Mixed Metal and Metal Oxide Nanofibers: Preparation, Fabrication, and Applications

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Abstract

The review presents the overview of the design, synthesis, fabrication, and applications of metal and metal oxide-based nanofibers. Furthermore, it also describe the recent trends in methodologies used and types of nanofibers produced; including polymers, nanoparticles and ionic liquid based nanofibers. The great deal of interest and attention is currently being focused by the scientific community on nano- and mesostructured materials. The quest for the establishment of these classes of nanosystems has created new challenges in their research development processes. Nanofibers are fibers with the size of around 100 nm, which are made from various inorganic materials such as ceramics, metal, and metal oxides. This review describes the synthesis, fabrication, and some of the selected applications of nanofibers in recent years. Mixed metals and their oxides give rise to a significant class of nanofibers with fascinating properties. The metals and metal oxides which are used for the synthesis of nanofibers are, inter alia, Si, Ba, Ti, Mn, Cu, Fe, Sn, Sb, Ni, Mo, CeO_2, TiO_2, CuO, Fe_2O_3, MnO_2, SnO_2, and $NiCo_2O_4$. Generally, the important parameters such as morphology, size distribution, and composition in homogeneous systems are difficult to control, and, in the fabrication of new materials, the nanometer size of the dimensions should be maintained within the accepted ranges. Presently, microscale hierarchically structured nanomaterial-based nanofibers are focused and developed toward future technology. Moreover, the increasing prominence and significance of the nanofiber industry are, in recent years, due to the heightened awareness of their capability to improve the performance of different forms of filter media. In addition, nanofibers have fascinating properties and advantages such as high surface area, small pore size, and high pore volume, over conventional fibers. Generally, nanofibers are applied in various fields such as wound dressings, filtration and cleaning processes, tissue engineering scaffolds, drug release materials, energy storage, environmental engineering, defense, and security systems.

Keywords

Nanofiber · Metal oxide · Electrospinning · Force spinning · Fabrications

Introduction

A great deal of interest and attention is being focused by the scientific community on nano- and mesostructured materials. The quest for the establishment and development of these classes of nanosystems has created new challenges and better outcomes on nano-based research. One new class that has arisen is nanofibers (NFs) which are fibers with the size of around 100 nm. NFs are currently produced from various inorganic materials such as ceramics, metal, and metal oxides. NFs have fascinating properties and advantages over conventional fibers such as high surface area, small pore size, and high pore volume. Mixed metals and their oxides give rise to the importance of NFs with attractive properties. The metals and metal oxides which are used for the synthesis of NFs include Si, Ba, Ti, Mn, Cu, Fe, Sn, Sb, Ni, Mo, CeO_2, TiO_2, CuO, Fe_2O_3, MnO_2, SnO_2, and $NiCo_2O_4$. Generally, important parameters, such as morphology, size distribution, and composition, in homogeneous systems, are difficult to control, and, in the fabrication of new materials, the nanometer size of the dimensions should be maintained within the accepted ranges. Very small NFs, having less than 1 μ in diameter, have been fabricated and produced for many years. Electrospinning is one of the most important processes for manufacturing NFs. This chapter describes the synthesis, fabrication, and some selected applications of NFs in recent years.

The National Science Foundation (NFS) describes NFs having a minimum size of 100 nanometers (nm) or less, while modern industries define NFs as containing a diameter of less than 1 μ. The great properties of the low-dimensional structured NFs and nanowires such as high surface area to volume ratio and remarkable porosity are the main reason for the fabrication of excellent sensing nanomaterials [1]. Presently, microscale hierarchically structured nanomaterial-based NFs are focused and developed toward future technology. Moreover, the increasing prominence and importance of the NF industry in recent years are due to the heightened awareness of their capability to improve the performance of different forms of filter media. NFs are new types of material used for numerous value-added applications such as energy storage, insulation, composites, garments, wipes, filtration, barrier, personal care, aerospace, transistors, battery separators, fuel cells, information technology, pharmaceutical, wound dressings, cleaning processes, tissue engineering scaffolds, environmental engineering, defense, and security systems.

Metal/Metal Oxide NFs

Recently, a huge scientific interest on one-dimensional metal oxide NFs has arisen due to the improved physical and chemical properties and potential applications of new materials in various fields. The unique characteristic of NFs such as high porosity, large surface area to volume ratio, high aspect ratio, continuous structured morphology like controllable fiber diameter, and low density makes them the perfect candidates for wider applications especially in water treatment [1] and energy storage materials [2, 3]. When NFs are applied to electrodes in lithium-ion batteries, these great properties make advantageous nanostructured fibrous materials compared to their nanowires, thin films, nanopowder, and crystals. A stable cycle performance, superior low-temperature performance, and enhanced capacity are the most comparable electrochemical performance delivered through electrodes of these metal/metal oxides and ceramic composite NFs. During the charge and discharge cycles in lithium-ion battery, short diffusion distance of Li-ions was achieved due to physical properties such as large surface, high labyrinth-like porous structure, and interfacial areas of improved composite NFs.

In contrast to nanoparticles, the structure of nanofibrous materials permits their retrieval and ease of handling throughout the processes of treatment. Important metal oxides such as TiO_2, ZnO, NiO, Fe_2O_3, SnO_2, CuO, ZnO/TiO_2, ZnO/SnO_2, etc. have been used alone or as a system of composites in the removal of bacterial pollutants, in the separation of toxic heavy metal ions, and in the elimination of hazardous chemical dyes. A recent investigation [2] reported on the removal of chromate ions from wastewater samples. This involved the application of newly prepared Fe_2O_3 NFs, and their resulting efficiency was compared with Fe_2O_3 nanoparticles: the results clearly highlighted the excellent efficacy of the Fe_2O_3 NF activity. Different classes of nanostructured fibers were synthesized from various materials such as ceramics, metals, metal oxides, and nanocomposites and subsequently developed for application in several energy storage devices. In particular, more reactive sites are created, while the porous surface of the structure delivers buffering effect to accommodate the huge changes of volume frequently associated with the alloying/dealloying of lithium with ceramic, metal/metal oxide composite materials [2].

NFs display superior gas sensing properties due to their excellent physicochemical characteristics of large surface area and high porosity. These unique traits of the metal oxide NFs contribute to their applications as gas sensors, due to pseudo-one-dimensional structure, semiconducting, and large surface area. Moreover, the controlling of morphology and microstructures of metal oxide NFs further enhances the sensitivity of toward various gases. Their outstanding properties of flexible transport characteristics and unique surface chemistry strongly influence their utility to harvest mechanical, chemical, solar, and thermal energy and electrical as well as electrochemical applications [3]. Electrospinning is an appropriate facile and versatile approach to synthesis of polymeric NFs of inorganic, organic, and hybrid materials with long uniform structure of fibrous and size diameter that ranges from submicron to nanometers. This is one of the best methods to build polymeric NFs compared to other methods [4]. Researchers initially reported on the metal oxide precursor, namely, ferric acetylacetonate $Fe(acac)_3$, which subsequently led to many of the electrospun of nonwoven structured fibrous membranes.

Metal/Carbonaceous NFs

Generally, metal oxides such as $Fe(acac)_3$ magnetite dissolved in polyacrylonitrile (PAN)/N and *N*-dimethylformamide (DMF) followed by carbonization are used to prepare metal oxide/carbon nanocomposite fibers. This is done by using either electrospinning methodology [5] or PAN/DMF solution that is used as a medium for dispersing formerly synthesized metal oxide nanoparticles. This solution is used for electrospinning solution and finally for carbonization [6]. The functionalized electrospun NFs were further developed using composite nanostructures such as photocatalytic semiconductor, carbon materials, and metals [7]. High reversible capacity ranges from 600 to 1200 mAg^{-1} have been achieved by the fabrication and demonstration of several nanostructured carbon materials such as carbon spheres, carbon NFs, graphene, carbon nitride, and carbon nanotubes [8]. Furthermore, the availability of the huge specific surface area of modified reversible capacity of the nanostructured carbon materials contributes to improved electrode/electrolyte interaction and large reaction active sites for lithium ions. However, the high-rate charge/discharging conditions led to poor structural stability, and safety performance was compromised. Therefore, the scope for the development of numerous anode materials is available for high-performance lithium-ion batteries.

The one-dimensional nanostructured carbon NFs (CNFs) exhibit better properties, among several carbonaceous materials, such as superior thermal and electrical conductivity, and in particular it helps to reduce the diffusion paths for electrons and ions during the electrochemical process [9]. Recently researchers have investigated ways to strengthen the carbon NFs using composites of metal oxides. The post-synthetic procedure could be more suitable for the fabrication of metal/metal oxide-carbon NF composites; generally, electrospinning, chemical vapor deposition (CVD), or catalytic chemical vapor deposition (CCVD) methods are used to pre-synthesize the carbon NFs, and thereafter impregnation and reduction process would be applied to the metal salts [10]. These synthesized composites have loosely bonded metal oxides, which are randomly spread over the external surface area and finally eroding the reactivity and stability of these composite materials. Instead of this inefficient preparation, an efficient fabrication of consistently dispersed oxides of metals steadfastly attached to the carbon NFs is proved by the pyrolysis of electrospun fibers within metal and carbon forerunners [11]. However, subsequent composites displayed extremely low surface area and porosity which often involve some pore-forming reagents such as silica and surfactants, which are generally used to control the power capacity [12]; thus, one big challenge faced by the scientific community is the facile fabrication of ultrafine high-performance metal oxide-carbon NFs composites.

Recent Trends

Recently, nanostructured fibrous metal and mixed metal oxide materials are being widely used, due to their much larger surface area, shorter diffusion paths for Li-ions, and good strain relaxation compared with other bulk materials, which helps to enable the insertion/extraction of Li-ions [13]. This is a significant development in the usage of nanostructured fibrous electrode materials for the performance of LIBs. Electrospinning techniques were applied to prepare numerous classes of ternary transition metal oxides; for example, tin ternary composites (SnO_2/ZnO) and heterogeneous nanostructured fiber-based materials, have been developed for LIBs. Furthermore, electrochemical performance was enhanced, when applying alloy carbon NFs of Co-Sn and coaxial NFs of Fe_3O_4/SnO_2 as anodes in LIBs [14]. Among various NFs, nanostructured fibers of spinel oxide materials work well; for instance, $MnFe_2O_4$ NFs not only have great surface area to volume ratio but also have unique properties, such as good electrical conductivity and high thermal stability. Therefore, nanosensors, nanofiltration, and nanoelectronic industries are embarking on using spinal oxide NFs.

The oxides of transition metals such as NiO, MnO_2, Co_3O_4, RuO_2, and SnO_2 influence the pseudocapacitance of a material. However, their efficient application has been hindered by high cost, low electrical conductivity, poor rate capability, and reversibility during the charge-discharge process. Consequently, theoretical studies and research experiments are developed for low-cost alternatives. Numerous metal oxides such as TiO_2, ZnO, and Fe_2O_4 have been considered: TiO_2 is extensively applied in the field of many energy storage systems such as Li-ion batteries, solar cells, and photocatalysts due to their thermochemical stability, economic abundance, and environmental benignity. The development of one-dimensional ceria-based nanostructured fibers was achieved using the versatile technique of electrospinning, and the binary systems with no samarium doped such as CeO_2-ZnO, CeO_2-ZrO_2, CeO_2-Nd_2O_3, CeO_2-CuO, and CeO_2-TiO_2 were studied intensively. Literature on ternary composition of Ce-Sm in the ratio of $Sm_{0.5}Sr_{0.5}Co_{0.3}$ and other oxides of multiple mixed metals configuration like those of $Sm_{0.5}Sr_{0.5}CoO_3$ δ $Gd_{0.2}Ce_{0.8}O_{1.9}$ is available [15]. Compared to several oxides of transition metals, copper oxide has been much used for energy applications, due to their unique properties such as nontoxicity, being environmentally friendly, high theoretical capacity, abundance, chemical stability, and ease of synthesis of the materials. Lately, researchers found that incorporation of highly active catalyst support such as NiO/TiO_2, NiO/SiO_2, NiO/PVA, and NiO/ZnO composite preparation of NFs could be an efficient method to address these issues.

Fabrication and Preparation of NFs

In the coming years, many experimental methods (see Table 1) could be developed such as electrospinning, electrospray, reactive magnets spinning, liquid shearing, and centrifugal and rotary jet spinning techniques applied to produce many NFs [60]. Besides this, the cost, as well as properties, could be greatly affected by these routes of synthesis. In particular, metal oxide NFs could be synthesized through several new techniques such as spin coating, thermal evaporation, dip coating, hydrothermal technique, vapor deposition, mechanochemical methods, electrospinning, and electrodeposition [61]. Of these, electrospinning appears as an economic, simple, and well-used technique for the fabrication of NFs. Generally, electrospun NFs are effective for a wide range of industrial applications such as sensors, laser ablation, nano-lithography, self-assembly, tissue engineering scaffolds, sol-gel, microwave-assisted synthesis, adsorption, catalyst, and filtration.

Electrospinning

Electrospinning technique is one of the most important approaches for fabrication of one-dimensional nanostructured materials such as NFs, hollow NFs, Janus NFs, Janus nanobelts, nanobelts, and ribbon-shaped coaxial nanocables via convenient, direct techniques, and it has economical implication as well. It is the best and effective technique for the fabrication of ultrathin nanostructured fibers, due to the evaporation of solvents and the electrostatic repulsions between the surface charges; thus continuous fibers with high quality can be produced. The fabrication of various classes of NFs such as metallic NFs, carbon NFs, and polymeric NFs can be accomplished using electrospinning techniques. Specifically, metal oxide NFs are synthesized with porous morphology and huge surface area to volume ratio through an efficient and versatile technique of electrospinning. The products from electrospinning are more appropriate for enhancing the sensitivity of sensing materials. Generally, several significant advantages of NFs which are prepared by electrospinning

Table 1 Source of metal substance, synthesis methodology, and their application of various classes of metal/metal oxide nanofibers

NF name	Metal/metal oxide	Synthesis methodology	Application	Ref
CuO NFs	CuO	Electrospinning technique	Electrochemical performance as lithium-ion battery anode	[16]
ZnO NFs	ZnO	Electrospinning technique	–	[17]
Fe_2O_3 NFs	Fe_2O_3	Electrospinning technique	Chromate adsorption from contaminated water sources	[18]
Cobalt NFs	Co	Electrospinning technique	–	[19]
CeO_2 NFs	Ce	140 nm were successfully synthesized by single-capillary electrospinning	Pollutant removal	[20]
Co_3O_4 NFs	Co_3O_4	Electrospinning and post-thermal and hydrothermal treatments	Electrode for fast discharge and charge of LIBs	[21]
Nickel oxide NFs	NiO_2	Electrospinning technique	Gas sensing behavior (hydrogen and ammonia sensors)	[22]
$CoMn_2O_4$ NFs	$CoMn_2O_4$	Electrospinning technique	Electrochemical energy storage	[23]
Hydroxylated α-Fe_2O_3 NF	Fe_2O_3	Electrospinning technique	Anionic dye adsorption	[24]
Beaded manganese oxide (Mn_2O_3) NFs	Mn_2O_3	Electrospinning technique	Capacitive energy storage	[25]
Electrospun manganese (III) oxide NF		Electrospinning technique	Electrochemical DNA nanobiosensor for zeptomolar detection of dengue consensus primer	[26]
Ag-doped $LaFeO_3$ NFs	Ag, $LaFeO_3$	Electrospinning technique	Formaldehyde gas sensor	[27]
CuO-ZnO composite NFs	CuO-ZnO	Electrospinning technique with post-calcination	Efficient adsorbents for removal of Congo red dye and as antibacterial agents	[28]
CuO/TiO_2 hybrid NFs	CuO/TiO_2	Facile electrospinning technique followed with calcination	Photocatalytic activity for organic dye degradation	[29]
NFs of CeO_2 and CuO/CeO_2	CuO/CeO_2	Electrospinning technique	–	[30]

(*continued*)

Table 1 (continued)

NF name	Metal/metal oxide	Synthesis methodology	Application	Ref
SnO_2-doped MoO_3 NFs	SnO_2/MoO_3	Wet-chemical method	Sensors	[31]
$ZnO/CoNiO_2$ hollow NFs	ZnO, $CoNiO_2$	Electrospinning method and post-calcination	Gas sensing	[32]
TiO_2 NF by fluorination	TiO_2	Fabrication	Excellent water desalination performance	[33]
Nickel tungstate ($NiWO_4$) NF	Ni, WO_4	Electrospinning technique	Anodes for Li-ion batteries	[34]
Spinel gallate (MGa_2O_4; Ni, Cu, Co) NFs	Cu, Co, Ni, Ga_2O_4	Electrospinning technique	Gas sensing properties for benzene detection	[35]
$CuO/ZnO/TiO_2$ NF	$CuO/ZnO/TiO_2$	Electrospinning technique	Highly efficient photocathodes	[36]
BaFCl and $BaFCl:Eu^{2+}$ NFs	Ba, Eu	Calcination of electrospinning-made	Photoluminescence	[37]
Pt/TiO_2 hollow NFs	Pt/TiO_2	Uniaxial electrospinning method combined with photodeposition	Photocatalysts	[38]
Eu-doped SnO_2 electrospun NFs	Eu/SnO_2	Synthesized via a simple electrospinning technique and subsequent calcination treatment	Sensors	[39]
Metal-mixed ceramic NFs	Composed of CuO, Al_2O_3, and $Cu_2Al_4O_7$ phases	Fabricated through two-nozzle electrospinning method followed by calcination	Nanocatalyst	[40]
Tungsten trioxide NFs decorated with palladium oxide nanoparticles	Tungsten trioxide (WO_3)-based NFs	Electrospinning combined with chemical deposition processes	Photodegradation of methylene blue	[41]
Palladium-decorated TiO_2 NFs containing anatase-rutile mixed phase	Pd, TiO_2	Alkaline hydrothermal synthesis and calcination	Photocatalyst	[42]
$CaCu_3Ti_4O_{12}$ NFs	$CaCu_3Ti_4O_{12}$	Electrospinning technique	–	[43]
NFs of spinel-$CdMn_2O_4$	$CdMn_2O_4$	Electrospinning technique	Supercapacitor and Li-ion batteries	[44]

(*continued*)

Table 1 (continued)

NF name	Metal/metal oxide	Synthesis methodology	Application	Ref
Composite NFs of NiO/γ-Al_2O_3	NiO/Al_2O_3	Processing and electrospinning followed by calcination	Investigated by evaluating the photodegradation of hazardous dye Rhodamine B	[45]
Electrospun NFs	Co-NF, Ni-NF, and Cu-NF	Electrospinning technique	Efficient catalysts for hydrogen release through ammonia borane (NH_3BH_3) hydrolysis	[46]
Transition/alkaline earth metal oxide composite NFs (TAMNs)	$Ca_3Co_4O_9$ and $MgCo_3O_5$	Electrospinning followed by calcinations	Hydrogen evolution	[47]
CuO/NiO composite NFs	CuO/NiO	Electrospinning technique	Electrocatalytic oxidation of hydrazine	[48]
SnO_2-CuO composite NFs	SnO_2-CuO	Electrospinning method and followed by annealing	Detecting of CO	[49]
Molybdenum (Mo-Pt_3Ni/CN) NF	Mo, Ni, Pt	Electrospinning technique	Counter electrode in dye-sensitized solar cells (DSSC)	[50]
Electrospun NiO-SnO_2 NFs	NiO-SnO_2	Electrospinning technique	Excellent performances for humidity sensors	[51]
ZnO/Zn (OH)F NF	ZnO-Zn	Inner surface of confined capillary microchannels (CMs)	Photodegrade methylene blue	[52]
MoS_2/CdS-TiO_2 NFs	Mo, TiO_2	Electrospinning-mediated photodeposition	Highly efficient photocatalytic H_2 evolution	[53]
Co-doped SnO_2 NFs	Co, SnO_2	Electrospinning technique	Gas sensing properties	[54]
LaOCl-doped SnO_2 NFs	La, SnO_2	Simple one-step electrospinning technique	CO_2 detection	[55]
TiO_2-SnO_2 core-shell heterostructure NFs	Ti, SnO_2	Coaxial electrospinning approach	Gas sensor	[56]
WO_3 NFs functionalized by protein-templated RuO_2 nanoparticles	WO_3 NFs, RuO_2	Chemical reduction technique	Electrocatalytic oxidation and nonenzymatic detection of urea	[57]

(*continued*)

Table 1 (continued)

NF name	Metal/metal oxide	Synthesis methodology	Application	Ref
GO/SnO_2/TiO_2 NFs	SnO_2/TiO_2	Electrospinning and hydrothermal processes	Photovoltaics	[58]
Graphene oxide/ thornlike titanium dioxide NF aerogels	TiO_2	Electrospinning, silica etching, and hydrothermal combination method	Supercapacitor	[59]

include high aspect ratio, design flexibility for chemical/physical surface functionalization, large surface area, small diameter, and diversity in composition. This method produces small NFs in several size diameter ranges from the nanoscale up to microscale in an economical way. Recently, composite nanostructured fibrous materials such as polymer/inorganic and carbon NFs with Fe_3O_4 nanoparticles have been prepared using electrospinning techniques as shown in Table 1 [6]. Furthermore, numerous polymeric composite NFs have been synthesized using mixtures of metal oxide nanoparticles, carbonized materials, and organic/inorganic compounds with PVP through electrospinning techniques. The principle of electrostatic attraction of charges plays an important role in the technology of fiber formation. These polymeric solutions have particular surface tension inside the syringe. The tip of the needle subjected to high-voltage power supply to charge this polymeric solution. The ejected fibers were collected in oppositely charged collectors, which are placed at some distance (10 cm) apart. While supplying high voltage (10–30 kV), due to the surface tension, the precursor solution emerges out on the syringe, and drops get ejected at the tip of the needle to form a cone-like structure is known as "Taylor cone." The high charge density on the collector can be used to elongate the Taylor cone. Figure 1 shows the working principle of electrospinning technique. This technique of electrospinning is a process of electrostatic fiber formation using electrical forces to produce polymer fibers from polymer solution. This electrostatic method involves the ejection of polymer solution droplets from a syringe needle which is under high voltage [62].

Force Spinning Method

The force spinning (FS) method is another important technique used to synthesize different types of NFs (Table 1). The development and various features of FS such as producing 100% yield, ability to accompany dual material feed that permits continuous material feed, and melt spinning with melt up to 350 °C with solvent-free processes are attractive. Moreover, this technique could produce an eco-friendly, safe, and low-cost method of operation compared to other methods which are used to making of NFs such as electrospinning, in-house built centrifugal and melt-blowing systems [64]. Generally, FS technique operates using centrifugal forces to extrude the polymer solutions or melts through the spinneret. The quantity of the precursor solutions (2 mL)

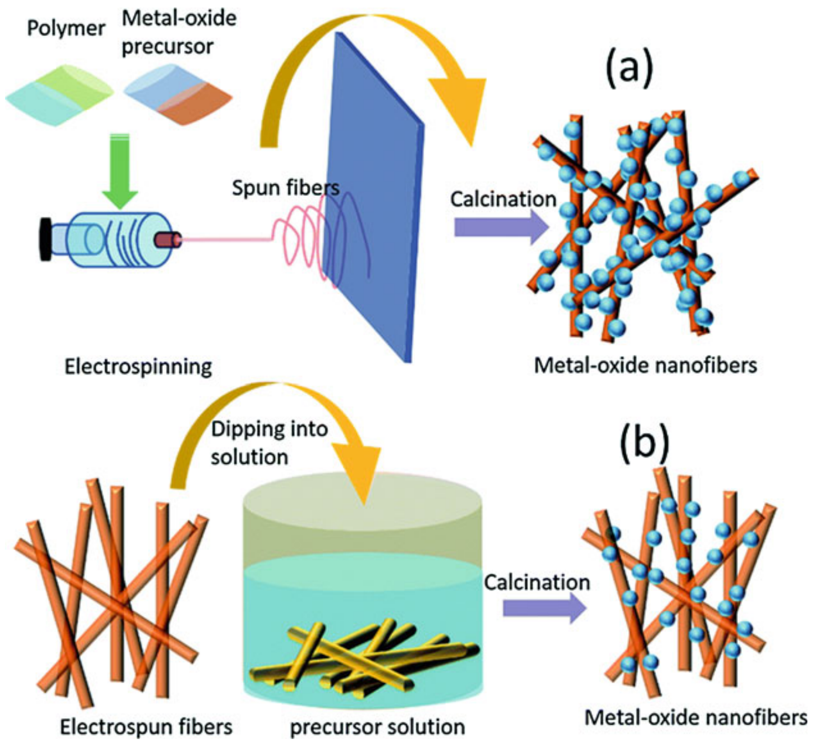

Fig. 1 Preparation of metal oxide nanofiber using electrospinning method [63]

is injected into the spinneret containing 30-gauge half-inch regular bevel needle. The speed of rotation for spinneret is fixed at 8000 rpm. After each run the substrate is rotated to 90°, and after each run the needles are changed; for example, Sn/PAN and SnO_2/NiO/PAN nanostructured fibrous materials were synthesized in the method of force spinning as well as carbonization of the material after collection from the substrate drum. It was dried at 120 °C under vacuum. Furthermore, stabilization of force spun NFs in air at 280 °C for 5 h was followed by calcination at 800 °C for 2 h in argon atmosphere that produces Sn/C and SnO_2/NiO/C composite NF materials.

General Synthesis Methodology

Synthesis of Metal and Metal Oxide NF

Synthesis of Fe_2O_3 NFs

The procedure for synthesis of Fe_2O_3 NFs begins with polymer solution preparations. Iron (III) 2-ethyl hexane-isopropoxide (3 mL) and acetic acid (0.3 mL) and the required quantity of PVP were added to a 30 mL glass beaker, and a magnetic stirrer was used to agitate for 30 min at the rate of 300 rpm. In a setup for an electrospinning process, a distance between the needle and collecting drum was fixed at 10 cm, the rate of feed was varied from 0.2 to 0.45/hand, and the voltage was varied from 10 to 24 kV. After feeding the electrospinning, the annealing process was started for Fe/PVP NFs at 500 °C and kept for 3 h at the rate of 3 °C/min. The PVP polymer was removed from prepared NFs by the annealing process, and Fe precursor was crystallized into a pure metal oxide Fe_2O_3 NFs (see Table 1). The final Fe_2O_3 NFs were dried after the annealing process and were kept as dry samples in 20 mL glass vials for characterization and preparation of stock suspensions using these NFs in double-deionized water for zeta potential and test for adsorption. The confirmed synthesized Fe_2O_3 NFs were compared with commercial Fe_2O_3 nanopowder.

Fabrications of NF (Preparation of Metal/PAN NFs)

Generally, the fabrication of NFs especially metal NFs were obtained using the following procedure: generally, certain specific concentration of a polymer solution such as PAN has been prepared in DMF based on the weight and ensured that the solution was homogeneous for electrospinning. Various inorganic salts, for instance, $Co(CH_3COO).24H_2O$ and $Ni(CH_3COO).24H_2O$, or nanoparticle samples were prepared in different millimolar concentrations. Furthermore, these precursors of metals were added prior to the electrospinning process. In cases where it was needed, any class of ligands could be added to the solution which contains metal precursor premix before mixing with the polymer solution. In particular, the multi-spinneret was used for electrospinning process with three syringes at a flow of 2–3 mL for 1 h, 15 cm distance, and 29.9 KV and collected at 300 rpm rotation. The thermal treatment, in a carbolite furnace, was completed in a multistage process based on the first step of heating at 1 °C/min up to 280 °C; the time for dwelling is 1 h at 280 °C under air, and then the second step for heating at 5 °C/min up to 1000 °C with a time for dwelling is 1 h under N_2 atm. The same process was used to prepare pristine CNFs.

Preparation of Porous Metal Oxide/GO NFs

Normally, natural flakes of graphite powder were used to prepare graphene oxide, according to the Hummers modified method. The precursor solution is prepared by dissolving 67.5 mg of GO and 6.0 g of $Fe(acac)_3$ in 50 mL of DMF in ultrasonic condition for 3 h at room temperature, and thereafter this solution applied for electrospinning porous GO/Fe_3O_4 NFs. PAN (3.8 g) and PMMA (2.0 g) were also added to this solution which was agitated for 5 h at 80 °C. A syringe with single nozzle was used to load these attained solutions. The conditions for electrospinning process were 20 kv of voltage and 0.5 mL/h flow rate, and a 15 cm distance was maintained from the needle to the collector. The prepared precursor fibers were removed from the collector and stabilized in the air for 8 h at 250 °C. Finally, the prepared NF samples were heated at 650 °C at the heating rate of 2 °C/min and then kept at 650 °C for 10 h. Moreover, the same procedure could be applied to synthesize various metal oxide/nanoparticles/GO composite NFs.

Preparation of Metal Oxide/Carbon NFs

A typical procedure for the synthesis of metal oxide/carbon NFs is as follows: Mn- and Zn-containing metal organic framework NFs using $MnCl_2.4H_2O$, $Zn(CH_3COO)_2.2H_2O$, and trimesic acid (H_3BTC) as precursors were dissolved in DMF. Specifically, the mixture of precursor metal solution was prepared in particular molar ratio of Mn and Zn metal ions in a set of several concentrations. Classically, H_3BTC (1.25 g) was combined with a certain amount of metal salts dissolved at constant stirring in 50 mL of DMF, and thereafter this mixture was subjected to a solvothermal process for 750 min at 140 °C. The final precipitate was collected from reaction mixture; after decantation of the mother liquid, the precipitate washed with DMF and good yield was obtained after drying under vacuum to get Mn-ZnBTC NFs. A quartz boat was used to place the synthesized Mn-ZnBTC NF, and the tube furnace was introduced to heat the NF up to 950 °C for 2 h and at the rate of 50 min under N_2 atmosphere to obtain a high yield of the manganese oxide-carbon composite NFs of Mn-CNF.

Synthesis of Thornlike TiO_2 NFs

The thornlike TiO_2 NF (TTF) was synthesized via scalable electrospinning techniques. Initially, a homogeneous mixture of 2.1 mL of TBT, 0.5 mL of TEOS, 34 mL of MeOH, and 1.5 mL of HAc using magnetic agitator for 1 h was prepared; 1.5 g of PVP was added to this mixture and stirred for the next 6 h at room temperature. A syringe (12 mL) with 23G needle, was used to transfer the solution. A voltage of 18 kV was applied, and a distance from the needle to the collector of aluminum foil of 12 cm was maintained, at the flow rate of 1 mL/h, which was attained using a syringe pump. During spinning process, the temperature and relative humidity were maintained at 25 ± 2 °C and $50 \pm 5\%$, respectively. The resulting TiO_2/SiO_2 NFs were dried for 12 h at 80 °C and thereafter calcinated for 2 h at 500 °C. Further, the process of alkaline treatment was done by the use of a silica-etching technique. In addition, the prepared TiO_2/SiO_2 NFs were transferred to a Teflon beaker which contained 75 mL of 5 M solution of NaOH. The final solution was heated for 6 h at 60 °C under constant stirring in a water bath. Subsequently, the removal of the residual sodium salt from these prepared NFs was achieved by transferring this NF to a beaker containing a solution of 0.1 M HCl and kept undisturbed for 12 h. Moreover, the product was filtered from acid solutions and washed with deionized water until the filtrate reaches neutral pH. The yield of the final product of TTF was dried for 24 h at 80 °C in vacuum.

Characterization of NFs

In recent decade, numerous advanced techniques were used to characterize nanomaterials such as NFs by determining their morphologies, crystalline nature, functional groups which are present, nature of elemental bonds, and surface smoothness. Basically, Fourier-transform infrared spectroscopy (FTIR) was used to determine the presence of the functional groups, ligands, and capping agents in synthesized NFs.

X-ray diffraction (XRD) has been used to examine the crystallinity and purity of the NFs. In particular, XRD has helped to deduce the specific changes in the reduction of metal ions such as Co^{2+}, Ni^{2+}, Fe^{2+}, or M^{2+} to $Co^{(0)}$, $Ni^{(0)}$, $Fe^{(0)}$, and $M^{(0)}$. Scanning electron microscope (SEM) was applied to inspect the morphology and average diameter of NFs and also explore the overall structure and surface of the porous NFs. This information showed the diameters ranged from 100 to 150 nm with random distribution of NFs through high-resolution SEM operated at 5 kV. The detailed morphology of NFs was evaluated using transmission electron microscopy (TEM). Furthermore, the advanced high-resolution transmission electron microscope (HRTEM) was used to investigate the specific microstructure of the NFs such as composite, carbon structure, and nanoparticles in NFs. This clearly describes their uniqueness of the nanostructure, and it was operated at 200 kV. The highly disordered structure of the synthesized porous N-doped NFs was identified using information from a Raman spectrum. X-ray photoelectron spectroscopy (XPS) yielded significant information about the chemical bond energies of the particles which are on the surfaces. Additionally, the whole chemical composition of the NFs was evaluated using XPS. The following parameters were used, Al K radiation, 1486 eV; pass energy, 23.5 eV; step size, 0.1 eV; takeoff angle, 45°; and X-ray spot size, 200 μ, and were maintained for XPS. The carbon 1s peak was used as a reference peak to judge the XPS peak positions, which displayed closely the same intensity in all the spectra of XPS, because of some negligible amount of organic contaminations on the surface of the NFs. The gas analyzer is used to examine the textural properties of the NFs via nitrogen adsorption-desorption isotherms at the liquid nitrogen temperature (77 K). Brunauer-Emmett-Teller (BET) and Barrett-Joyner-Halenda (BJH) techniques were used to investigate following specific surface area and pore size distribution. Brunauer-Emmett-Teller (BET) method was used to estimate the surface area of the NFs.

Applications of Metal/Metal Oxide NFs

Metal oxide nanofibers have attracted considerable attention due to thier one-dimensional morphology and unique chemical and electrical properties. applications being developed for nanocomposites include gas sensors, biosensors, battieres, supercapastors, Photovoltaic Devices, and others (Fig. 2).

Gas Sensor

Compared to different forms of nanostructured metal oxides, the NFs have evoked special interest especially for gas sensing applications. The following physical proper ties, namely, small diameter, huge specific surface area and long length are believed to contribute to sensing applications of nanofibers. This makes the gas sensing applications by ideal nanostructured NFs more suitable. The remarkable behavior of gas sensing has been reported in recent times using several physical and

Fig. 2 Different applications of nanofibers from gas sensors and batteries to water treatment

chemical routes to synthesizing a wide range of metal oxide NFs. The summarized resulting data of several metal oxide NFs and their characterization for gas sensing properties have been reported in numerous review articles. To optimize the morphology and microstructure is a significant key area for commercializing the metal oxide NFs used as an appropriate material for gas sensing. Although the potential of various metal oxide NF-based gas sensors was established by the recent extensive research, the gas sensing behavior could be more related to the existing morphology and microstructure of these metal NFs. Incidentally, the advantages of the electrospinning technique are that it is not only scalable, versatile, and economical, it also delivers ready control over morphology of NFs by switch of the processing variables such as composition of solution and parameters of electrospinning such as rate of flow, voltage, etc. Among various methods, the electrospinning technique is the only method capable of providing NFs with very long length which is frequently not possible via other fabrication methods. The semiconducting metal oxide NFs have the most favorable metal system for conductometric gas sensors.

Analytical

Metal/metal oxide NFs have been applied for the purpose of determining the activity of biologically materials and drug molecules. The antibacterial properties against both gram-positive and gram-negative bacterial activities were investigated by using CuO-ZnO NFs. In addition, the effect of aqueous Pd(II) on the uptake capacity of CdO codoped Fe_2O_3 NFs for metal (II) ions was found to be significant.

Recently the electrochemical biosensor efficacy of in situ electro-synthesized manganese oxide (Mn_3O_4)$^{-}$polyaniline (PANI) NFs through the detection of biologically active bisphenol A (BPA), an endocrine-disrupting chemical, was reported.

Capacitor

The great physical and chemical properties of metal NFs contribute to the application of metal NFs in capacitors. For example, facile and economic single nozzle NFs like coaxial-cable Mn_2O_3 were fabricated by electrospinning method and followed by applied calcination process. Furthermore, the synthesized Mn_2O_3 NF was utilized as an electrode for supercapacitor in six MKOH aqueous conditions, due to their excellent physical properties of large specific surface area and porous structures. In addition, these NF-based electrodes exhibit outstanding cycling stability of 93% capacitance retention after 1000 cycles, and also the specific capacitance was up to 216 F/g at A/g. The result points to the great potential of a coaxial cable such as Mn_2O_3 NF material as an electrode material in a supercapacitor (shown in Table 1). Specifically, the composite NF which is the combination of MoS_2 and CNFs material has been produced using CVD techniques with well-controlled hybrid nanostructure with high electrical conductivity, improved cycling stability, and excellent rate performance. This material could be used as a significant candidate for SIBs with great capacity and stable cycling performance. Hydrothermal treatment in electrospinning technique is a new method to synthesis NFs. This technique was used to synthesize graphene oxide-coated Co_3O_4 NFs which behave as a capacitor. Normally, Fe_3O_4 with graphene oxide coating had huge higher ICE and reversible capacity than the pristine Fe_3O_4 nanowires.

Batteries

Energy storage devices have become more important in recent years. Therefore, researchers are trying to develop many devices and applying new technology to create unique materials such as NFs, nanocomposites, ionic liquids, and Li-ion-based novel materials. NFs are one of the advanced developing materials for several applications, particularly in improvement of energy storage field. The porous NFs of Co_3O_4/GO anode show the high reversible capacity, excellent cyclic stability, and rate capability in both half cells and full cells with a commercial $LiMn_2O_4$ cathode. The greater electrochemical performance of the porous Co_3O_4/GO NF can be qualified to the exclusive hybrid structure of anode, which could be helped to avoid the aggregation of Co_3O_4 for improved structure stability of electrode and SEI layer. However, rapid transport of Li^+ ions and electrons between the electrodes is necessary for fast discharging and charging of LIBs. The performance of lithium-ion battery depends on the temperature of annealing process because the lithium-ion battery equipped by the $NiWO_4$ NF annealed at 670 °C as anode material exhibits an outstanding lithium storage performance with great initial coulombic efficiency,

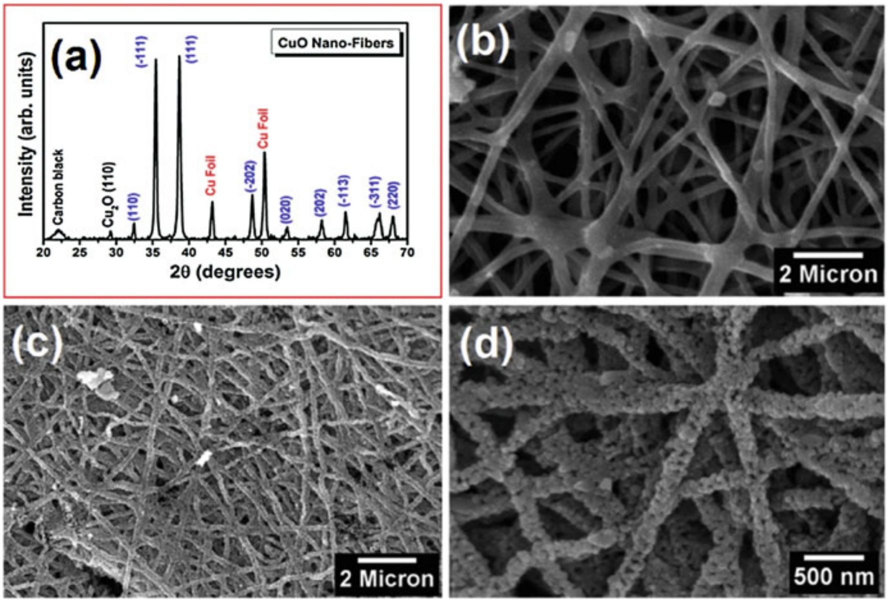

Fig. 3 Electrospun CuO nanofibers as lithium ion battery anode: (a) XRD of the CuO nanofibers, (b) SEM image of the as-spun nanofiber, and (c and d) SEM images of annealed nanofibers at two different magnifications [16]

huge specific capacity, excellent cycle performance, and overall rate performance, while an increase in the annealing temperature to 700 °C for $NiWO_4$ NF provides the moderate performance only. Moreover, this information reveals that $NiWO_4$ NF was an excellent candidate anode material for lithium-ion battery. The fabrication of electrospun CuO NFs has been tested as an anode material in lithium-ion battery. Moreover, these fabricated CuO NFs well performed in half cells in maintaining a specific capacity of 310 mAh g^{-1} at 1 C rate for 100 cycles and stabilized capacity around 120 mAh g^{-1} at 5 C rate for 1000 cycles (Fig. 3) [16].

Catalysis

Advanced development for creating and fabricating nanostructured fibrous catalyst/photocatalyst with significant characteristics such as great reactivity, easy recovery, and economic is important for efficiency of energy and remediation of environments. A simple fabrication electrospinning technique was used to fabricate new hybrid NF of CuO/TiO_2 with various CuO substances, followed by calcination process in air. Furthermore, these hybrid CuO/TiO_2 NFs exhibit better photocatalytic activity (see Table 1) in both simulated sunlight and visible light via degradation of organic dyes compared to normal pure TiO_2. The reduction of organic dye in terms of nitrophenol and these NFs display a significant catalytic activity with sodium borohydride, and this catalytic activity was enhanced by the further addition of CuO to NFs. The anatase-rutile TiO_2 NFs which were calcined for 4 h at 800 °C have been shown to have the highest decomposition rate to prove that it is better than the commercial Degussa P25, which is an excellent photocatalyst. Furthermore, the photocatalytic hydrogenation performance was enhanced by the addition of palladium-based nanoparticles on the anatase-rutile TiO_2 NFs surfaces. A new 3D nanostructured material of alumina copper oxide NFs has been prepared by electrospinning with two-nozzle techniques using polyacrylonitrile (PAN) with boehmite nanoparticles and metal salts. Further, these synthesized 3D NFs were tested as a catalyst in the organic reaction of Suzuki coupling as well as oxidation of hydrazine hydrate. The developing research work represents the fabrication of composite NFs with various mass ratios of Cu/Ni salts.

Wastewater Treatment

The application of metal/metal oxide NFs in the treatment of wastewater has been developed in recent times and is well established. The efficient adsorbent for the removal of Congo red dye by the exploitation of electrospun CuO-ZnO (CZ) composite NFs and the same composite acts as an antibacterial agent. The diazo anionic dye of Congo red is used in the textile industries due to its bright shade. Furthermore, the investigation of the maximum adsorption capacity at the rate of the adsorption through adsorption isotherm and kinetics was well performed. Therefore, the composite NFs depicted the best efficacy compared to their single counterparts. In recent years, there is a basic demand to remove this type of toxic material from water to save nature. In addition, the abovementioned composite NFs of CuO/ZnO were further applied for inspection of antibacterial activity against antibiotic-resistant GFP-E and *S. aureus*. The optimization of TiO_2 NFs through the changes in diameter and crystal phases was reported previously. Besides, the incorporation of Ag as a co-catalyst in TiO_2 could further optimize the photocatalytic reactivity. In environmental application, titania is observed as a promising material, due to its exceptional properties of remarkable photostability, high photocatalyst, low toxicity, and low cost. The titania NF-based membrane was designed and fabricated by fluorination and filtration using vacuum modification. An interconnection of the pore structure is fabricated by entwined NFs to endow the equipped membrane with more than 80% of porosity, compared with ceramic particle aggregated membrane. The membrane which was produced using NFs showed an outstanding performance of desalination with flux of 12 LNH and rejection of salt up to 99.92%. Moreover, an excellent stability was used to prepare membrane for long-term MD operation in pure water and even desalinating high saline water. Fluorinated modification of super hydrophobic titania nanofibrous ceramic membrane holds promise for essential applications due to their superb performance for desalination of water.

An efficient removal of heavy toxic metals from wastewater via integrated nanotechnology treatment systems using NFs of electrospun Fe_2O_3 is a remarkable solution. Therefore, chromate (CrO^{4-})-containing aqueous solution was analyzed for their adsorption capabilities by the newly fabricated Fe_2O_3 NF that was found to be better than that of Fe_2O_3 nanoparticles which are commercially available. Additionally, the removal of methyl orange dye and their kinetic studies were examined by the adsorption performance of NFs. The investigation of hazardous Rhodamine B dye photodegradation through the photocatalytic activity of the NiO/γ-Al_2O_3 NFs was shown to be superior to those normal TiO_2 nanoparticles. Composite NFs work better than TiO_2 nanoparticles due to the fast degradation and recovery as well as recycle process was easy to compare TiO_2. The metal composite NFs are an efficient material for removal of dyes as confirmed by investigation with methylene blue dye: it was degraded from wastewater using an effective catalyst of PdO/WO_3 NFs in UV or visible light illumination, and these data gave better results compared to their single counterpart substrates of WO_3 NFs without PdO nanoparticles. Moreover, the toxic harmful organic pollutants such as azo dyes, acetaldehyde, and other classes of inorganic salts also could be degraded using these types of nanocomposite NFs.

Photovoltaic Devices

In recent times energy storage has become a very important need for electronic devices which are used in day-to-day life. Researchers are focusing on reducing energy demand by decreasing the nonrenewable resources. Therefore, devices like photovoltaic (PV) can help to resolve energy problems. High potential performance has been attained from the first- and second-generation solar cells and dye-sensitized solar cells (DSCs), due to modest design, flexibility, huge feedstock, and superior performance to competitors for indoor uses [58]. One of the main reasons for their improvement is that the doping of SnO_2 enhances the electron transfer from LUMO state of the dye to output circuit via the conduction band of this nanocomposite. Another reason is the electron transport of the presenting GO could decrease the resistance of the architect photoanode. Moreover, the enhancement of nanostructure can be contributed, for example, SnO_2/TiO_2 NF photoanode displays an excellent PV performance.

Challenges in NFs

The metal/metal oxide NF production cost is quite high compared to normal fibers which are produced by conventional methods due to the low rate of production and advanced equipped technology which is expensive. Furthermore, the vapors emitted during formation of nanofibers by the electrospinning method have to be recovered and/or used other purposes which render them harmless. Therefore, equipment and cost could be added additionally to this process. Production of NFs may lead to health hazards due to the fineness of fibers and possible toxicity of vapors. This may affect the health if they are inhaled. Moreover, challenges such as economic, health hazards, vapor of solvent, packaging, shipping, and handling are evident. Due to its outstanding qualities, there is a continuing effort to strike an equilibrium between the advantages and the cost.

Current Scenario and Future Work for NFs

The change in the method of production of NFs from laboratory scale to industrial scale is possible through a promising technology of an electrospinning method. In recent years, numerous articles reported on the fabrication, characterization, and applications of NFs. Generally, the nanomaterials have been applied in many fields, due to their high surface area, which is more important for some industries. In particular, biomedical field is especially interested in electrospun NFs due to its adhesiveness, sterile nature, and biocompatibility. The field of applications for NFs is extensive and includes energy storage devices, wound dressing materials, catalyst for reduction, oxidation and coupling reactions, filters, membranes, scaffold materials, protective clothing, drug delivery systems, and sensors. Furthermore, the influence of NFs in medical applications has been developed because few companies were producing commercial NFs for that purpose. An enhancement of energy storage capacity in batteries and fuel cells using NFs as a new material is possible. A vast majority of applications such as sensors, biomedical, and field of photocatalyst have been reported. However, more attention should be expended on pharmaceuticals, fine chemicals, energy storage devices, and catalyst for the synthesis of advanced organic compounds. Currently, specialized reaction conditions have to be applied for multistep preparation methods for pharmaceuticals. In view of this, it is suggested that electrospun NFs could be used as catalyst to synthesize these much-needed organic compounds by simpler routes.

Conclusion

This book chapter described relevant information about the basic aspects of metal/metal oxide NFs such as fabrication, characterization, and applications in various fields. The wide applications of NFs such as biomedical, catalyst, filtration membranes, sensors, defense, environmental remediation, and material chemistry were explained. From a chemist's point of view, NFs are promising candidates for energy storage purposes like solar cells, fuel cells, and batteries for harvesting energy and overall storage drives. Metal NFs have been functionalized with various materials such as polymers like PAN, organic functional compounds such as carboxylate anion, metal nanoparticles, carbon nanotubes, and carbonized materials. These functionalized NFs produce excellent results via better catalytic systems to photodegradation of toxic dyes and organic compounds. Additionally, they can act as heterogeneous catalysts for reduction, coupling, and oxidation reactions. Although there are several reports related to electrospun NFs, there is still much scope for research and new applications of NFs in the fields such as formulations, agriculture, packages of food, etc. Herein large-scale production of NFs is necessary which can be achieved by the electrospinning process. Hence, the electrospinning techniques are growing tremendously, and it is envisaged that within the next 10 years it will reach greater prominence. In essence the production of novel NFs by the electrospinning methodology has great potential for the development of new technology for applications in industries and laboratories.

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