper is to review some of the unpopular methods used for short fiber production such as mechanical cutting, UV cutting, precipitation method, microtome cutting, cryo-microcutting, cryogenic milling, ball milling, razor blade cutting under liquid nitrogen as well as one of the most popular methods, ultrasonication.

2. Scission Methods

2.1 Mechanical Cutting

A technique for fabricating the short electrospun fibers could be by mechanical cutting. As reported by Stoiljkovic & Agarwal, the heat released during the cutting process melts the fibers together due to the heat generated therefore it needs to be carried out in liquid nitrogen [23]. The distance between two successive blades determines the length of fibers. Prior to many of the methods discussed in the following sections require mechanical cutting of a certain length to proceed with further scission process in order to achieve short fibers on the submicron scale. According to Zhang et al., another method of mechanical cutting has been reported by mechanically stirring the fibers in a solvent and allowing it to swell prior to cutting without affecting the morphology of the fiber [24]. This process was claimed to produce fibers ranging in length from 50 to 700 meters. It is noted that short fiber production using this method might limit to certain fibers and solvent only. The combination of this two is crucial to ensure no chemical reaction took place that can change the chemical properties of the fibers.

2.2 UV Cutting

According to Stoiljkovic & Agarwal, fibers of specific lengths can are obtained by UV masks with slits which rely on the photocross-linking reactions by UV light in the presence of photoinitiator and cross-linkers. The process produces fibers with lengths ranging from 100 to 150 nm depending on the mask used. These masks have different width of slits which result in different lengths of fibers. UV light that passes through the mask illuminates the fibers causing the irradiated parts of the fibers to cross-link. It then becomes insoluble and the remaining fibers which are non-cross-linked are removed by dissolving it in a solvent for a certain amount of time at room temperature. This method is provides a way to fabricate nanofiber mats on a nanoscale [25]. Choi et al. showed that UV treatment on the electrospun fibers have caused the fibers to retain its circular shape compared to the fibers with lower UV irradiation[25]. It is also reported that the UV irradiated fibers did not dissolve in the solvent proving the method to be effective. Figure 1 shows the schematic diagram of electrospun fibers undergoing UV cutting. It is noted that the suitable fiber-solvent systems are needed to ensure no chemical changes and deterioration of the fibers would result from this process.

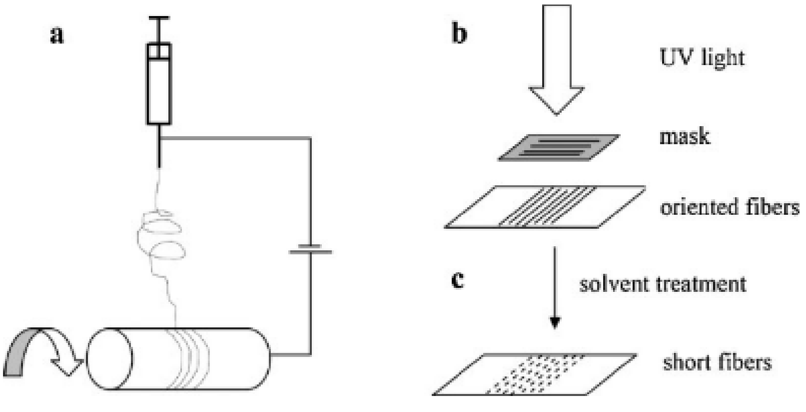

Fig. 1 - UVcutting of electrospun fibers adapted from Choi et al[23] (a) electrospinning of oriented fibers; (b) Illumination of the fibers with UV light via a mask, and; (c) solvent treatment of lighted fibers to eliminate uncross-linked fiber segments [23]

2.3 Precipitation

The fabrication of short fibers can also be produced by solution-precipitation technique where the polymer solution concentration affects the length of nanofibers. In this study, it is reported that lower concentration of polymer concentration which consisted of the poly(ethylene-co-acrylic acid)(PEAA) reduces the average length of PEAA nanofibers produced just as increasing the shear rate in the solution [26]. In example, decreasing the polymer concentration from 6% to 3% results in a 1/4 reduction in the average length of PEAA nanofibers of 4.5μm and an average diameter of 113 nm, since less material can be generated. It is also reported in this paper that higher stirring speed from 4000rpm to 6400rpm reduces the average fiber length from 5.91µm to 3.58µm for samples BSM12 and BSM13 respectively. Exemplification of the nanofiber production method based on solution dispersion is depicted in Figure 2. The polymeric solution (purple) is emulsified in minute droplets when it is introduced fast into the precipitating medium under shear stress (blue arrow). The polymer begins to gel as the inner solvent of aqueous ammonia is drained from the droplets by the outer solvent, while the droplets of increasing viscosity are distorted under shear. Due to the high-speed shear, solid nanofibers can be further broken apart after they have been produced.

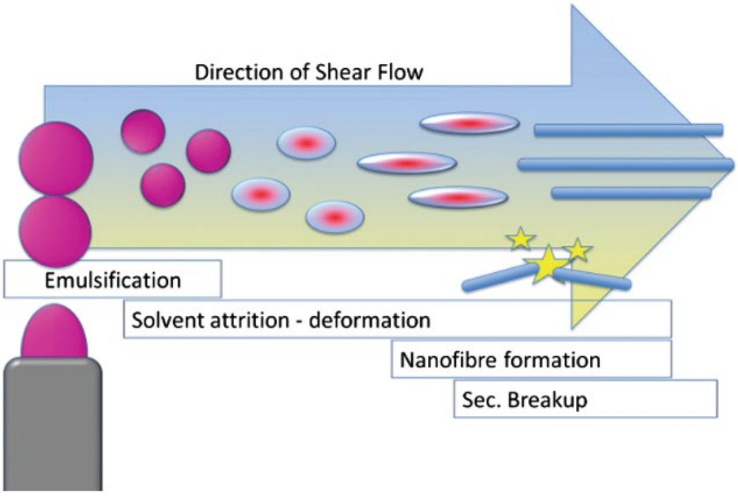

Fig. 2 - Schematic diagram of solution-precipitation mechanism [26]

2.4 Microtome Cutting

The study of microtome cutting dates back to the 1940's which mostly focused on an inexpensive way of obtaining tissue sections such as tissue specimen from the organs of small animals by microtome cutting under dry ice [27]. The apparatus was driven by a crank shaft which had to be manually operated [28] which then turned into a fully automated machine by the early 1960s [29]. In the 1970s modifications to the microtome instrument have also allowed the microtome cutting method to cut plastic embedded blocks down to 1µm [30].

Microtome cutting is employed in an experiment to reduce fiber length prior to further scission using ultrasonication to render them aqueous suspendible. According to research by Mark et al., microtome cutting was applied to the electrospun nylon 6 (poly(caprolactam) or 'polyamide 60 nanofibers and results have proven it to be successful in creating shorter fibers with length of 4.1µm as average. The dispersion of the nylon fibers was unsuccessful when employing ultrasonication alone prior to this procedure (microtome cutting). A cryostat microtome was used to cut electrospun polymer fiber constructions into shorter, aqueous suspendible nylon 6 nanofibers with a constant width of around 80 nm and lengths ranging from 1µm to 12µm. For a more uniform nanofiber dispersion, a step of split-flow thin-cell fractionation is suggested. Figure 3 shows a microtome cut.

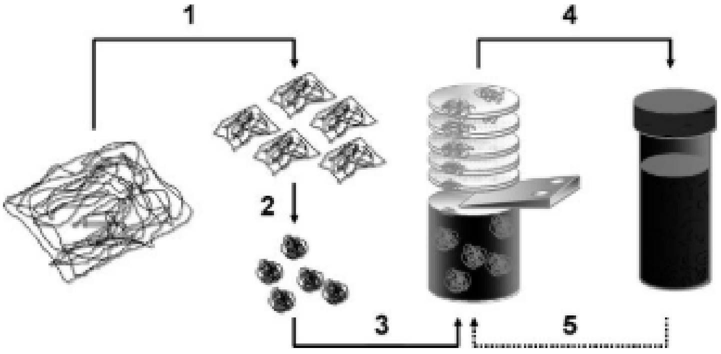

Fig. 3 - Schematic diagram of microtome cutting [31]

2.5 Cryo-Microcutting

According to a study by Liao et al., short individual poly(acrylonitrile)(PAN) microfibers of well-controlled length can be produced through cryo-microcutting prior to ultrasonication treatment. The short PAN microfibers were obtained by freezing the fibers in liquid nitrogen prior to cutting into lengths of 0.4mm or 1.0mm followed by cutting of the fibers with a homemade cutting tool as shown in Figure 4. This produces a narrow fiber length distribution compared to mechanical cutting by using a mixer. As the length of the short fibers depend on their alignment, short fibers of two different lengths 416 ± 83µm and 1034 ± 156 µm (with coefficient of variations of 15% and 21% respectively) was produced from heat stretched fibrillar yarns whereas fibers that were scissioned mechanically using a mixer had ununiform length distribution averaging with 145 ± 129 µm but with 89% coefficient of variation. It can be concluded that cryo-microcutting multifibrillar electrospun fibrillar yarns produces short, controllable fibers [32]. Figure 4 shows the illustration of the homemade cutter for cryo-microcutting.

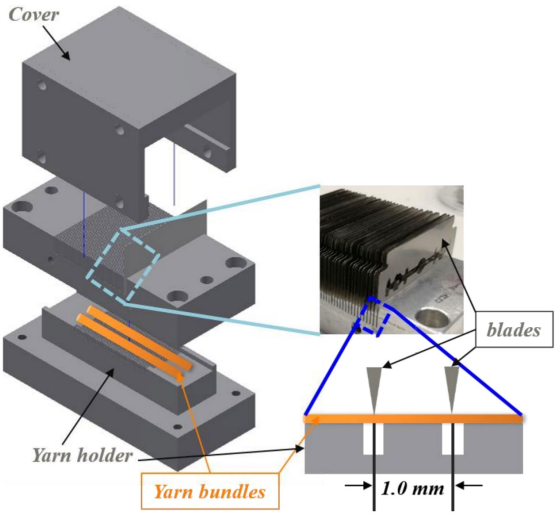

Fig. 4 - Schematic illustration of the home-made cutter with optional interval length of two blades [32]

2.6 Cryogenic Milling

In cryogenic milling, the fabric is cooled during the milling process with liquid nitrogen. Verreck et al. produced short fibers of 27 nm from the electrospun nonwoven fabric by cryogenic milling for 10 minutes. Results of the research indicates that the milling process contributed to the increase in endothermic events which could also cause recrystallization of the drug in a small amount. Similarly, in an effort to produce isolated microfibrils, Chakraborty et al. carried out high-impact grinding on refined fibers under cryogenic conditions with purpose of providing enough energy to break the microfibrils apart from the fiber walls and generate individual fibrils with very small diameters. This yields an excess of 75% of microfibrils being < -1µm.

2.7 Ball Milling

Ball milling is a method that generates high pressure locally by colliding small, hard balls in a hidden container. Multiple research has reported improved results in fibers that were milled with the addition of metal powders as it increases surface area and obtains short fibers in a shorter amount of time [34]–[36]. In some cases, ball milling shows a promising method to obtain short fibers of even up to 200 - 300nm. However it still puts the fibers at risk of forming dense agglomerates due to crushing by the balls especially in wet conditions [37]. Investigations by Khengkhetkit and Amornsakchai reveals that ball milling of dried pineapple leaf fibers (PALF) provided substantial results of short elementary fibers but shown to be the least effective, mechanically speaking, as reinforcement for the PALF-PP composite due to the formation of agglomerates. Aside from that, BM-PALF would have been a better reinforcement material if it were of appropriate length. Similar to this technique, ball milling or mechanical grinding, is only applicable to fibers composed of brittle materials. Grinding can break down the fiber network in ductile polymeric fibers, but it also affects the fiber shape owing to fiber stretching and the heat created during grinding.

In an effort to use high impact force to produce carbon nanoparticles, Li et al. [12] proves ball milling to be successful; as the steel balls continually collides, it provides sufficient energy to break the shell structure leading to the production of nanoparticles. Addition of metal powder which acts as additional microscopic milling balls increases the formation rate of carbon nanoparticles. The same observation can be seen in research by [38]. However, it is noted that using ball milling alone for electrospun fiber would not be possible due to the nature of the electrospun fiber that is soft like wool. It will cause the fiber to lose its fibrous structure due to the crushing impact from the metal balls.

2.8 Razor Blade Cutting Under Liquid Nitrogen Cooling

Razor blade cutting under liquid nitrogen cooling is a method used to pre-cut short fibers before further scission and dispersion by ultrasonication which will then be filtered to obtain the short microfibers (Matsuura et al., 2018). Besides, this method of scission is also used to produce rods of fibers with an average length of 50 to 100µm and which do not agglomerate for the purpose of composite reinforcement [40]. Based on the observation of SEM images by McCall et al., compared to fibers cut using scissors, razor blade-cut portions are rather undistorted; however it is more splayed out which may result in less precise measurements.

2.9 Ultrasonication

Among the most common scission methods of electrospun nanofiber is by means of ultrasonication. It effectively disperses particles through cavitation. Depending on the speed of ultrasonication, fibroin nanofibers can be tuned in differences of up to 75nm, specifically within the range of 30 to 120 nm. In water, the natural fibers disintegrate into individual nanofibers due to ultrasonication. The ultrasonic shock waves that induce erosion of the surface of the fibers that split along the axial direction progressively degrade the micron-sized natural fibers into individual nanofibers. The speed of disintegration is determined by the ultrasonic wave's strength and frequency. In 2011, Lee et al. showed that ultrasonication increases pore size and thickness of the three-dimensional nanofibrous scaffold. Similarly, in 2017, Ya et al. have also concluded that there is an increase of mixture viscosity as well as increase in the generation of voids due to ultrasonication treatment. Lee et al. ultrasonicated the nanofibers for 5 minutes and it was found that the fibers became unstable and decreased in their mechanical properties as well as no more increase in porosity. Ultrasonicated for 0, 15, 30, 45, and 60 minutes, Zhang et al. reported the results of the dispersion of PLLA/ZnO to be successful with a distribution of short fibers within the range of 100–1000 nm while having optimum characteristics when sonicated for the duration of 45 minutes [42]. The minimum fiber diameter obtained from this experiment was found to be 271.65±170.35 nm. In a research by Niemczyk-Soczynska et al., PLLA fibers ultrasonicated in different sonication media; water, ethanol and isopropanol were reported to be least effective with water as the sonication media whereby fibers with average length of 237 ± 6.7 µm is produced whereas isopropanol was more effective by having an average fiber length of 50 ± 4.9 µm followed by ethanol with the average length of 91 ± 6.9 µm [43].

3. Conclusion

Short fibers prove to be a very useful due to their unique characteristics of having large surface areas, high porosity, good continuity and many other unique properties. These fibers have been widely applied in bio-related applications such as for drug delivery, tissue engineering scaffolds, as well as wound dressing. The methods used to create these short fibers which are discussed in this paper are ultrasonication, mechanical cutting, UV cutting, precipitation method, microtome cutting, cryo-microcutting, cryogenic milling, ball milling, and razor blade cutting under liquid nitrogen. Each method has its advantages and disadvantages and depends very much on the polymer types which includes its mechanical properties and interaction with the solvent used in a specific method.

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