

TiO₂ Nanofibers for Biomedical Applications: From Fabrication to Bacterial and Fungal Selectivity :A Review

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Abstract

The titanium dioxide (TiO₂) nanofibers have garnered much interest particularly in the biomedical sector whereby its characteristics, i.e. high surface volume ratio, high photocatalytic activity and high biocompatibility have made it very popular. To expand the magnitude of production of such nanofibers, various and diverse synthesis techniques such as electrospinning, sol-gel processing and template assisted techniques, have been used. In particular, the methods also enable optimization of the morphology and the crystal structure of the final materials by the research. The TiO₂ nanofibers have been used in these fields, leading to their application in tissue engineering scaffolds up to antimicrobial coating. They are activity selective in relation to bacterial and fungal pathogens due to their efficacy. The review will comment on the history of manufacturing of TiO₂ nanofibers and the uses that the developments have been put in biomedical applications. This review also features their antimicrobial effect and selectivity towards different kinds of microorganisms is also discussed in relation to next-generation TiO₂ nanofibers when applied to the biomedical materials.

Introduction

Iraqi journal Due to the exceptional physicochemical properties, titanium dioxide (TiO₂) is regarded as a highly versatile and promising functional materials in the current scientific field. These features are found to be high chemical stability, tuned bandgap, good photocatalytic activity, biocompatibility and cheapness [13]. It was Fujishima and Honda who first used TiO₂ photocatalysis in 1972 and since that time, many studies have been carried out on the regulation of the TiO₂ characteristics to apply in the energy, catalysis, and environmental cleanup fields [1,2]. The focus has been made in their potential in biomedical fields in the past. In this connection, there was a potential possibility of nanostructures that provided larger surface area and reactivity [3,4]. Electrospun nanofibers have attracted much

attention in biomedical research because of the structural properties of the natural extracellular matrix (ECM) which are very similar. Their high porosity and large surface area to volume ratio allows for good cell adhesion and proliferation and nutrient exchange. In addition, these nanofibrous structures can be designed in order to have therapeutic agents or bioactive molecule carried on them which increases their potential applications in the field of drug delivery, tissue engineering and regenerative medicine [5-7]. As far as its use in the medical field is concerned, Electrospinning is said to be a reliable and scalable technology which can be used to create continuous one-dimensional nanofibers of a wide range of polymeric and inorganic materials. This approach gives a high degree of control over fiber diameter, morphology and composition which also makes it suitable for a number of advanced biomedical applications [5, 6].

The dopants, fibers and therapeutic agents can be included into the fibers using electrospinning and the fibers can be accurate in terms of the diameter of the fibers, surface topography and crystallinity in the case of TiO₂ [7, 8]. The tuning effect of electrospun TiO₂ nanofibers has turned it into a proper antibacterial film, wound dressing, implant coating and a versatile biomedical scaffold material [910111]. Even though intrinsic photocatalytic activity of titanium dioxide allows one to produce reactive oxygen species (ROS) upon irradiation of the material with UV or visible light which contributes significantly to the antimicrobial effect, the interwoven porous structures of the nanofibers can allow the exchange of nutrients and cell adhesion [1, 3, 4].

ROS, such as (OH), (O₂⁻), and singlet oxygen, which destroys bacterial membranes, proteins, and DNA, is the principle cause of the antibacterial effect induced by TiO₂ nanostructures including nanofibers [2, 3]. This antimicrobial mechanism is mostly associated with oxidative stress that affects the integrity of the microbial cell-wall, alterations of metabolic processes inside the cell, and eventual death. Studies have indicated that electrospun TiO₂ nanofibers have very impressive antibacterial activity that is effective against the growth of the gram-positive and negative bacteria. Contact surface area, crystallinity of the fiber, and activity of metal nanoparticles added to these fibers (e.g. silver, zinc and copper) influence these fibers [9-11]. To exemplify this, Ansari et al. have claimed a case where the TiO₂ nanofibers electrospun exhibited a strong antibacterial effect against the *Pseudomonas aeruginosa* and *Staphylococcus aureus* on UV light-stimulated [9]. The analogous study showed that composite nanofibers containing silver nanoparticles had excellent antimicrobial properties, and this fact is tallyed by the fact that they are synergistically acting [11]. Cumulatively, these results illustrate the need to develop TiO₂ nanofibers to enhance the scope of the antibacterial effect without affecting the cytocompatibility of the fibers with the host tissues.

Recent reports have shown that TiO₂ nano-fibers have antifungal properties besides their antibacterial properties, a fact of special interest since there has been an increase in the occurrence of fungal related infection besides the ineffectiveness of traditional antifungal compounds [12-14]. *Candida albicans* and *Candida glabrata* among other fungi of clinical relevance have been noted to exhibit sensitiveness to TiO₂ nanofibers and their composite [12,13]. It is believed that antifungal action is caused by the same mechanisms as those involved in antibacterial activity, such as oxidative stress caused by ROS, biofilm formation inhibition, and membrane integrity disruption [14,15]. As an example, TiO₂ nanoparticles decreased *Candida albicans* cells viability [13] as shown by Ahmed et al, but carbon-TiO₂ hybrid nanofibers inhibited *Candida* biofilm development [12] as demonstrated by Firmino et al. There are numerous applications of TiO₂ nanofibers in medical use compared to antimicrobial therapy. Electrospun TiO₂ nanofiber membranes are applied in the wound healing to provide a protective environment to the microbes as well as increase tissue regeneration [16-18]. The exuding of exudates, taking up oxygen, and exchange of oxygen is possible as they are highly porous and promote fibroblasts due to the chemistry of its surface. As one case, Rezk et al. developed bilayered TiO₂ nanofiber dressings that significantly enhanced the wound healing in animal models [16]. Recently, Ibrahim et al. found out that TiO₂ loaded membranes could enhance the wound healing process and reduce infection *in vivo* [17]. Similar results are also supported by a bigger literature base, indicating that TiO₂ nanoparticles and nanofibers could be effective wound-healing agents [18].

The other biomedical use of TiO₂ nanofibers that is very crucial is in altering the surface of the implants. Titanium and titanium alloys have also been widely used as orthopedic and dental implants because it has good mechanical and biocompatible characteristics. However, the issue of bacterial colonization and biofilms formation on the implant surfaces remains as a major clinical problem [19-21]. It has been found that surface finishes of TiO₂ nanostructures like nanofibers can help facilitate the process of osseointegration as well as provide them with an antibacterial property [19,20]. In their case, Lee et al. discovered that *in vivo* TiO₂ nanotube arrays on titanium dental implants enhanced its rate of osseointegration [19]. Similarly, TiO₂ multi-purpose coating was shown to reduce bacterial adhesion and increase its absorption in the host tissue [20,21].

Induction of tissue bonding and inhibition of infection are these two activities that are important in the long-term performance of the biomedical implants. One key barrier to the development of TiO₂ nanofibers to biomedical uses is that, due to their inherent bandgap (≈ 3.2 eV (anatase)) photocatalysis can be effectively active only at UV light wavelengths; this is due to the fact that the photocatalytic properties of nanofibers are poor at visible light wavelengths [1,2,22]. To overcome this, several

methods have been taken up to render the activity visible-light-active, including doping of the anatase with nitrogen, sulfur, or transition metals and the design of anatase/rutile mixed-phase structures [22-25]. The incorporation of nitrogen reduces the TiO₂ bandgap by creating mid-gap states, facilitating the absorption of visible light and increasing sustained photocatalytic reactions [22], with anatase/rutile systems having been reported to make up this gap synergistically [24,25]. All these developments are both expanding the range of antimicrobial activity and rendering TiO₂ nanofibers more clinically feasible since visible light is less harmful and more immune to UV than the latter. In short, TiO₂ nanofibers are one of the rapidly increasing research fields. They cross the boundaries of materials science, nanotechnology and biomedical engineering. Their special features of high surface area, tunable photocatalytic activity and structural similarity to ECM have made them very appealing in applications, including antibacterial and antifungal therapy, wound healing and implant coating. Nevertheless, several issues remain to be addressed, including effective activation of the visible light, cytocompatibility, and the ability to translate the results of the in vitro research to clinical practice. The paper is a review of the antibacterial and antifungal selectivity of the titanium dioxide nanofibers with emphasis on recent improvements on their manufacturing, properties, and their use in biomedical practice. This is given importance on strategies of production and the effects of structural features on performance in various settings. Through the analysis of not only experimental outcomes but also application-oriented research, the discussion provides the current issues concerning the problem as well as indicates the future opportunities of the clinical translation of TiO₂ nanofiber-based materials.

Fabrication of TiO₂ Nanofibers

TiO₂ nanofibers have drawn significant attention to the area of biomedicine. This was mainly because they had a high surface area, fine-tuned morphology and their excellent bioactivity. The synthesis process determines the controlling role in the control of the structural, crystallographic and functional properties of the nanofibers. Easy to perform electrospinning as compared to other fabrication techniques has resulted in a high usage of the process since it can produce continuous fibers, which have a diameter ranging between tens and hundred nanometers [26-28]. In the case of electrospinning, titanium precursor (e.g. titanium isopropoxide, TTIP or titanium butoxide) is dissolved in a solution with a polymer (e.g. polyvinylpyrrolidone, PVP). This mixture is followed by solgel reactions and then a high voltage is used such that a small jet is sprayed which is concretized into nanofibers at a grounded collector. The fibers are then heated to 400-800 °C to oxidize the polymer and bring about the crystallization into anatase or rutile as per the conditions of the heat-treatment [29, 30]. As shown in

Figure 1, the TiO₂ nanofibers produced by electrospinning have a regular organization following the calcinations performed on the products.

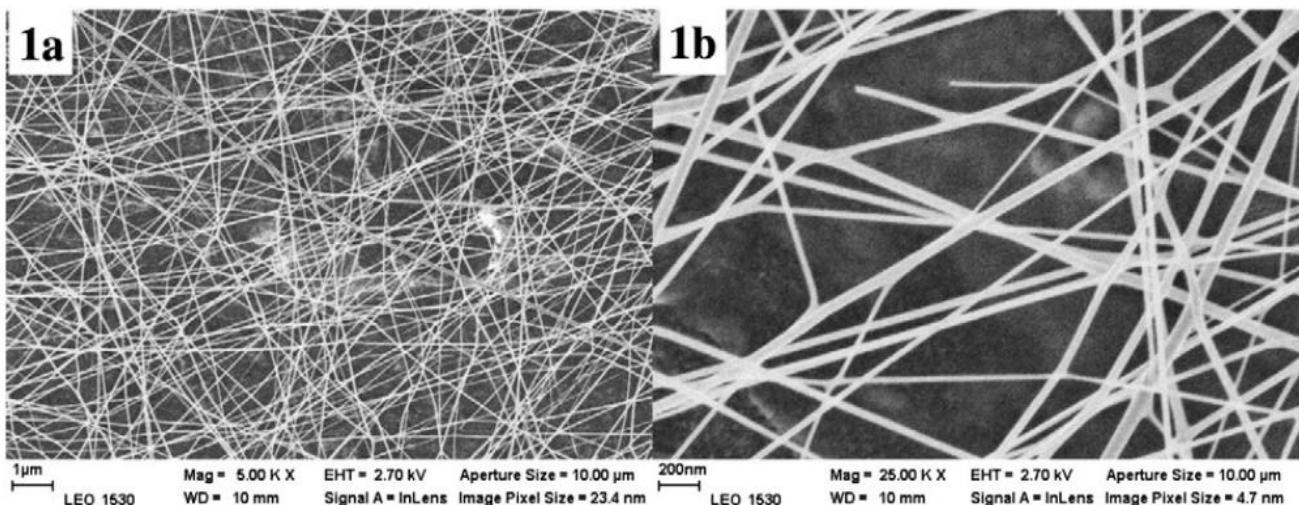


Figure 1: SEM image of electrospun TiO₂ nanofibers after calcination at 500 °C showing uniform morphology.

Besides the electrospinning, Hydrothermal synthesis has also been employed to prepare TiO₂ nanofibers which were described in terms of desirable crystallinity and aspect ratio at relatively mild conditions. The method is quite advantageous in biomedical applications in particular because it provides extremely high surface reactions of nanofibers, which enhances osteointegration and antibacterial activity [31-33]. A figure of morphology and development of crystal growth is presented in figure 2. Hydrothermal treatment produces SEM images of TiO₂ nanofibers which are conformed fibrillar morphology.

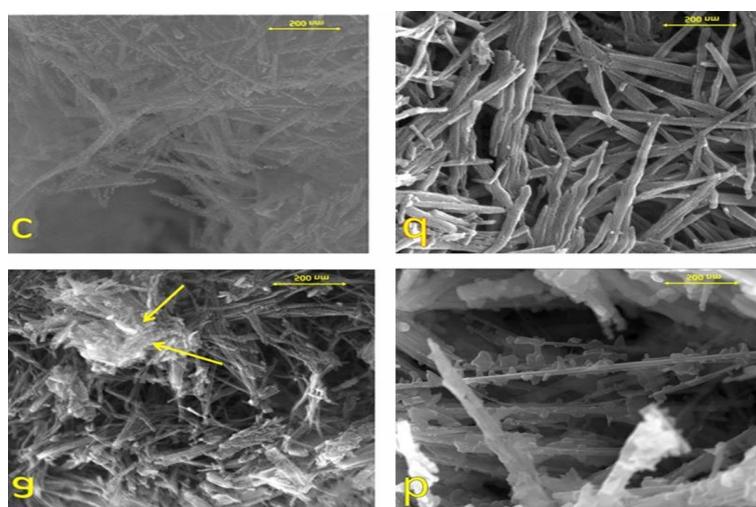


Figure 2: SEM images of TiO₂ nanofibers synthesized through hydrothermal treatment: (a) sample a3, (b) sample a4, (c) sample b3, (d) sample b4 [Hassan et al., 2024].

A second sol-gel template-assisted sol-gel is a second generic process. Titanium alkoxides are introduced under these technologies and processed by condensing to create TiO_2 networks via hydrolysis. Hollow or porous TiO_2 nanofibers can also be obtained through calcification with the help of sacrificial polymer templates [34]. These rankings are applicable in these hierarchies in drug loading and controlled release. The fabrication process of solgel/template is schematically represented in figure3.

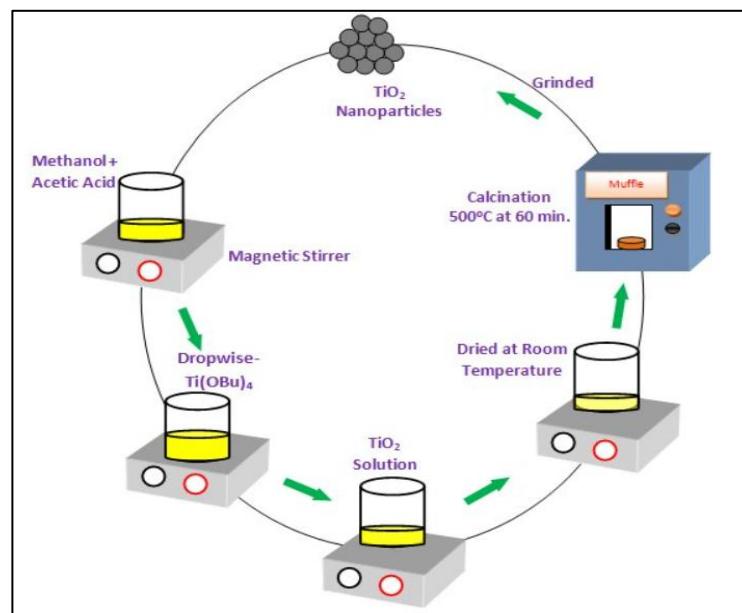


Figure 3: Schematic illustration of the sol-gel and template-assisted fabrication route for TiO_2 nanofibers.

The precursor concentration, applied voltage, the solvent composition and calcination atmosphere are the processing parameters that have a severe effect on the fiber diameter, crystallinity and porosity. Indicatively, higher concentration of the precursor results to thicker fibers whereas higher temperatures of the calcinations results to higher crystallinity but with the possibility of acquiring grain growth and

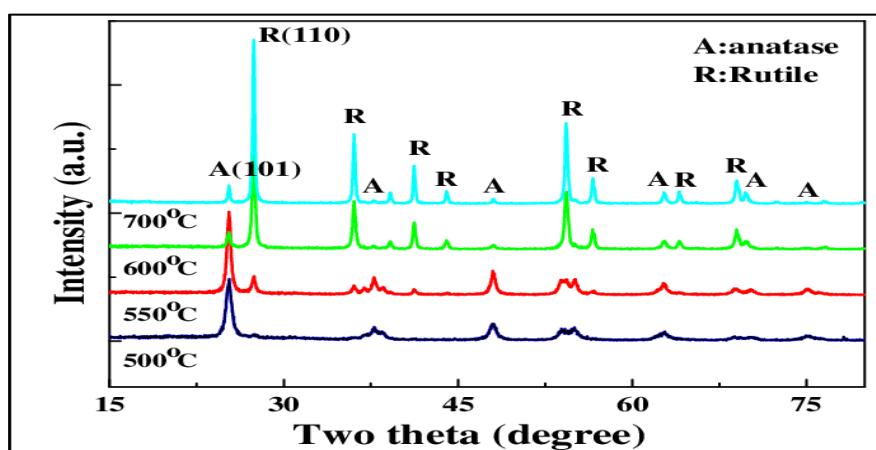


Figure 4:XRD pattern showing amorphous-to-anatase phase transformation after calcination

decreasing the surface area [35,36]. The XRD pattern of the transition between amorphous and anatase TiO_2 after calcinations is depicted in Figure 4.

Furthermore, it is mentioned that the doping of the TiO_2 nanofibers with bactericidal metals (e.g. Ag, Cu, Zn) during the nanofibers preparation could be useful in order to enhance bactericidal activity of nanofibers, which can produce reactive oxygen species (ROS) [37-39]. The dopant salts are typically combined with the electrospinning solution before the calcination of the two to produce metal doping. The metal doping of TiO_2 nanofibers is able to change the surface morphology as well as change the distribution of the elements in the fibers. The Homogenous distribution of dopant elements is important in the achievement of increased antibacterial and photocatalytic activity. Dopants adjust surface charge status and give rise to oxygen vacancies which stimulate the transfer of electrons and increase the formation of ROS on TiO_2 surfaces. As seen in the Figure 5 the SEM micrograph and the associated EDX elemental maps clearly show the homogenous distribution of Ti, O and dopant species within the nanofiber matrix, and the success in doping and maintaining the integrity of the fibers.

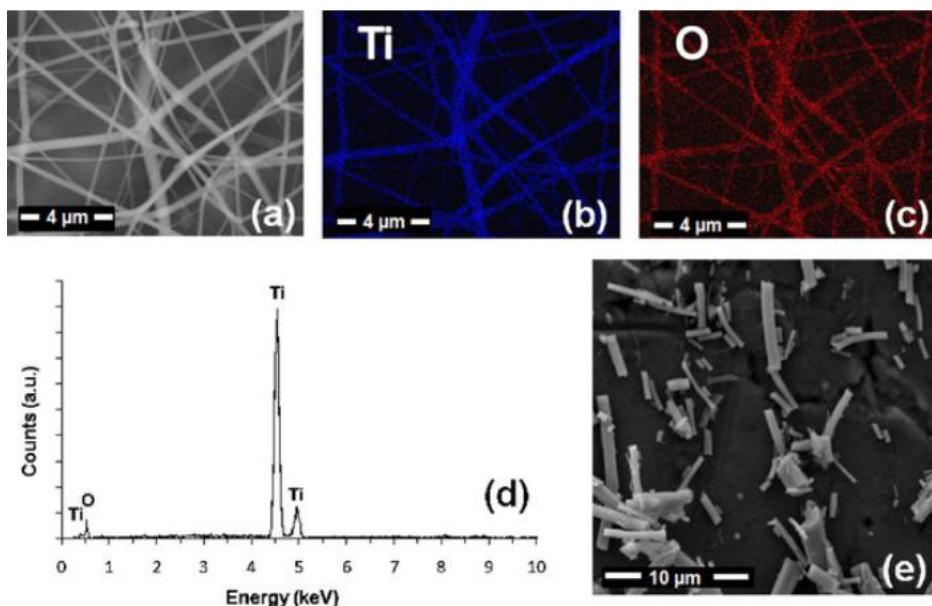


Figure 5: SEM image and EDX elemental maps of electrospun TiO_2 nanofibers, indicating the uniform distribution of dopant elements (Ti, O and additional dopants). Based on Deniz et al., 2011.

Finally, scalable production strategies such as multi-jet electrospinning and centrifugal spinning are being developed to meet industrial demand for biomedical applications [40].

Bacterial & Fungal Selectivity applications

Titanium dioxide (TiO_2) nanofibers have received interest due to their high photocatalytic and antimicrobial activities. Their high surface area and anatase crystalline phase promotes the production

of ROS when they are under UV or visible light. These species such as superoxide anions (O_2^-), (OH) can destroy the cell wall of microbial cells, interfere with membrane activity, and cause oxidative stress, which eventually causes cell death [41 -43]. TiO_2 Nanofibers have been studied on a broad spectrum of pathogens including Gram-positive and Gram-negative bacteria and opportunistic fungi. Gram-negative such as *Escherichia coli* and *Pseudomonas aeruginosa* contain an additional lipopolysaccharide membrane that can affect their susceptibility whereas Gram-positive such as *Staphylococcus aureus* contain thicker layers of peptidoglycan. Due to their capacity to generate biofilms and their resistance to widely used antifungal drugs, the pathogenic fungi such as *Candida albicans* also pose a severe clinical challenge [44, 45]. In the experiment, TiO_2 nanofiber mats were placed on the culture plates inoculated with the test strains. In order to ascertain the selectivity of the antimicrobials, inhibition zone diameters were measured on a post-incubation basis of one day. The results of the **figure 6** halo showed that a distinctive inhibition halo surrounded the nanofiber samples in respect to all the microorganisms used. It substantiates the idea that TiO_2 nanofibers possess the broad spectrum antimicrobial effect and suggests the potential application in the form of medical implants, wound covers, and sterilization surfaces coatings. The latter is further justified by direct contact mechanisms of killing and not just by the ROS generation which destabilizes the microbial membranes with or without light activation being performed [46,47].

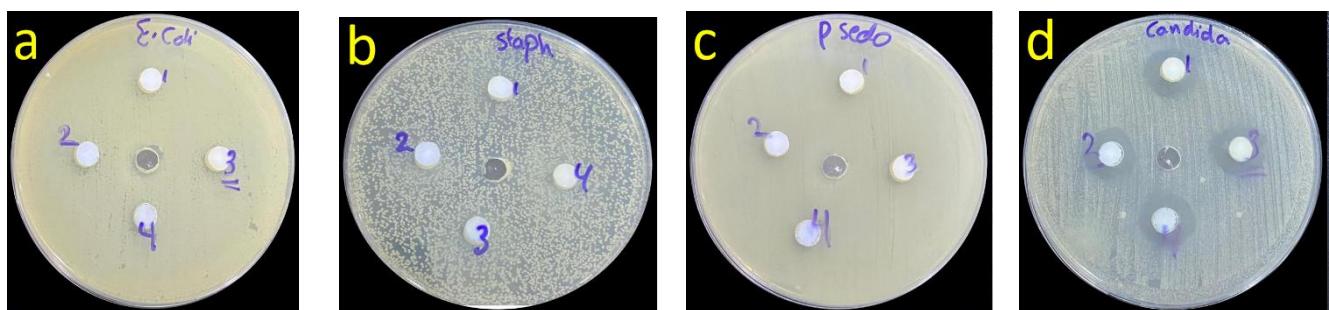


Figure 6: Overnight culture of (a) *E. coli*, (b) *Staphylococcus aureus*, (c) *Pseudomonas aeruginosa*, and (d) *Candida albicans* in the presence of TiO_2 nanofibers (adapted from reference).

Other Applications of TiO_2 Nanofibers

TiO_2 nanofibers have unique structural and physicochemical properties for instance: high surface-to-volume ratio, tunable band gap, and excellent biocompatibility:

1. Photocatalysis and Environmental Remediation

TiO_2 nanofibers have a wide range of application as photocatalysts in the degradation of organic contaminants as well as the removal of dyes in wastewater. This is because they have a high surface

area and crystallinity, which improved the light absorption and production of reactive oxygen species, resulting in the high photocatalytic efficiency relative to bulk TiO_2 . Based on the studies, up to 90% the methylene blue and phenol derivatives were degraded under the influence of UV light or visible light through TiO_2 nanofibers mats. The materials could also be used in self-cleaning surfaces and air purification systems because they can dissolve VOCs (volatile organic compounds) [48-50].

2. Energy Conversion and Storage (Solar Cells & Batteries)

TiO_2 nanofibers are electron transport layers used in dye-sensitized solar cells (DSSCs) to enhance light collection and reduce recombination loss. Nanofiber-based electrodes offer greater direct channels through which electrons can be moved, compared to traditional nanoparticle films, and this allows the device to be more efficient. They have also proven to be useful in lithium-ion batteries as anode material because of its porous one-dimensional structure, which provides them with high capacity and good cycling behavior [51-53]. More recently, they have been used in studies of perovskite solar cells in an effort to increase further the photovoltaic efficiency.

3. Biomedical Applications (Antibacterial and Implants)

The ability of TiO_2 nanofibers to react with surfaces and their high biocompatibility has seen their use in antibacterial wound dressing and medical implantation applications. TiO_2 provides an alternative approach to antibacterial action because, at UV light wavelengths, it produces reactive oxygen species (ROS), which destroy bacterial membranes and cause cell damage instead of using antibiotics. Ag or Zn nanoparticles are also added to enhance their antibacterial action against *Staphylococcus aureus* and *E. coli*. TiO_2 nanofibers have also been used as implants which have been shown to improve osseointegration and reduction in post-surgical infection [54-56].

4. Gas Detection and – Environment.

TiO_2 nanofibers are highly sensitive to gases, such as H_2 , NO_2 and ethanol and therefore, make good materials to be exploited as a gas sensor. They will be porous and thus will absorb and release the gas molecules at a very high rate basically resulting to their response and recovery time will be very low. The selectivity is also increased with metal doping (e.g. Pd, Pt) and surface functionalization to limit detection. Control over the environment and safety in the industry The sensors may be enumerated as part of the issues that are often researched [57-59].

5. Hydrogen Production by photocatalytic processes.

In the creation of hydrogen, TiO_2 nanofiber can be needed in the photocatalytic splitting of water. The TiO_2 nanofibres under sunshine have also been shown to have increased quantum efficacy, through

maximisation of anatase to rutile ratio, and co catalysts (e.g. Pt) incorporation. This makes them the possible clean energy generating materials as far as the issue of sustainability is involved [60-61]. However, the potential problems such as the cytotoxicity of the human cells and the structural degradation of the TiO_2 nanofibers during the continuous irradiation should be put into consideration.

Conclusion and Future Perspectives

Titanium dioxide nanofibers (TiO_2 NFs) have become one of the most versatile nanostructures due to its good photocatalytic activity, biocompatibility and, lastly, physical-chemical properties. The main synthesis methods, which consist of electrospinning method, hydrothermal and sol-gel methods, have been identified in this review, alongside optimization of the sintering conditions to control the crystallinity and morphology of fibers. The TiO_2 NFs are excellent in many applications like photocatalysis, biomedical implants, environmental cleanup machines, and energy transforming machines. These characteristics render them the exceptional candidates at the interdisciplinary researches as well as industrial level technologies. Such developments notwithstanding, there exist some issues that ought to be addressed. An example of a current challenge is, e.g., the attainment of reliable large scale production with morphology and phase composition. There is also a need to further test the stability and the recyclability of TiO_2 nanofiber-based systems in the long run in real situations. It has been demonstrated that photo catalytic and antimicrobial reactions can be improved when TiO_2 NFs are coexcited with other useful nanomaterials, such as doped metals or carbonaceous supports. Further studies should also be conducted on such hybrid systems. It is important to note that TiO_2 nanofibers can be used as bone regeneration scaffolds, as antimicrobial implant coatings, highlighting their biomedical importance. As a way of ensuring clinical safety and effectiveness, however, additional research on cytotoxicity, immune interactions, and degradation behavior will be necessary. In addition to medicine, they are also being studied as hydrogen generators, lithium-ion battery, and high-performance solar cells, though the question of enhancing the electron transport and light-gathering efficiency is a major issue. To conclude, the nanofibers of TiO_2 have a promising future in various fields of technology. In the future, it is necessary to focus on the ways to scale production routes, focused surface changes, and strict performance testing in practice in order to make its practical effects complete.

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