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Review Article

Producing Cellulose nanofiber from Cotton wastes by electrospinning method

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ABSTRACT

One of the main issues of nanotechnology is producing materials with new properties. Nanotechnology, as a powerful tool, has the ability to create evolution in the agricultural system and food–medicinal industries across the world. Producing a high-performance material from reclaimed cellulose material will increase motivation to recycle these materials at all phases of paper production and remove them from the waste stream. Electrospinning typically produces nonwoven mats of nanofibers, which could provide nanoscale pores for industrial filters. Electrospinning (ES) technique is a method to produce nanofibers by applying an electric field on a fluid jet. Nanotechnology also play a role in recycling of agricultural crops residues and converting them into energy and industrial chemicals by using natural processes of biological, physical and chemical. For example, unfortunately, since cotton harvesting time up to producing textile more than 25% of the fibers are converted to wastes. By using an electro spinning method of cotton waste some products such as cotton balls, yarns and cotton batting are produced. In addition, you can use this method to produce nanocellulose fibers that constitute 90 percent of cotton yarn and also produce fibers less than 100 nanometers which are 1000 times smaller than the current produced fibers. The technique relies on electrical rather than mechanical forces to form nanofibers. Different applications of nanofibers can be used in fields such as nanomembrane filters, protective clothing, electronical and optical equipment, and biomedical applications reinforced composites.

Keywords: *Cellulose nanofiber; Cotton; Electrospinning; Fibers; Nanotechnology; Papermaking.*

INTRODUCTION

In recent years, nanotechnology has become a topic of great interest to scientists and engineers, and is now established as a prioritized research area in many countries [1]. Electrospinning is a straightforward method to prepare fibers with diameters as small as several tens of nanometers [2].

Although the term ‘‘electrospinning’’, derived from ‘‘electrostatic spinning’’, was used relatively recently, but its fundamental idea dates back more than 60 years earlier. From 1934 to 1944, Formulas published a series of patents [3,4], describing an experimental setup for the production of polymer filaments using an electrostatic force. Usually, solvent mixtures are used to control solvent evaporation during the electrospinning process, and thus avoid clogging of the spinneret by early solidification of the polymer. A polymer solution, such as cellulose acetate, was introduced into the electric field [5]. Materials in the form of nano- and micro-fibers present unique properties when compared to other morphologies such as Electrospun materials which have a larger specific surface area and small pore size compared to commercial non-woven fabrics. They are of interest in a wide variety of applications including semi-permeable membranes, tissue engineering scaffolds and reinforced paper composites [4,6-9]. When the diameters of polymer fiber materials are Shrunk from micrometers (e.g. 10–100 μm) to submicrons or nanometers (e.g. 10×10-3–100×10-3μm), there appear several amazing characteristics such as very large surface area to volume ratio, flexibility in surface functionalities, and superior mechanical performance (e.g. stiffness and tensile strength) compared with any other known form of the material. Cellulose, the most abundant natural and renewable polymer, which makes it a cheap raw material for various applications [3], is used traditionally in the papermaking industry. The use of cellulose and its derivatives to obtain nano-fibers by the electrospinning process presents a great opportunity for better utilization of cellulose and development of new applications. However, processing via electrospinning of biopolymers such as cellulose usually present challenges. This is due to their limited solubility in typical solvents and their tendency to aggregate or form gels.

Few solvents are capable of dissolving cellulose so that it can easily be processed by electrospinning [4]. These outstanding properties make the polymer nanofibers to be optimal candidates for many important applications [5].

Recently, electrospun nanofibers based on cellulose and its derivatives have been studied as potential candidates for applications within the field of cellulose nanofibers. This interest in cellulose-based materials is primarily driven by its environmental value as a biomaterial [10-13]. However, little research has been done on the use of cellulose and cellulose derivatives as a raw material within electrospinning. By using an electrospinning method of cotton waste some products such as cotton balls, yarns and cotton batting are produced. The complications involved in electrospinning of cellulose are mainly due to the many difficulties ascribed to the material, one being its reluctance to interact with conventional solvents. Therefore, the choice of solvent systems is very important.

A number of processing techniques such as drawing [14], electrospinning [15,16], etc have been used to prepare polymer nanofibers in recent years. The drawing is a process similar to dry spinning in fiber industry, which can make one-by-one very long single nanofibers. The electrospinning process that is a versatile technique in terms of its simplicity and relative low-cost, seems to be the only method which can be further developed for mass production of one-by-one continuous nanofibers from various polymers. The diameter and morphology of fibers obtained by electrospinning depend on the process conditions and variables [5].

The evidences clearly demonstrated that the electrospinning has attracted increasing attentions recently. Since 1980s and especially in recent years, the electrospinning process has regained more attention probably due in part to a surging interest in nanotechnology, as ultrafine fibers or fibrous structures of various polymers with diameters down to submicrons or nanometers can be easily fabricated with this process [17]. Up to date, it is generally believed that nearly one hundred different polymers, mostly dissolved in solvents yet some heated into melts have been successfully spun into ultrafine fibers using this technique. Strangely enough, although the electrospinning process has shown potential promising and has existed in the literature for quite several decades, its understanding is still very limited [5].

In this paper, a comprehensive review is made on the researches and developments about producing cellulose nanofibers from cotton wastes including processing, structure and property characterization, applications, and simulations.

Cellulose

Cellulose is a linear homopolymer composed of β-D-glucopyranose units which are linked together by (1→4)-glycosidic bonds the length of a native cellulose molecule which depends on the source of cellulose is at least 5000 nm [18]. For lignocellulosics, a degree of polymerization up to 10,000 was found. Three hydroxyl groups, placed at the positions C2 and C3 and C6 can form intra- and intermolecular hydrogen bonds. These hydrogen bonds allow the creation of highly ordered, three-dimensional crystal structures [19]. The microfibrils have both crystalline and amorphous regions. Both linear cellulose molecules and the supermolecular microfibrils have a dominant influence on the behavior of wood as a material. The molecular aggregations of cellulose in the wood cell wall contribute to its unique polymer properties.

Cellulose fibers on the nanoscale are prepared in four different ways: (1) bacterial cellulose nanofibers, (2) cellulose nanofibers by electrospinning, (3) microfibrillated cellulose plant cell fibers and (4) nanorods or cellulose whiskers. Processing techniques have a significant impact on the adhesion properties of the resulting cellulose nanofibers in composite material applications [18].

- ***Cellulose Nanofiber***

Nanomaterial technologies have attracted wide attention because of their super functionalities due to their extremely large and active surface areas. Nanofibers are included in an analogous category, and are also expected to have unique properties. Cellulose fibrils play a significant role in the contribution to the high strength of plant bodies through hierarchal structures consisting of plant cell walls [20].

The study of cellulosic nanofibers as a reinforcing recycled phase in nanocomposites started two decades ago. Since then a huge amount of literature has been devoted to cellulose nanofibers, and it is becoming an increasingly topical subject [19]. The initial studies indicated that nanocelluloses having inherent properties like low toxicity, biocompatibility and biodegradability together with excellent mechanical properties are excellent candidates to develop cellulose based nanocomposites for various applications. These composites are being evaluated using in vitro studies for biocompatibility and in vivo studies for biomechanical performance in order to develop these materials to a commercial level [21]. Figure 1 shows the molecular structure of cellulose as a carbohydrate polymer presented in nanofiber products.

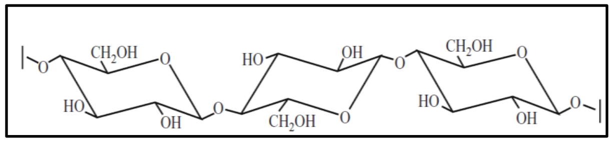

Fig. 1. Molecular structure of cellulose

- ***Cellulose Nanocomposites***

Nanocomposites are produced from cellulose and generally are two-phase materials in which one of the phases has at least one dimension in the nanometer range (1 - 100 nm) [22]. Depending on the geometrical shape of the nano-reinforcement, nanocomposites can be divided into three groups: one dimensional nanocomposites, when the reinforcements are sheets; two dimensional nanocomposites, when reinforcements are tubes or whiskers, and three dimensional nanocomposites where spherical particles are used as reinforcement [23].

The advantages of nanocomposite materials when compared with conventional composites are their superior thermal, mechanical and barrier properties at low reinforcement levels, as well as their better recyclability and low weight [24]. Biodegradable polymers, in particular, may require improvement in terms of brittleness, low thermal stability and poor barrier properties [22].

- ***Applications of Polymer Nanofibers***

Nanofibers produced by electrospinning are industrially and scientifically interesting due to their unique and interesting features such as small diameters and a high surface area per unit volume. Moreover, the diversity of materials suitable to transform by electrospinning and the simplicity of this process make electrospun nanofibers attractive for a number of applications. There are different application fields where the use of electrospun polymer nanofibers has gone growing in the recent years [25]. Most of the applications of polymer nanofibers have been just developed to laboratory level, however, they have a huge potential to be attractive for the industry [5].

So far, most achieved nanofibers are in non-woven form, which can be used for applications such as filtration, tissue scaffolds, medical prostheses, implant coating film, wound dressing, drug delivery, cosmetic, protective clothing, and electrical and optical applications. In the case of textile industry, continuous single nanofibers are necessary. The use of nanofibers as reinforcement is also interesting since the interaction between the reinforcement and the matrix is better than in conventional fibers.

In addition, using techniques such as sputtering, evaporation in vacuum or chemical deposition, it is possible to coat polymer nanofibers with metals, carbon, and molecular solids; thereby, their application fields can become greater [25].

Reasons for use of nonwood plant fibers in paper production

Although some of the nonwood fibers used for papermaking are used because of their fine paper making qualities, majority of nonwood fibers is used to overcome the shortage of wood fibers. As a result their use is more widespread in countries with shortage of wood. Although till recently, use of nonwood fibers for pulp and papermaking was concentrated in countries with limited wood supply, it is now showing an increasing trend even in countries with adequate wood supply due to environmental considerations. This trend can be expected to grow further and it can be safely said that the future of nonwood plant fibers as pulping and papermaking raw material looks bright [26].

- ***Shortage of wood fibers around the world***

Asia is presently the largest fiber deficit region, followed by Western Europe. At the same time, Asia is the focus of fiber demand growth for pulp and paper, housing and wood for fuel. If this assessment is accurate, pulp and paper industry's dependence on virgin fibers must be reduced by expansion in the use of recovered paper and growth in the use of nonwood plant fibers in Asia [27].

In India, forest based resources are being rapidly depleted while the demand for paper is increasing. In order to resolve this situation, for instance, the government of India has extended concessions and relief to stimulate use of agriculture-based cellulose raw materials. As a result, use of forest based raw materials has decreased from 84% in 1970 to 43% in 1994 and use of agriculture-residues has increased from 9% to 32% the rest being made up of recovered fibers. Use of nonwood plant fibers for pulp production in China is not a matter of choice but a matter of necessity. China is not rich in wood resources. Its forests are limited in size and area available for future plantation. Even with expansion of the national forest, nonwood plant fibers will constitute a major source of fiber for pulp [28].

- ***Surplus of nonwood plant fibers***

Cotton has a long historical role in socio economic development in the world and also is one of the oldest known fibers[29]. The abundance of nonwood fibers in some countries is a factor for its use in papermaking. Sometimes, using nonwood fibers in papermaking is considered the best way to dispose them. In Europe and Americas, the use of agricultural residues in pulping has a further advantage because it averts the need for disposal, which currently increases farming costs and environmental deterioration through pollution, fires, and pests [30]. Figure 2 shows two different types of cotton which is used as a raw material in papermaking.

Fig. 2. Cotton plant as a abundant plant suitable for papermaking

Electrospinning method

Electrospinning is a novel process which has been recognized as one of the most efficient techniques for the production of polymer nanofibers. It started to be used into nanotechnology and materials science since 1980s and has attracted increasing attention recently. In addition, this seems to be the only process that can be developed for mass production of one-by-one continuous nanofibers from a variety of polymers [5]. The number of scientific publications about this topic has increased considerably. Theoretically, this is a fast and simple process driven by the electrical forces on the surface of polymeric fluids, forming fibers with submicron scale diameters through the action of electrostatic forces [31]. Interesting new properties are achieved when the diameter of polymer fiber is reduced from micrometer to nanometer range [32].

In the last years, the number of reports of the electrospinning of cellulose nanofibers has been growing due to the excellent features that can be obtained. In addition, the study of electrospun cellulose nanofibers with different compositions and arrangements could significantly expand its applications and usages [33,34]. One example is composites materials where either non-woven or randomly arranged nanofiber mats; do not improve the mechanical properties of the polymer. Hence it is necessary to employ fibers arrangements in pre-determined directions in order to achieve the superior structural properties [5]. Figure 3 shows a schematic of electrospinning method with its components.

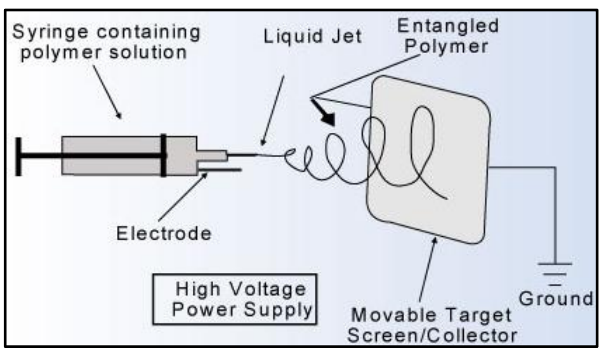

Fig. 3. Electrospinning method

- ***Polymers Materials for Electrospinning***

Polymers such as polyolefins, polyamides, polyesters, polyurethanes, polypeptides, and polysaccharides, have been successfully electrospun into micro and nanofibers mats [35].

- ***Electrospun Nanofibers from Cotton Waste Fibers***

Cotton is a soft, fluffy staple fiber that grows in a boll, or protective capsule and is the most popular natural fibers used textile fiber in the world today. As described before, It is harvested from the cotton plant composed of 88-96% cellulose; the remainder is waxes, protein, and pectin materials [36]. This fiber is spun into yarn and used to make soft and breathable textile. Several step processes are required for converting cotton plants to cotton fabric such as opening, blending, carding, etc.

During step processes, over 25% cotton fibers are lost as waste [26]. Typically, cotton waste is used for low value products such as cotton balls and cotton batting. In this method, cotton wastes are converted into higher-value products by using electrospinning technique [30].

- ***Cellulose and Cellulose Derivative Electrospinning***

Electrospinning of cellulose can be facilitated by using cellulose esters solutions followed by regeneration [37]. For example, cellulose acetate can be easily dissolved and processed in non-polar solvents, suitable for electrospinning such as acetone, dichloromethane, chloroform and methyl acetate [4]. Cellulose acetate is a semisynthetic polymer obtained from esterification of highly pure cellulose with acetic anhydride using sulfuric acid as a catalyst. Its properties depend on the esterification degree (degree of substitution) defined by the number of OH groups substituted by acetate groups which in turn defines the obtained material as acetate, diacetate or triacetate [38].

Traditional raw materials for the manufacture of Cellulose Acetate include renewable, biodegradable, and inexpensive wood pulp but alternative sources have also been considered, including residues from sugarcane bagasse, bacterial cellulose, newspapers and fruit seeds [4].

A number of reports have shown that regenerated cellulose nano-fibers can be obtained by electrospinning cellulose acetate. Here the cellulose acetate component is converted into regenerated cellulose by stripping the acetyl groups by alkaline hydrolysis [26]. One of the most relevant properties of fibers of regenerated cellulose is the chemical resistance to almost all organic solvents and aqueous solutions in a broad pH range (from 3 to 12). Additionally, regenerated cellulose membranes have shown to be more permeable to water than conventional microporous membranes [15,37]. This property makes it attractive for applications such as affinity membranes and membranes with anti-microbial properties.

Cellulose nanocrystals have significant potential as reinforcement of polymers which require very low filler content. Cellulose nanocrystals offer several advantages including their high aspect ratio and surface area, excellent chemical, mechanical and thermal properties. Cellulose nanocrystals have a significantly high elastic modulus (120–150 GPa) and they are interesting due to their application as mechanical reinforcement in thin and ultrathin polymer films and webs made from nano- and micro-fibers[39,33].

The influence of the rheological properties of cellulose acetate solutions in the electrospinning process was also investigated.

This technique relies on electrical rather than mechanical forces to form fibers. The fiber produced is less than 100 nanometers in diameter.

Whenever cotton is converted to fabric and garments, fiber (cellulose) is lost to scrap or waste. At present it is largely discarded or used for low-value products, such as cotton balls, yarns and cotton batting.

Producing a high-performance material from reclaimed cellulose material will increase motivation to recycle these materials at all phases of paper production and remove them from the waste stream. In Figure 4, electrospun nanofibers of cotton are shown. As we see, this method typically produces nonwoven mats of nanofibers, which could provide nanoscale pores for industrial filters and etc.[38].

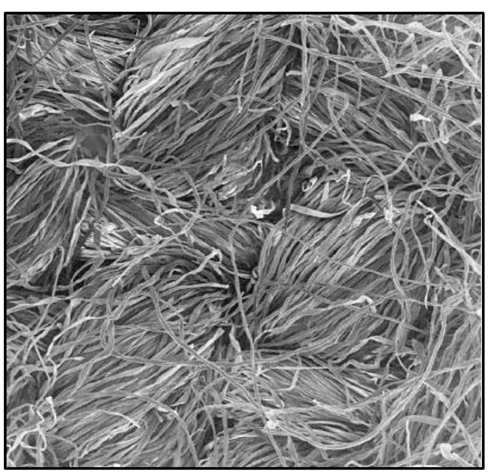

Fig.4 . Close-up of nanofibers from electrospun cotton

Mechanical Characterization: Tensile Testing

So far, no specific methods have been standardized to analyze mechanical properties of a single nanofiber produced by electrospinning due to its very small dimension. For this reason, nanofibers nonwoven membranes are used to perform mechanical characterization through conventional testing techniques. However, in the last years some interesting researches have been conducted in relation to the mechanical characterization of single nanofibers. The strength, strain and Young modulus of samples in this method can be determined through a tensile testing which called Dynamic Mechanical Thermal Analyzer. Preliminary experiments were performed in order to get the most suitable sample for the tensile testing. The first method used to get electrospun samples consisted in the preparation of fibers mats with a thicker enough to cut. Thereby, samples were at different times such 30, 60 and 120 min on the collector [5]. Finally, these bundles of nanofibers can be used as samples for the tensile test and at least three bundles can be tested for each material to ensure accuracy of data. However, it was not easy to obtain suitable samples for the tensile testing since the fibers manipulation was complicated. Different types of problems such as no homogeneous tension over the fibers, some crossed over fibers and bundles that were not alignment on the frame and etc, were presented during the sample preparation [26].

CONCLUSIONS

Producing a high-performance material from reclaimed cellulose material will increase motivation to recycle these materials at all phases of paper production and remove them from the waste stream. Electrospinning typically produces nonwoven mats of nanofibers, which could provide nanoscale pores for industrial filters.The cross section areas of the samples in form of bundles which are visualized under SEM have an overview of the morphology of electrospun cellulose nanofibers. Regarding the diameters of the electrospun fibers, it can be observed in the photos that there was a size distribution; diameters fibers that ranged from few nanometers until several micrometers were encountered as will be shown by AFM results later.

I. Nanocomposites in the form of aligned fibers can be successfully prepared by electrospinning of cellulose acetate reinforced with different cellulose nanowhiskers concentrations.

II. The diameters of the produced fibers are between 300 and 3000 nm. This difference may have been because of non-homogenous cross sections as well as the aspect mentioned about the measurements by AFM.

III. Cotton waste fibers can be successfully electrospun into nanofibers using trifluoroacetic acid as the solvent. This process occurs at room temperature and is not required concentrated salts and post-spun treatment. In the case of applied voltage, the diameter of nanofibers tends to decrease with increasing electrospinning voltage. The cellulose electrospun nanofibers were decrystallize to amorphous cellulose and the functional groups of electrospun nanofibers are the similar to cotton fibers.

IV. The interest in electrospinning as a novel cellulose production technology has increased over the past 5 years. Through the electrospinning process it is possible to produce fibers from 50 to 500 nm in diameter, with high specific surface area and small pore size. Also, although efforts have been made to characterize the surface chemistry of films or membranes prepared with electrospun fibers, the surface chemistry of the individual electrospun fibers has not been clearly determined.

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