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Photochromic Nanofibers

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http://dx.doi.org/10.5772/intechopen.74663

Abstract

Photochromic compounds exhibit a reversible color change via UV irradiation. The use of photochromic nanofibers in the field of functional materials such as optical sensors, processing media, optical data storage devices, and functional components for smart surfaces can be attractive. This review chapter gives an overview of the production of photochromic nanofibers by electrospinning.

Keywords: nanofiber, photochromism, photochromic textile, electrospun, UV-responsive nanofibers

1. Introduction

Nanotechnology applications and nanofiber production have become increasingly important in terms of functional textile material production. Nanofiber surfaces have potential to be used in a wide range of applications, such as filtration, sensor, composite materials, medical textiles, etc., because of their wide surface area, high porosity, and possibilities of using different polymer types [1–3].

Nanofiber production can be achieved by different methods such as electrospinning, drawing, self-assembly, phase separation, and template synthesis. While the applications of all these methods are generally laboratory scale, the electrospinning process can be scaled, and it has a potential for industry processing [4]. Electrospinning method has also some advantages such as ease of application, low cost, and possibility of using many different polymers. Because of all these properties, electrospinning is perhaps the most preferred method for the nanofiber production [1, 3–7].



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Nanofiber surfaces may gain functional properties with the addition of different substances such as drugs, cosmetics, dyes, etc. in the polymer matrix, thereby increasing the industrial application possibilities [1, 5, 7–11]. A photochromic compound, which can change its color upon irradiation with ultraviolet (UV), is also one of the materials used in the nanofiber surface functionalization. This chapter describes the functionalization of nanofiber surfaces produced by electrospinning method with photochromic compounds.

2. Electrospinning

In the electrospinning process, the polymer solution or polymer melt is subjected to a high potential tension, and the polymers are electrically charged. The polymer solution jet, which comes from a fine nozzle, travels toward the target, which is loaded with the reverse polarity and positioned opposite the nozzle. During this flow, the solvent evaporates (or begins to solidify if electrospinning from the melt is applied), and the polymer jet is scattered as very fine fibers with a diameter at the nano-level [1–4]. The set-up of the electrospinning method is presented in **Figure 1**.

The polymers which are soluble in a solvent and have an enough high molecular weight to be electrospun can be used in nanofiber production by electrospinning. Many different natural polymers, synthetic polymers, and the polymer blends have been successfully electrospun into nanofibers by electrospinning method [12–21].

The main features that distinguish this method from other conventional methods are that the diameters of the fibers produced are below the micrometer range and it is possible to work with polymer solution or polymer melt [7, 22]. The application areas of the fibers produced by electrospinning method are quite extensive, and the material properties vary according to the application area.



Figure 1. The set-up of electrospinning method.

There are various parameters which affect the electrospinning process. These parameters can be divided into three groups as solution parameters, process parameters, and ambient parameters (**Table 1**) [1–3].

Nanofibers obtained by electrospinning method can be functionalized by loading some compounds such as drugs, dyes, metals, etc. to the polymer matrix [1, 5, 7–11]. Photochromic nanofiber, one of these types of functional nanofibers, is obtained by incorporating photochromic compounds into the polymer solution. Thus the nanofiber surface exhibits photochromic property, thereby increasing the different application possibilities.

Solution parameters	Process parameters	Ambient parameters
Concentration	Distance from nozzle to collector	Temperature
Molecular weight of the polymer	Flow rate	Humidity
Viscosity	Voltage	Atmospheric pressure
Electrical conductivity	Collector type	
Surface tension		

Table 1. The parameters affecting electrospinning process [1–3].

3. Photochromism

Photochromism is described as "the reversible absorption spectrum changes of a compound when the sample is irradiated with ultraviolet (UV) light" [23, 24]. Photochromic materials exhibit a reversible color change upon the change of UV light intensity. These compounds are generally colorless in the absence of UV light, while the compounds are colored with the increase in UV light intensity in the environment, and then the compound can return to its colorless state due to the decrease of UV light intensity. This photochromic behavior is stated as "positive photochromism." However, negative photochromism occurs if the compounds change their colored form into the colorless structure with UV light irradiation [25]. Photochromic materials may also be categorized into two groups as P-type and T-type according to the back reactions which occur with light (other wavelengths than UV) or heat in P-type and T-type photochromism, respectively [23, 26].

Photochromic compounds are widely used not only in optics but also in areas such as plastics, cosmetics, inks, and textiles [27]. The use of these compounds in textile field is problematic due to their poor affinity to the material, low solubility in water, sensitivity to high temperature, etc. [24, 28, 29]. Many different methods such as encapsulation, sol-gel processing, etc. have been developed to improve the application of photochromic compounds [24, 30–32]. Electrospinning can be seen as an alternative method for the use of photochromic compounds in textile field.

Application of photochromic compounds by electrospinning process provides advantages such as increasing sensitivity of photochromic compounds or reducing the time necessary for photochromic compound to respond to the UV irradiation due to large surface area of the electrospun mats [33]. Photochromic electrospun nanofibers could find applications in areas



Table 2. Photochromic compounds and their photochromic reactions [23, 25, 26].

such as optical sensors, processing media, optical data storage devices, and functional components for smart surfaces [34, 35].

There are many different types of photochromic compounds as inorganic and organic. In the production of electrospun photochromic nanofiber, the organic photochromic compounds, such as spiropyran, spirooxazine, naphthopyrans, diarylethenes, and fulgides, which change their color based on pericyclic electrocyclic reactions, and azobenzene and stilbene, which change its color based on cis-trans isomerization, have been widely used (**Table 2**). Apart from such organic photochromic compounds, various different photochromic systems such as metal oxides have been also used in the production of electrospun photochromic nanofibers.

4. Photochromic nanofiber production

The production of electrospun photochromic nanofiber has been studied by a large number of authors. These studies which have used many different types of polymer and photochromic compound are discussed in depth below.

In the study of Akçakoca Kumbasar et al. [36], spirooxazine-based photochromic thermoplastic polyurethane (TPU) nanofiber mat, which is shown in **Figure 2**, has been obtained by electrospinning method. The photochromic coloration of the sample has occurred after UV irradiation (**Figure 2**). As shown in **Figure 3**, the neat nanofibers have been bead-free, and the inclusion of photochromic compounds in TPU polymer solution has not affected the smooth morphology of the resulting electrospun nanofibers (**Figure 3**).

Genovese et al. [34] have investigated the optochemosensing properties of the spiropyran (SP)-doped-poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-HFP) electrospun fibers. They have irradiated the SP-doped PVDF-HFP films, nanofibers, and fibers with UV light and then exposed to acidic vapors to determine both the photochromic and acidochromic properties of the samples. They have stated that the samples changed their color from colorless spiropyran form to red-colored merocyanine form with UV irradiation, and then, after the exposure to acidic vapors, the samples changed their red-colored structure to yellow protonated merocyanine form. In addition, the nanofibers have exhibited faster photochromism and protonation with respect to the fibers and films due to higher surface area of the nanofibers.

Durasevic [37] has reported the preparation of electrospun photochromic polyurethane (PU) fibers by incorporating the spirooxazine-based photochromic compound into the polymer solution. The samples have changed their colorless structure to deep blue color form with UV irradiation. However, this coloration has varied according to the UV light source type. The samples have exhibited darker color upon UVA irradiation with respect to the UVB irradiation. Durasevic [37] has specified that these electrospun photochromic materials can be used as UV sensor for medical textile application.

Khatri et al. [38] have prepared photochromic polyvinyl alcohol (PVA) nanofibers for the application of recording and erasing quick response (QR) codes. They have used spiropyran



Figure 2. Images of the photochromic TPU nanofiber mat before and after UV irradiation [36].



Figure 3. SEM images of (a) neat TPU nanofibers and (b) 20% photochromic compound-loaded TPU nanofibers [36].

and spirooxazine as photochromic compounds. They have stated that PVA is a useful polymer for the production of photochromic nanofibers due to the presence of OH groups in the polymer which leads to the formation of hydrogen bonding between the polymer and the photochromic compound, causing the uniform dispersion of the compounds within the nanofibers. They have also reported that the PVA-spiropyran nanofibers showed higher photocoloration and photo-reversibility than the PVA-spirooxazine nanofibers.

Liao et al. [35] have synthesized photoswitchable nanoparticles by grafting the fluorescent carbon nanoparticles with copolymers of styrene and spiropyran. Then, they have fabricated the nanofibers by electrospinning of these nanoparticles. The electrospun samples have exhibited reversibly photoswitchable fluorescence between blue-green and red colors.

In the study of Ali et al. [39], electrospun spiropyran-poly(ε -caprolactone) (PCL) fibers have been studied for patterned color image recordings. It has stated that coloration of the

photochromic samples with UV irradiation has been faster than their decoloration. Their results have shown that the average fiber diameter of the neat fibers was larger with respect to spiropyran-PCL fibers. They have also performed the image recording on the electrospun photochromic fiber mat by write-erase-write technique with UV and visible lights.

Zillohu et al. [40] have investigated the structure and erasable writing properties of the electrospun spirooxazine-polyvinylidene fluoride (PVDF) fibers. Addition of spirooxazine compounds into the polymer matrix has made the fibers less polar and more amorphous. The authors have performed the pattern creating on the electrospun photochromic fibers by using water drops as lenses. They have also compared the UV absorption properties of the water drops loaded with silver and gold nanoparticles. The water drop containing silver nanoparticles has exhibited more absorption than the water drop loaded with gold particles.

The stability of electrospun naphthopyran-polyvinylpyrrolidone (PVP) nanofibers has been investigated in the study of Liu et al. [41]. They have used three different types of naphthopyran. The average diameters of naphthopyran-containing PVP fibers have been smaller with respect to the neat PVP fibers. The electrospun naphthopyran-polyvinylpyrrolidone (PVP) nanofibers have changed their colorless structure into the colored structure with UV irradiation after 5 min. However decoloration of the photochromic samples has been very slow. After removal of the samples from UV light, the color of the samples has remained even after 3 days. The authors have indicated that hydrogen bonding between PVP and naphthopyran caused the inhibition of naphthopyran fading.

Lee and Kim [42] have prepared the diarylethene-loaded polystyrene (PS) and polyacrylic acid (PAA) fibers by electrospinning method, and then they have studied on the image recording properties of the electrospun photochromic fibers. The diameter of the diarylethene-doped PAA fibers has been smaller than the diameter of the diarylethene-doped PS fibers, thereby increasing the resolution of patterned color images which were created on the electrospun mat upon photomasked UV irradiation. It has been found that these patterned color images have remained for 30 days.

Zhang et al. [43] have synthesized spiropyran-based PS nanowires and then used electrospinning method to obtain photochromic fibers. They have stated that the electrospun fibers have changed their color from white to pink under UV light.

In the study of Li et al. [33], spiropyran-based photochromic nanofiber mats have been obtained by electrospinning method. They have used poly(methyl methacrylate) (PMMA) and gelatin as the polymers. The hydrophilicity and photochromic properties of the samples have been investigated by the author. The water contact angle of spiropyran-gelatin nanofibers could have not been measured due to the water drop on the samples penetrated into them in 10 s. The water contact angles of spiropyran-PMMA nanofibers have decreased with UV irradiation as a result of the structural change of the photochromic compound from spiro to merocyanine form.

A photochromic spironaphthoxazine/isophorone-based fluorescent dye system has been developed by Lee et al. [44], and then they have used this dye system in the production of photochromic PMMA nanofiber by electrospinning method. Lee et al. [45] have also prepared

the electrospun PMMA nanofiber-loaded photochromic spironaphthoxazine and D- π -A-type fluorescent dye. They have studied on the spectral and erasable writing properties of the photochromic samples at both studies. The authors have indicated that the electrospun photochromic PMMA nanofibers have a potential for optical data storage applications.

The hydrophilicity and photo-coloration properties of the electrospun spiropyran-PMMA and spiropyran-cellulose acetate (CA) nanofibers have been examined by Nammoonnoy et al. [46] and Shuiping et al. [47], respectively. They have reported that the photochromic nanofibers have turned from hydrophobic structure into hydrophilic structure by UV irradiation.

Lee et al. [48] have investigated the photocoloration of the electrospun spiropyran-loaded poly(ethylene oxide) (PEO) and polystyrene (PS) nanofibers. The electrospun spiropyranloaded nanofibers have colored after photomasked UV irradiation, and then the samples have reverted back their original form with heat treatment at 120°C. However, after UV irradiation, the color of spiropyran-doped PEO nanofiber has been purple, while the color of spiropyran-doped PS nanofiber has been blue. The authors have indicated that spiropyrans tend to exhibit purple color in a polar environment and blue color in a nonpolar environment after UV irradiation. Thus, the spiropyran-loaded PEO nanofiber which is more polar than PS nanofiber has exhibited purple color after UV irradiation.

De Sousa et al. [49] have prepared spiropyran-cyclodextrin (SP- β CD) inclusion complexes and then they have electrospun SP- β CD and poly(methacrylic acid) (PMAA) blends into nanofibers. They have also fabricated spiropyran (SP)-PMAA nanofibers to analyze the effect of CD on photocoloration of spiropyran. The coloration rate of SP- β CD-PMAA nanofibers has been faster than the coloration rate of SP-PMAA nanofibers due to the hydroxyl groups of the β CD which lead to a stabilization of the merocyanine structure, in a more efficient way than those hydroxyl groups of PMAA polymer. In this study, water contact angles of the electrospun photochromic nanofibers have also been investigated. Incorporating SP- β CD inclusion complexes in the PMAA polymer matrix has increased the hydrophobicity of PMAA nanofibers. Additionally, the water contact angle of SP- β CD-PMAA nanofibers has decreased about 15° after UV irradiation.

In the study of Wang et al. [50], spiropyran-loaded poly(vinylidene fluoride-co-hexafluoropropylene) (PVDF-co-HFP) nanofiber mats have been fabricated by electrospinning method. They have obtained more uniform fibers upon addition of spiropyran to PVDF-co-HFP nanofiber due to increasing of the solution conductivity. The authors have stated that the spiropyran molecules have located near the core of the fibers according to the test results of XPS and water contact angle measurements.

Benedetto et al. [51] have reported the preparation and the characterization of electrospun photochromic PMMA fibers by incorporating the spiropyran-based photochromic compound into the polymer solution. They have specified that they used PMMA as polymer due to its high glass transition temperature, which supplies a more stable environment for the merocyanine form, thereby decelerating of the thermal decoloration rate. They have investigated the photocoloration and wettability properties of the electrospun photochromic nanofibers. It has been concluded that the electrospun spiropyran-loaded PMMA nanofibers has an opportunity to use as the light-driven nanometer-scale elements to be incorporated within optical interconnects, lab-on-a-chip technologies, and sensors.

Bianco et al. [52] have obtained diarylethene-loaded polyamide-6 nanofiber by electrospinning method and then analyzed morphology and photocoloration of the electrospun fibers. They have found a strong dichroism in the IR spectra of the diarylethene, which has confirmed the alignment of the diarylethene molecules with the main molecular axis along the fiber axis.

Gao et al. [53] have prepared photochromic fluorescence PVA nanofiber by electrospinning method. They have used three cyanostilbene derivatives as photochromic fluorescence compounds. The samples have changed their luminescence in different extents with UV irradiation for less than 1 min. The color of the fibers has been green before UV irradiation, while the color of the fibers has changed to cyan with UV irradiation. The authors have stated that the photochromic fluorescence PVA nanofibers have exhibited good reversibility and reproducibility, thereby showing potential for future practical sensor applications.

Bućko et al. [54] have synthesized azobenzene-based hybrid materials by sol-gel method and then used electrospinning method to obtain photochromic fibers. They have obtained the more beadles fibers with increasing the concentration of azo dyes in the sols. They have also measured the wettability of the samples to analyze the effect of the trans-cis isomerization on the water contact angle of the samples. The contact angle values of the samples have decreased with UV irradiation and have generally increased with increasing of electrospinning time and the dye content in the sols.

The electrospinning of azobenzene-cyclodextrin inclusion complex without using any polymer has been investigated in the study of Chen et al. [55]. They have also examined the UV response of the inclusion complexes before, during, and after electrospinning process. Before electrospinning process, the precipitation of azobenzene from the aqueous inclusion complex solution has occurred with UV irradiation. During the electrospinning process, UV irradiation has caused wider diameter distribution due to the interruption of inclusion complexes. After the electrospinning process, UV irradiation has modified the topography and adhesion forces of the electrospun nanofiber surfaces.

Photochromic superabsorber particles containing cross-linked hydrophilic core and hydrophobic azobenzene have been developed by Chen et al. [56], and then they have used various contents of these particles in the production of photochromic nanofiber by electrospinning method. Two different polymers as thermoplastic polyurethane (TPU) and polyamide (PA) have been used as carrier polymer matrix. The absorbency rate of the nanofibers has been fast; however, most of the photochromic superabsorber particles were released from the PA nanofibers after immersion in water for 24 h. The particle loss from the TPU nanofiber after third immersion cycles in water has been around 12 wt.%. It has been concluded that TPU was more stable matrix polymer for the particles with respect to PA. The absorbency capacity of the samples has increased with increasing of the photochromic superabsorber particle content

in the nanofiber. The desorption rate of the TPU nanofibers has increased with UV irradiation due to the isomerization of the azobenzene compounds.

Organic photochromic compounds have been used in all of the studies described above. However, there are also photochromic nanofiber production studies using various different photochromic systems such as tungsten oxides.

Wei et al. [57] have prepared flexible rewritable nanofiber through electrospinning of tungsten oxide (WO₃)-loaded polyvinylpyrrolidone (PVP). The images have been written onto the electrospun samples with photomasked UV irradiation, and the time necessary for photochromic nanofiber to respond to the UV irradiation has been about 10 s. The erasure time of the image has been 1–2 days under ambient conditions, while the erasure time has been 20 min by heating to 80°C and 5 min by ozone treatment. The decoloration rate of the samples has also decelerated with increasing the WO₃ concentration in the fiber. The time necessary for the total decoloration has been extended to 10 days by adding 10% polyacrylonitrile (PAN) into the PVP matrix.

Zhang et al. [58] have developed PVP nanofiber mats consisted of tungsten-doped titanium dioxide (TiO_2) with different doping concentrations to mineralize toluene under visible light. The increasing of tungsten content in the nanofibers has increased the surface area while decreased the pore diameter. The mineralization degree of toluene has increased with increasing of tungsten content although increasing more than 20% tungsten concentration has caused decreased the mineralization degree due to the distortion of the anatase TiO_2 network.

Nguyen et al. [59] have synthesized mesoporous tungsten oxide nanofibers by electrospinning and surfactant-templated sol-gel process. Polyvinylpyrrolidone (PVP) has been used as polymer matrix. They have electrospun the WO₃-PVP nanofibers and then exposed the nanofibers to the heat treatment at 120°C overnight and then calcined at 500°C in air for 3 h. The nanofiber surfaces have become rough, and the diameters of the fibers have decreased after the heat treatments due to the calcination of organic polymer ingredients.

Jin et al. [60] have investigated the detection of volatile organic compound such as acetone by W-doped TiO_2 -PVP nanofibers. The W-doped nanofibers have changed their colorless form to blue-colored form upon UV irradiation. The adsorption of acetone by the photochromic nanofibers has increased with UV irradiation, and this increase has become more apparent when the percentage of W was higher than 6%.

Unlike all these studies, Fischer and Hampp [61] have developed the photochromic PVA nanofibers with bacteriorhodopsin which is a biological photochromic pigment isolated from *Halobacterium salinarum*. They have studied on the process parameters for the electrospinning of bacteriorhodopsin-loaded PVA nanofibers.

All these studies of electrospun photochromic nanofiber production are listed in Table 3.

Photochromic compounds	Polymer matrices	Fiber diameter (nm)	References
Spiropyran	Poly(methyl methacrylate) (PMMA)	820-1430	Li et al. [33]
	Gelatin	340-870	
Spiropyran	Poly(vinylidene fluoride- cohexafluoropropylene) (PVDF-HFP)	800 ± 70	Genovese et al. [34]
Spiropyran	Polystyrene (PS)	500	Liao et al. [35]
Spirooxazine	Thermoplastic polyurethane (TPU)	700–900	Akcakoca Kumbasar et al. [36]
Spirooxazine	Polyurethane (PU)	610–4720	Durasevic [37]
Spiropyran	Polyvinyl alcohol (PVA)	307 ± 53	Khatri et al. [38]
Spirooxazine		349 ± 54	
Spiropyran	Poly(ε-caprolactone)	1130 ± 235	Ali et al. [39]
Spirooxazine	Polyvinylidene fluoride (PVDF)	1000 ± 200	Zillohu et al. [40]
Naphthopyran	Polyvinylpyrrolidone (PVP)	200-400	Liu et al. [41]
Diarylethene	Polystyrene (PS)		Lee and Kim [42]
	Polyacrylic acid (PAA)	800	
Spiropyran	Polystyrene (PS)	70	Zhang et al. [43]
Spironaphthoxazine/isophorone-fluorescent dye	Poly(methyl methacrylate) (PMMA)	400-1000	Lee et al. [44]
Spironaphthoxazine/D-p-A-type fluorescent dye (TCF)	Poly(methyl methacrylate) (PMMA)	400-1000	Lee et al. [45]
Spiropyran	Poly(methyl methacrylate) (PMMA)	_	Nammoonnoy et al. [46]
Spiropyran	Cellulose acetate (CA)	30-490	Shuiping et al. [47]
Spiropyran	Poly(ethylene oxide) (PEO)	2	Lee et al. [48]
	Polystyrene (PS)		
Spiropyran	Cyclodextrin molecule (βCD)	422 ± 40	De Sousa et al. [49]
	Poly(methacrylic acid) (PMAA)	526 ± 24	
Spiropyran	Poly(vinylidene fluoride- co-hexafluoropropylene) (PVDF-co-HFP)	2250 ± 190	Wang et al. [50]
Spiropyran	Poly(methacrylic acid) (PMAA)	50-400	Benedetto et al. [51]
Diarylethene	Polyamide 6	150	Bianco et al. [52]
Stilbene	Poly(vinyl alcohol) (PVA)	_	Gao et al. [53]

Photochromic compounds	Polymer matrices	Fiber diameter (nm)	References
Azobenzene	Triethoxyphenylsilane	300-1000	Bućko et al. [54]
Azobenzene	Hydroxypropyl-b-cyclodextrin (HPbCD)	$460 \pm 170 -$ 2320 ± 1140	Chen et al. [55]
Azobenzene/photochromic superabsorber particles	Thermoplastic polyurethane (TPU)	500-1000	Chen et al. [56]
	Polyamide (PA)		
Tungsten oxide (WO ₃)	Polyvinylpyrrolidone (PVP)	200–1000	Wei et al. [57]
Tungsten-doped titanium dioxide (TiO ₂)	Polyvinylpyrrolidone (PVP)	200–500	Zhang et al. [58]
Tungsten oxide (WO ₃)	Polyvinylpyrrolidone (PVP)	233	Nguyen et al. [59]
Tungsten-doped titanium dioxide (TiO_2)	Polyvinylpyrrolidone (PVP)	800–900	Jin et al. [60]
Bacteriorhodopsin	Poly(vinyl alcohol) (PVA)	300-800	Fischer and Hampp [61]

Table 3. The studies of electrospun photochromic nanofiber production.

5. Conclusions

In this review, electrospun nanofiber production, functionalization of the nanofiber with photochromic compounds, and recent research development in this area have been described. Although the studies on photochromic nanofibers have been carried out, more detailed studies should be done on the production of the electrospun photochromic nanofibers, and alternative usage areas can be generated for these fibers.

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