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Review of the recent advances on the fabrication, modification and application of electrospun TiO_2 and ZnO nanofibers for the treatment of organic pollutants in wastewater

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The heightened occurrence of emerging organic pollutants (EOPs) in aquatic bodies has been the subject of global apprehension due to the toxicity they pose to the environment, humans and animals alike. The presence of EOPs has soared due to industrialization and is further exacerbated by human activities like the overuse and poor disposal of dyes, pesticides, pharmaceuticals, surfactants, personal care products and food additives. The complete treatment and removal of EOPs from industrial wastewater and sewage has remained a challenge because of their pseudo-persistence and resistance to degradation. Due to their impressive light absorption properties, high surface-area-to-volume ratio, high porosity, superior mechanical strength, electrospun titanium dioxide (TiO_2) and zinc oxide (ZnO) nanofibers have been proposed for the photocatalytic treatment of EOPs. Therefore, this review first highlights the fabrication and modification methods of TiO_2 and ZnO nanofibers. A systematic survey of the latest progress in the application of TiO_2 and ZnO nanofibers for the degradation of EOPs is then elaborated. Thus, the main goal is to shed light and give insight to researchers on the possibilities surrounding the elimination of EOPs by applying electrospun TiO_2 and ZnO semiconductor materials. In addition, the loopholes associated with fabrication and modification processes are discussed with the aim of encouraging innovation for prospective technology advancement and commercialization, as well as to enhance research efforts in wastewater treatment and environmental sustainability.

KEYWORDS

electrospinning, photocatalysis, TiO_2 nanofibers, ZnO nanofibers, wastewater treatment, organic pollutants

1 Introduction

Increased industrialization and frequent natural calamities have resulted in the shortage of water, which in turn, poses a threat to the development and livelihoods of the affected communities. The occurrence of emerging organic pollutants (EOPs) such as, industrial products/by-products, pesticides, pharmaceuticals, personal care products and food additives in water streams also contributes to the water crisis (Arman et al., 2021; Paumo et al., 2021; Singh et al., 2021). The overuse and poor disposal of EOPs results in them entering drinking water sources through runoff, artificial recharge, or effluents from wastewater treatment plants (WWTPs) (Mukhopadhyay et al., 2022). Generally, these EOPs have no environmental monitoring or emission standards and have negative ecological effects (He et al., 2022). In addition to jeopardizing environmental sustainability, EOPs can compromise human health when ingested through contaminated water (Guillotin and Delcourt, 2022). They possess endocrine disrupting properties and interfere with the hormonal system (Kasonga et al., 2021; Ng et al., 2021). Moreover, they tend to bio-accumulate in the aquatic environment and exert toxic effects on aquatic animals, which can easily be transferred to humans through the food chain (Impellitteri et al., 2023).

Methods including adsorption, membrane filtration, coagulation-flocculation, solvent extraction and photocatalysis have been explored for the treatment of EOPs (Lhomme et al., 2008; Khoo et al., 2022; Mahmood et al., 2022; Ajala et al., 2023; Tahraoui et al., 2023). With the exception of photocatalysis however, most of these treatment technologies are not efficient at low concentrations of EOPs. They are also not completely destructive and merely transfer the contaminants from one phase to another (He et al., 2022). The use of photocatalysis for the treatment of EOPs has therefore gained interest due to the possibility of achieving high/complete mineralization of the target pollutants. In addition, the process generally does not require high temperatures and pressures, and it has relatively low operating costs (Kumar et al., 2014).

In essence, photocatalysis is based on the light-driven *in-situ* generation of oxidant species that are highly reactive towards organic and inorganic contaminants (Ramírez-Malule et al., 2020). Due to their impressive light absorption properties and concurrent ability to promote the formation of reactive oxygen species, titanium dioxide (TiO_2) and zinc oxide (ZnO) are the most studied semiconductor based photocatalysts (Li et al., 2014; Kumari et al., 2023). They also possess attractive properties including prolonged excited-state lifetimes, enhanced charge transport features, good chemical and optical stability, relatively low cost, abundance, corrosion resistance and non-toxicity (Hernández et al., 2015).

Both TiO_2 and ZnO have been synthesized to afford a range of morphologies including nanoflowers, microspheres, nanoribbons, nanoparticles, nanotubes, nanocages and nanofibers (Wang and Lou, 2012; Samanta et al., 2015; Marien et al., 2017; Samadipakchin et al., 2017; Lan et al., 2018; Zhang et al., 2018; Qu et al., 2020). Recently, research interest on nanofibers has soared due to their attractive properties including their high surface-area-to-volume ratio, high porosity, superior mechanical strength, versatile surface functionalization and tunable properties (Prabhu, 2019). Generally, nanofibers can be fabricated using techniques such as sol-gel, hydrothermal, template synthesis, and electrospinning (You et al., 2012; Gupta et al., 2018; Ademola Bode-Aluko et al., 2021; Chen et al., 2021; Vakhrushev and itsova, 2021). The sol-gel and hydrothermal methods suffer from drawbacks including long processing time and high cost of equipment, respectively (Modan and Plaiasu, 2020). The template synthesis method cannot produce nanofibers with a long fiber length because the templates come with preconfigured layouts and designs (Alghoraibi and Alomari, 2018). Electrospinning however has become the most commonly employed nanofiber fabrication technique due to its simplicity, low cost of production and its ability to produce continuous fibers with diameters ranging from tens of nanometers to several micrometers. Electrospinning also allows for easy modification of the nanofibers and is easily reproducible (Ademola Bode-Aluko et al., 2021). Hence, the focus of this review is on electrospun TiO_2 and ZnO nanofibers for the treatment of EOPs in wastewater.

Thus, this article is intended to give insight to researchers on the possibilities surrounding the elimination of EOPs by applying electrospun TiO_2 and ZnO nanofibers. Recent advances on the fabrication and modification of semiconductor-based nanofibers are discussed and a critical analysis of their efficiencies in the photocatalytic treatment of different EOPs is conducted. Lastly, challenges and possible future prospects in their application in wastewater treatment are identified and deliberated.

2 Electrospinning inorganic TiO_2 and ZnO nanofibers

Purely inorganic nanofibers (e.g., the TiO_2 and ZnO nanofibers) are generally made out of nano-size grains or crystals wherein the size of the grains influences their specific surface area and thus the resultant properties. The nanofiber fabrication process using electrospinning technique is depicted in Figure 1 (Na et al., 2021a). Briefly, a solution consisting of a polymer, volatile solvent and metal precursor is prepared. Examples of some of the commonly employed metal precursors for the preparation of the TiO_2 nanofibers include titanium (IV) isopropoxide, titanium tetraisopropoxide, titanium (IV) n-butoxide and tetra-n-butyl titanate (da Silva et al., 2023; Li and Xia, 2003; Secundino-Sánchez et al., 2022a; Lv et al., 2021), whereas the ZnO nanofibers are commonly synthesized from salts including zinc acetate and zinc nitrate (Mali et al., 2013; Di Mauro et al., 2016). The preparation of both the ZnO and TiO_2 nanofibers using electrospinning also requires the use of a sacrificial polymer that is chosen based of its thermal degradation (Zagho and Elzatahry, 2016). Some examples of commonly used sacrificial polymers are polyvinyl pyrrolidone, polyvinyl alcohol and polyacrylonitrile amongst others (Nataraj et al., 2012; Zagho and Elzatahry, 2016). The as-prepared solution is then electrospun wherein upon exposure to high voltage (5–40 kV); nanofibers are formed on a conductive collector (Barhoum et al., 2019). In order to obtain the purely inorganic nanofibers, the obtained inorganic-organic composite nanofibers are subjected to thermal treatment, known as calcination.

During calcination, the temperature and heating rate influence the properties of the resultant nanofibers, including morphology, the phase and chemical composition (Nakonieczny et al., 2021; Vasiljević et al., 2021). This is due to the processes associated with calcination such as removal of residual solvents and water vapour from the nanofibers (Shendokar et al., 2008). In addition, nanofiber shrinkage often takes place as the organic polymer is removed and condensation, as well as structural relaxation proceed

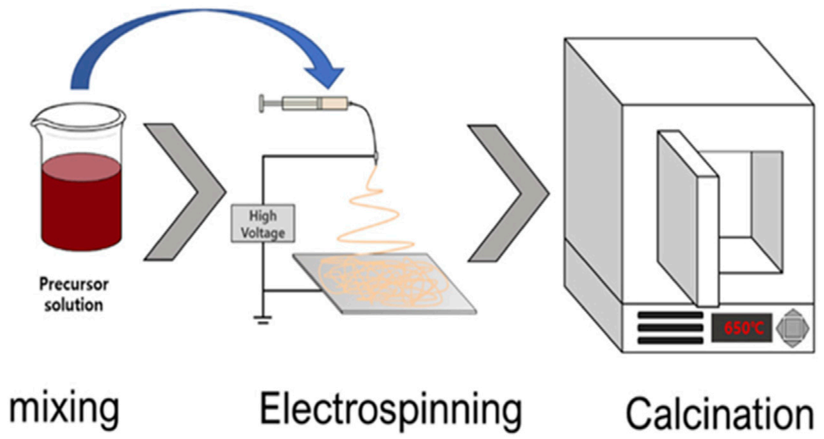

FIGURE 1
Schematic diagram of the inorganic nanofiber fabrication process with electrospinning and calcination. Modified and reprinted with permission (Na et al., 2021a).

(Barhoum et al., 2019). In most cases, no catalytic activity is associated with the polymer, it merely acts as a carrier of the embedded photocatalysts and allows for nanofiber formation. Thus, calcination of the inorganic-organic composite nanofibers not only yields purely crystalline nanofibers, but also removes the polymer and all other organic components thereby resulting in inorganic nanofibers. The resulting inorganic nanofibers exhibit interesting optical and electronic properties as their size approaches the nanoscale. They also possess high crystallinity and flexibility due to their high surface area-to-volume ratio and nanograins (Barhoum et al., 2019; Gugulothu et al., 2019).

Depending on the calcination temperature, TiO_2 nanofibers generally exist in different polymorphs, viz. anatase, mixed anatase–rutile or rutile (Nuansing et al., 2006). Both anatase and rutile exhibit tetragonal crystalline geometry, however the angles between the Ti-O bonds of the former are more distorted from 90°. These configurational differences result in a lower thermal stability and larger band gap for anatase (~3.2 eV) relative to rutile (~3.0 eV) (Paunović et al., 2021). The ZnO nanofibers on the other hand generally exist as a wurtzite phase (~3.3 eV) as it is the most stable phase of ZnO relative to the zinc blende, and rock salt phases (Wróbel and Piechota, 2008; Bolarinwa et al., 2017).

3 Modification strategies

Various strategies have been employed to modify TiO_2 and ZnO nanofibers to fine-tune and enhance their physical properties and promote versatility in their applications. Modifying the surfaces of the nanofibers also enhances their photocatalytic efficiency as it suppresses charge recombination. Additionally, both TiO_2 and ZnO have wide band gaps of ~ 3.0 eV, therefore their photoactivity is mainly under UV light (Etacheri et al., 2015). The sunlight reaching the Earth's surface however only contains about 5%–10% UV light, thereby limiting the real life applications of these nanofibers. To combat that and enhance their optical properties, the nanofibers have been modified using various methods including doping, dye photosensitization and by creating heterogeneous nanofibers as discussed next.

3.1 Doping

Dopants generally work by modulating the active sites of materials, thereby providing a powerful means for creating a large variety of highly efficient catalysts for various reactions (Zhang et al., 2021). Doping with metal cations, non-metal anions or non-metal molecules can strongly enhance the light absorption efficiency of a photocatalyst by influencing its electronic structure (Zhang et al., 2010).

As shown in Figure 2, doping semiconductors (TiO_2 used as an example) with non-metals like nitrogen can reduce the band gap of the former by creating an intermediate band for the electrons below the conduction band or above the valence band (Mahy et al., 2018). Moreover, nitrogen has the capability to change the surface-electronic properties $TiO_{2.}$ The comparable atomic size of nitrogen (155 pm) with oxygen (152 pm) allows nitrogen to substitute the oxygen atoms from the TiO_2 lattice, causing enhanced photocatalytic efficiencies (Natarajan et al., 2021).

Doping is therefore an effective method for fabricating visible light-active photocatalysts, due to the ability of the ions from the dopant to incorporate into the catalyst crystal lattice of the semiconductors and modify its electron properties (Zhang et al., 2010). For instance, transition metals possess several valences and a d-electron structure that is not full and can accept extra electrons, thereby restricting charge recombination in the semiconductors (Estévez Ruiz et al., 2023).

Possible limitations of doping include photocorrosion and promoted charge recombination at the metal sites (Ellappan and Miranda, 2014). Doping can also disrupt the structural integrity and induce distortions on the surface of the nanofibers. Nonetheless, researchers have doped TiO_2 and ZnO nanofibers with metals and non-metals, mainly for an enhanced photo-response and thus catalytic efficiency as discussed next.

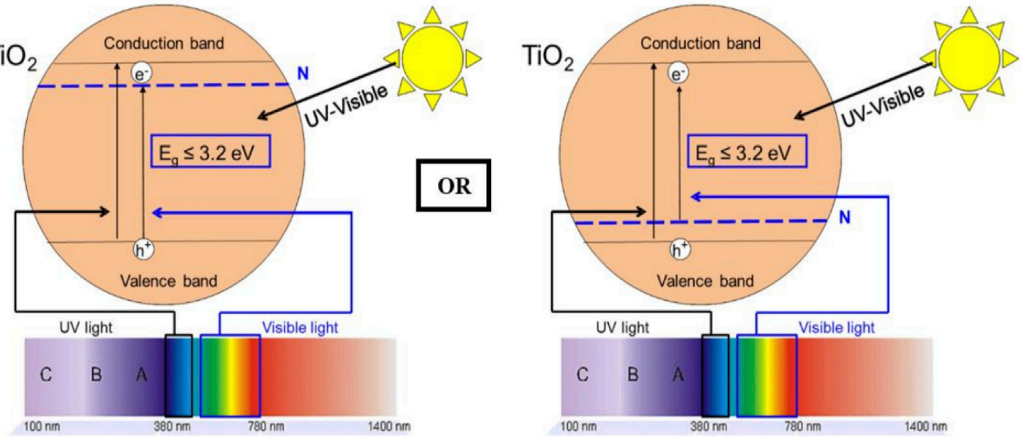

FIGURE 2
Effect of N-doping on TiO_2 photocatalysis. Reprinted with permission (Mahy et al., 2018).

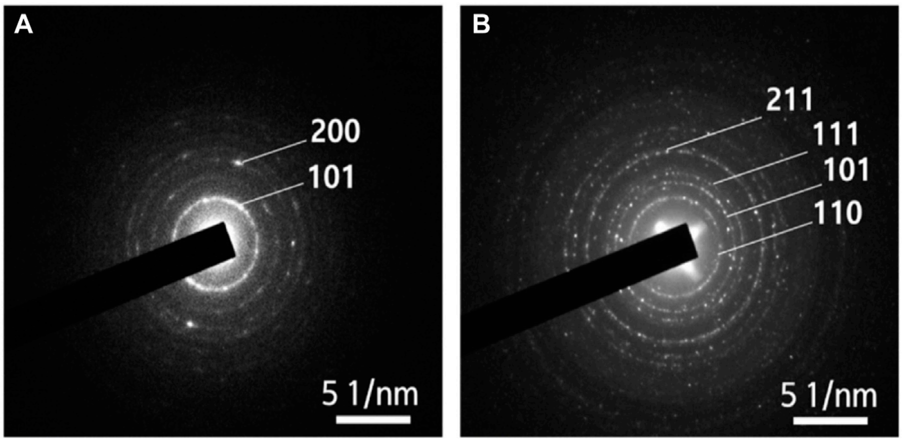

FIGURE 3
Selected area electron diffraction patterns obtained from nanofibers: **(A)** TiO_2 nanofibers and **(B)** Fe-doped TiO_2 nanofibers. Reprinted with permission (Na et al., 2021b).

3.1.1 Doping of TiO_2 and ZnO nanofibers

Ma et al. fabricated S-doped TiO_2 nanofibers, using thiourea and carbon disulphide, as sulphur precursors. Their results revealed that sulphur atoms were successfully incorporated into the bulk phase of the TiO_2 nanofibers. The S-doping could also effectively inhibit the growth of crystalline grain size and increase light absorbance in the visible region (Ma et al., 2014). Similarly, Camillio et al. prepared N-doped TiO_2 nanofibers. Doping enhanced the light absorption properties of the TiO_2 nanofibers, resulting in visible-light active nanofibers (Di Camillo et al., 2012). Roongraung et al. fabricated Ag-doped TiO_2 nanofibers with different Ag contents, resulting in nanofibers with even better properties than the commercial TiO_2 (P25) powder (Roongraung et al., 2020).

Song et al. fabricated W-doped TiO_2 nanofibers. Before doping, the TiO_2 nanofibers composed of anatase and rutile phases. Interestingly, doping the TiO_2 nanofibers with tungsten completely transformed their rutile phase to anatase phase (Song et al., 2016). Similarly, Na et al. prepared Fe-doped TiO_2 nanofibers wherein the crystal phase of the TiO_2 matrix was transitioned from anatase to rutile after doping. This is demonstrated in Figure 3 wherein the selected area electron diffraction results for the nanofibers showed a ring-type diffraction pattern, indicating crystallinity. As shown in Figure 3A, the diffraction pattern of the TiO_2 nanofibers had a [101] plane, attributed to the anatase phase, while the Fe-doped TiO_2 nanofibers (Figure 3B), were indexed to the rutile phase (Na et al., 2021b). It has been reported that phase transformations do occur when TiO_2 nanomaterials are doped, depending on several parameters including the method of doping, dopant type and concentration used, as well as the reaction atmosphere (Ahmad et al., 2007).

Moreover, ZnO nanofibers have also been doped to alter and enhance their properties. Wang et al. fabricated Mn^{2+}-doped and N-decorated ZnO nanofibers. The doped ZnO nanofibers exhibited enhanced photocatalytic properties due to their synergetic

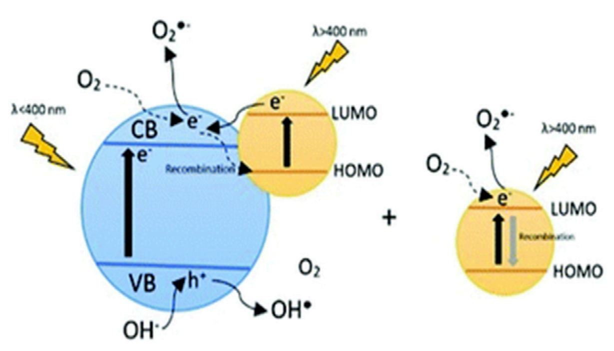

FIGURE 4
Mechanism of the dye sensitization process with the dye adsorbed onto the catalyst surface and dye in the bulk. Reprinted with permission (Diaz-Angulo et al., 2019).

interaction with both Mn^{2+} and N (Wang et al., 2016). Sun et al. (2023) prepared Rh-doped ZnO nanofibers wherein the doping introduced large amounts of surface oxygen vacancy defects and enhanced the optical properties of the nanofibers (Sun et al., 2023).

In addition to enhancing the visible light absorption, some dopants induce magnetic properties to the nanofibers, making their magnetic regeneration post-application easy. For instance, Baranowska-Korczyc et al. fabricated Fe-doped ZnO nanofibers exhibiting ferromagnetism at room temperature. This feature was found to either be mediated by the presence of oxygen vacancies or related to the presence of small precipitates of ferromagnetic phases of iron (Baranowska-Korczyc et al., 2012). Chen et al. fabricated Cu-doped ZnO nanofibers using two methods: adding Cu salt in the sol-gel precursor before electrospinning and thermally diffusing Cu atoms into the pure ZnO nanofibers. They showed that the nanofibers obtained by adding Cu salt in the sol-gel precursor before electrospinning were paramagnetic while those obtained by thermal in-diffusion exhibited ferromagnetism at room temperature (Chen et al., 2020).

3.2 Dye photosensitization

Dye photosensitization entails modifying TiO_2 and ZnO with either natural or synthetic dye extracts, thereby activating their visible light absorption capacity (Goulart et al., 2020). Most of the commonly used photosensitizing dyes reported in the literature include porphyrins and metallophthalocyanines (Duan et al., 2012; Atta-Eyison et al., 2021; Keşir et al., 2021).

As depicted in Figure 4, upon exposure to visible radiation, the excited dye passes an electron from its highest occupied molecular orbital (HOMO) to its lowest unoccupied molecular orbital (LUMO), and subsequently to the conduction band of the semiconductor (Bayrak et al., 2016; Diaz-Angulo et al., 2019). This results in an enhanced photocatalytic efficiency for the semiconductor and enables its activation using visible light. On the other hand, the dyes that are not adsorbed on the semiconductor and are in the solution can also absorb light, allowing for the electrons in the LUMO to react with dissolved oxygen in order to produce superoxide anion radicals ($O_2^{\bullet-}$) (Diaz-Angulo et al., 2019).

In order for dyes to be considered as efficient photosensitizers, they need to have strong absorption of visible light or part of the near infrared (NIR) region (Abrahamse and Hamblin, 2016). They also need to be photostable and have anchoring groups (e.g., SO_3H, –COOH, $–H_2PO_3$, etc.) to facilitate the binding of dye molecules onto the surface of the semiconductor (Diaz-Angulo et al., 2019). Moreover, the excited state of the dye photosensitizers should be higher in energy than the conduction band edge of the semiconductor to enable efficient electron transfer between the two (Yun et al., 2017).

Dye photosensitization does however have limitations including lack of long-term stability as sometimes the dye itself suffers degradation by irradiation, thereby changing its structure and properties (Belver et al., 2019). The synthesis of the dyes can also be an issue as the processes often involve organic reactions with several steps and low yields, making them expensive to generate. Nonetheless, a few researchers have reported on the dye photosensitization of TiO_2 and ZnO nanofibers.

3.2.1 Dye-photosensitized TiO_2 and ZnO nanofibers

Guo et al. modified TiO_2 nanofibers with 2,9,16,23-tetra-nitrophthalocyanine iron(II) (Guo et al., 2012). The resulting nanofibers exhibited an enhanced photo-response relative to the unmodified ones. Mkhondwanae et al. modified TiO_2 nanofibers using a Zn-5-p-carboxyphenyl-10,15,20-(tris-4-pyridyl)-porphyrin (amongst other things) and found that the nanofibers were highly active in visible light driven photocatalysis (Mkhondwane et al., 2022). Hlabangwane et al. modified TiO_2 nanofibers with free base, tin (II) and indium (III) tetramethoxyporphyrins for comparison of photo-response enhancement efficiencies (Hlabangwane et al., 2023). Arifin et al. enhanced the optical properties of their ZnO nanofibers by modifying them using a ruthenium complex dye (Arifin et al., 2020). Lastly, Mapukata et al. modified both TiO_2 and ZnO nanofibers with a zinc phthalocyanine, reporting that the modified nanofibers had a higher photo-response and photocatalytic efficiency than the unmodified ones (Mapukata and Nyokong, 2020).

3.3 Composite nanofibers

Some scientists have explored combining TiO_2 and ZnO together or with other semiconductors to create composite nanofibers. This is because the synergistic interaction between the individual semiconductor components provides enhanced physical and chemical properties (Tan et al., 2020). The interaction between composite semiconductors is shown in Figure 5. Upon photo-excitation, the individual constituents of the composite are excited and charge transfer occurs (Siwińska-Stefańska et al., 2018). This suppresses individual charge recombination and increases the lifetimes of the charge carriers, thereby enhancing the photocatalytic performance of the composite materials (Siwińska-Stefańska et al., 2018; Bai et al., 2021).

Composite nanofibers in particular usually have high strength, corrosion resistance, design flexibility and increased durability relative to their separate constituents (Toriello et al., 2020).

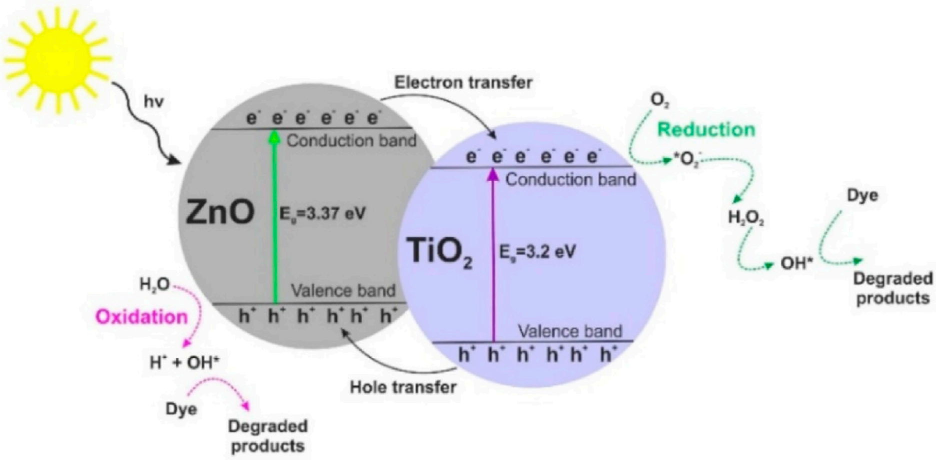

FIGURE 5
Mechanism of photo-activity of TiO_2-ZnO composite materials. Reprinted with permission (Siwińska-Stefańska et al., 2018).

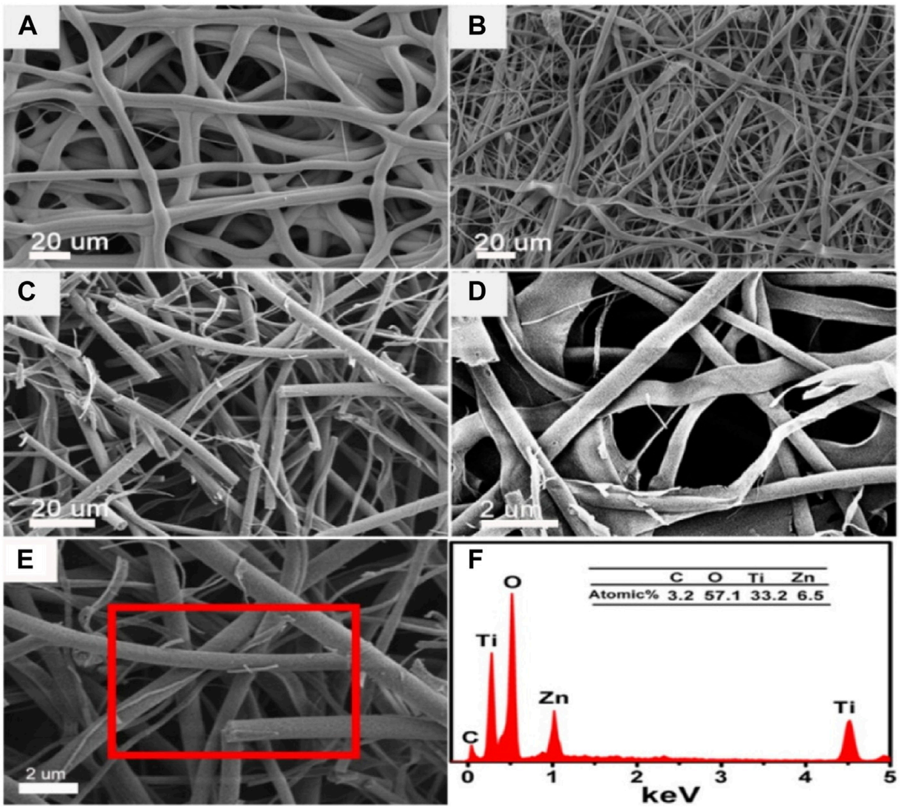

FIGURE 6
SEM pictures of the pure TiO_2 and the ZnO/TiO_2 nanofibers with different content of Zn(OAc)$_2$ after calcination: **(A)** bare TiO_2, **(B)** 0.5 wt%, **(C)** 1 wt%, **(D)** 2 wt%, **(E)** SEM image of ZnO/TiO_2 (1 wt%) nanofibers, **(F)** EDX spectra from the selected region in image E. Reprinted with permission (Chen et al., 2016).

However, the fabrication of composite nanofibers tends to require intricate optimisation as they can have entangled patterns with different cross-sectional structures, often with beads.

3.3.1 TiO_2 and ZnO based composite nanofibers

Someswararao et al. fabricated TiO_2/ZnO composite nanofibers with an improved photocatalytic activity relative to the separate

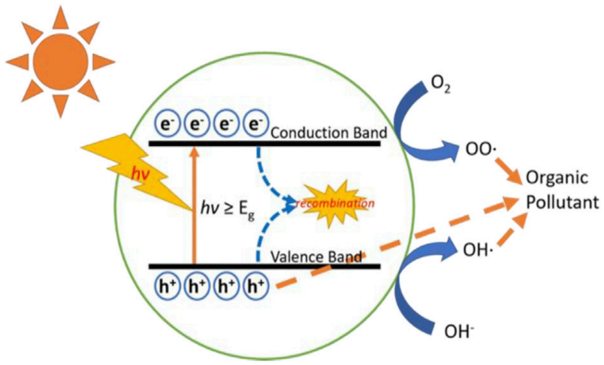

FIGURE 7
Schematic illustration of the photocatalysis process. Reprinted with permission (Gao et al., 2021).

TiO_2 and ZnO nanofibers (Someswararao et al., 2021). Chen et al. also fabricated TiO_2/ZnO composite nanofibers with varying amounts of zinc acetate (0.50%–2.00%) as shown in the scanning electron microscopy (SEM) images in Figures 6A–E. They reported that the nanofibers were smooth, even with an increase in the zinc acetate content and the overall reticular conformation was maintained. The nanofibers with 1 wt% zinc acetate did however possess more fractured surfaces existing as loose ends (Figure 6C). The elemental composition of nanofibers was confirmed as shown in Figure 6F. The fabricated nanofibers were composed of carbon, titanium, zinc and oxygen elements, demonstrating the coexistence of ZnO and TiO_2 in the samples (Chen et al., 2016).

Moreover, numerous researchers have also reported on various composite nanofibers of TiO_2 with other semiconductors like SnO_2 (Shi et al., 2014; Han et al., 2020; Wang and Cheng, 2021). The fabrication of composite nanofibers with ZnO and other semiconductors has also been extensively reported (Yan et al., 2015; Zhao et al., 2015; Naderi et al., 2019). They all reported that the heterogeneous nanofibers had enhanced optical and chemical properties than the individual counterparts.

4 Treatment of emerging organic pollutants

After fabrication, modification and characterization, the nanofibers are ready for application, which in this case is photocatalytic treatment of EOPs. As shown in Figure 7, during the photocatalytic treatment process, the photocatalysts (TiO_2 and ZnO nanofibers) absorb light (photon energy higher or equal to the band gap of the photocatalyst), thereby creating holes (h^+) in the valence band and electrons (e^-) in the conduction band (Al-Nuaim et al., 2023; Navidpour et al., 2023). The photo-generated e^-/h^+ pairs initiate the photocatalytic degradation process by mediating the formation of species including $HO^{\bullet}$ and $O_2^{\bullet -}$ from atmospheric oxygen and moisture (Fotiou et al., 2014; Pavel et al., 2023). These species are well known for having the ability to oxidize and break down organic pollutants (Kondrakov et al., 2016).

In order to determine the internal mechanism of a photocatalytic reaction, it is necessary to identify which reactive species plays a key role in the photocatalytic process (Choudhary et al., 2021). The main oxidative species in the photocatalytic process can be detected using quenchers such as isopropyl alcohol and 5,5-dimethyl-1-pyrroline N-oxide (DMPO) are scavengers for •OH, while ammonium oxalate is a scavenger for h^+. Additionally, diphenylisobenzofuran and p-benzoquinone are commonly used as scavengers for $^1O_2$ and $\bullet O_2^-$, respectively (Parrino et al., 2021). Techniques such as electron paramagnetic resonance spectroscopy (EPR) can be used to investigate and quantify the species formed in the presence of scavengers (Schneider et al., 2020). The radicals formed during photocatalysis have a very short lifetime (usually nanoseconds half-lives). As shown in Figure 8, the presence of diamagnetic spin trap reagents (using DMPO as an example) in the reaction medium generates more stable spin adducts that can be qualified and quantified (Al-Madanat et al., 2021). The other spin trap reagents also work in a similar manner to identify the exact reactive species/radicals doing the photocatalytic treatment.

Given its modular nature and variability of precursor chemistry, electrospinning can enable scaled-up production of photocatalytic inorganic micro- and nanofibers at reasonable cost. It therefore represents a promising fabrication method for generating highly functional nanomaterials for a range of applications (Ludwig et al., 2018). Although herein emphasis is made on their photocatalytic efficiencies for the treatment of EOPs, inorganic TiO_2 and ZnO nanofibers have also been successfully applied in sensing, energy storage, air quality and antimicrobial studies (Wang et al., 2017; Thakur et al., 2020; Tshabalala et al., 2021; Zhang et al., 2023a). The fabrication of the TiO_2 and ZnO nanofibers combines their elevated surface areas with their intrinsic semiconductor properties, thereby opening enormous potential for these materials. A critical evaluation of the literature published in recent years on the use of electrospun TiO_2 and ZnO nanofibers for the treatment of EOPs is discussed next. The pollutants of interest are mainly dyes and pharmaceuticals, although others are briefly discussed as well.

4.1 Dyes

Dyes are colored synthetic or natural pigmented substances that chemically bond to various substrates, thereby imparting a color change (Kumar et al., 2021). They are widely utilized in various industries including, textile, tannery, as well as paper and pulp (Gomes et al., 2016; Liu, 2020). The release of dye effluents into the environment and water reservoirs has thus increased, causing drastic aesthetic alterations to water bodies (Colin et al., 2016). Their presence in water also hinders the penetration of sunlight, causing a decline in photosynthesis and water oxygen levels, thereby killing aquatic fauna and flora (Lellis et al., 2019; Al-Tohamy et al., 2022). Moreover, upon ingestion of contaminated water, dyes can pose health complications due to their carcinogenic and mutagenic properties (Singh and Chadha, 2016).

Most dyes are also highly soluble and non-biodegradable, making them more persistent in the environment and difficult to remove by conventional methods (Lellis et al., 2019). This has prompted research interests in fabricating materials and devising efficient technologies for the complete eradication of dyes as well as other EOPs.

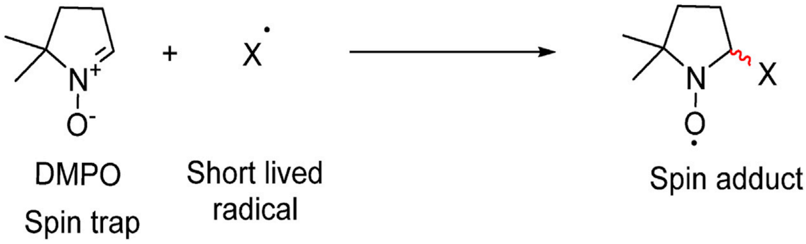

FIGURE 8
Illustration of the spin trapping method. Reprinted with permission (Al-Madanat et al., 2021).

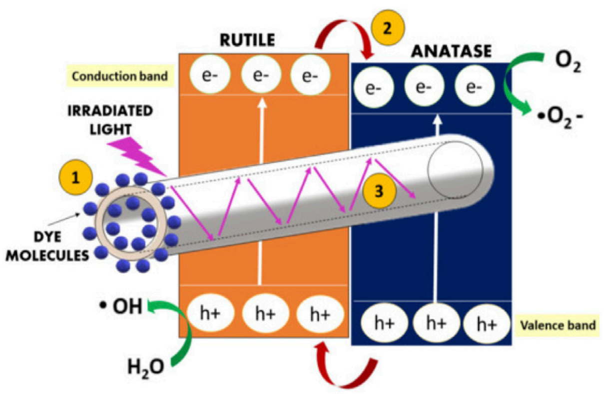

FIGURE 9
Titanium dioxide hollow nanofibers with enhanced photocatalytic properties. Reprinted with permission (Jafri et al., 2021).

4.1.1 Treatment of dyes using electrospun TiO_2 and ZnO nanofibers

Numerous researchers have reported on the efficiency of TiO_2 nanofibers for the photocatalytic treatment of dyes. For instance, Soo et al. fabricated mesoporous TiO_2 nanofibers with an anatase crystalline structure obtained after calcination at 500°C. The prepared nanofibers showed good photodegradation efficiency against methylene blue (MB) and methyl orange (MO). They attributed the efficiency of their TiO_2 nanofibers to a combination of high particle crystallinity as well as fiber porosity and surface area (Soo et al., 2018). On the other hand, Jafri et al. reported that having mixed polymorphs (anatase and rutile) in the TiO_2 nanofibers enhances their catalytic efficiency when compared to having one phase (anatase or rutile). They pointed out that their hollow TiO_2 nanofibers consisting of a mixture of 24.2% anatase and 75.8% rutile exhibited superior photocatalytic degradation of MB when compared to catalysts that consist of the anatase polymorph. They obtained their highest photocatalytic degradation efficiency of 85.50% at a catalyst loading of 0.75 g/L and dye concentration of 10 ppm. As shown in Figure 9, they ascribed the excellent performance of the mixed polymorph nanofibers to: (1) the extended surface area for UV irradiation and pollutant molecule adsorption; (2) accelerated electron-hole separation due to heterojunctions between the anatase and rutile phase; and (3) efficient light utilization due to light scattering effect in hollow nanofibers (Jafri et al., 2021).

In addition to the polymorphs being crucial for the photocatalytic behavior of the resulting inorganic nanofibers, modification frequently further enhances the photocatalytic performance of the final products. In their study, Formo et al. evaluated the effect of modification of anatase TiO_2 nanofibers with other nanomaterials on their catalytic performance. They fabricated and calcined the nanofibers in air at 510°C, followed by functionalization with Pt nanoparticles and nanowires. They found that the nanofibers showed excellent catalytic activity for the hydrogenation of azo bonds in methyl red (MR), even more so when modified with Pt nanoparticles (Formo et al., 2008). More examples of studies that have been conducted on the use of TiO_2 nanofibers for the photocatalytic degradation of dyes are listed in Table 1.

The use of ZnO-based nanofibers for the photocatalytic treatment of dyes has also been conducted and reported. In their research work, Pantò et al. fabricated ZnO nanofibers consisting of interconnected polycrystalline nanoplatelets with defect-rich hollow nanostructures. The as-prepared ZnO nanofibers were evaluated for the degradation of MB under 350 nm UV and outperformed most state-of-art electrospun pure ZnO photocatalysts (Pantò et al., 2021).

Doping ZnO nanofibers was also reported to further improve their overall photocatalytic performance. Ersöz et al. doped ZnO nanofibers with Ag to evaluate the catalytic effect of the resultant Ag-doped ZnO nanofibers. They used rhodamine B (RhB), MO and MB as model dyes and found that the Ag-doped ZnO nanofibers showed enhanced degradation efficiency relative to the unmodified ZnO nanofibers (Ersöz and Altintas Yildirim, 2022).

Similarly, Jian et al. prepared a series of La-doped ZnO nanofibers. The La-doped ZnO nanofibers were found to have a higher photocatalytic efficiency in the degradation of RhB when illuminated with visible light relative to the undoped ones (Jian et al., 2022). Baylan and Yildirim fabricated Mn-doped ZnO nanofibers wherein substitutional incorporation of Mn^{2+} and Mn^{4+} ions in ZnO resulted in the generation of additional energy levels within the band gap of ZnO. The substitutional incorporation of the dopant ions also resulted in slight morphological variations and enhanced the photocatalytic efficiency of the ZnO nanofibers in MB degradation. This was attributed to the formation of a greater number of charge carriers and the corresponding delay in the recombination process relative to the bare ZnO nanofibers (Baylan and Altintas Yildirim, 2019). More examples of studies that have been conducted on the use of ZnO nanofibers for the photocatalytic degradation of dyes are reviewed in Table 1.

TABLE 1 Recent literature on electrospinning parameters and photocatalytic performances of TiO_2 and ZnO nanofibers against dyes.

Nanofiber	Precursor	Polymorph	Pollutant(s)	Electrospinning conditions			Pollutant concentration (ppm)	Catalyst dosage (gL^{-1})	Time (min)	Degradation efficiency (%)	Ref
				Tip to collector (cm)	Voltage (kV)	Flow rate (mLh^{-1})					
TiO_2	Titanium (IV) isopropoxide	Anatase	Methyl Orange, Methylene Blue	12.00	20.00	1.00	10.00	1.00	45.00	97.20	Soo et al. (2018)
	Titanium (IV) isopropoxide	Anatase, Anatase-Rutile	Methylene Blue	15.00	12.00	1.00	10.00	0.75	240.00	85.50	Jafri et al. (2021)
	Titanium tetraisopropoxide	Anatase	Methyl Red	-	10.0	0.30	12.40	-	-	-	Formo et al. (2008)
	Titanium (IV) propoxide	Rutile	Methyl Orange	18.00	12.00	3.60	10.00	0.25	180.00	78.00	Thakur et al. (2021)
	Titanium (IV) n-butoxide	Anatase, Anatase-Rutile and Rutile	Remazol Black B	15.00	15.00	0.50	20.00	1.00	-	97.00	Secundino-Sánchez et al. (2022b)
	Titanium (IV) isopropoxide	Anatase	Methyl Orange	12.00	20.00	0.50	10.00	1.00	180.00	>90.00	Hamadanian et al. (2011)
	Titanium (IV) isopropoxide	Anatase-Rutile	Rhodamine B	15.00	16.00	0.50	-	-	120.00	100.00	Suphankij et al. (2013)
	Tetra-n-butyl titanate	Anatase-Rutile	Rhodamine B	15.00	15.00	-	10.00	0.20	100.00	92.10	Wang et al. (2019)
	Titanium-tetraisopropoxide	Anatase, Anatase-Rutile	Basic Blue 26, Basic Green 4, Basic Violet 4	20.00	0–35.00	-	10.00	-	180.00	78.70	Doh et al. (2008)
	Titanium (IV) isopropoxide	Anatase-Rutile	Methyl Orange, Methylene Blue	15.00	15.00	0.12	10.00	0.50	100.00	-	Nirmala et al. (2012)
	Titanium-tetraisopropoxide	Anatase	Methylene Blue	22.00	20.00	1.00	25.00	0.40	60.00	98.00	Kudhier et al. (2021)
	Zinc acetate	Wurzite	Methylene Blue	11.00	17.00	1.41	-	1.00	85.00	90.00	Pantò et al. (2021)
			Rhodamine B Methylene Orange Methylene Blue	15.00	15.00	0.40	5.00	0.20	120.00	66.00 51.00 97.90	Ersöz and Altintas Yildirim (2022)
			Rhodamine B	20.00	15.00	1.20	5.00	1.20	510.00	94.31	Jian et al. (2022)
			Methylene Blue	15.00	15.00	0.40	5.00	0.50	90.00	100.0	Baylan and Altintas Yildirim (2019)

(Continued on following page)

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TABLE 1 (*Continued*) Recent literature on electrospinning parameters and photocatalytic performances of TiO_2 and ZnO nanofibers against dyes.

Nanofiber	Precursor	Polymorph	Pollutant(s)	Electrospinning conditions: Tip to collector (cm)	Voltage (kV)	Flow rate (mLh^{-1})	Pollutant concentration (ppm)	Catalyst dosage (gL^{-1})	Time (min)	Degradation efficiency (%)	Ref
ZnO			Methylene Blue	15.00	15.00	0.30	-	-	360.00	96.40	Aquí-Romero et al. (2022)
			Methyl Orange	13.00	18.00	0.50	10.00	1.00	30.00	100.00	Zhang et al. (2023b)
			Malachite Green								
			Methylene Blue								
			Congo Red	15.00	20.00	0.75	10.50	0.28	240.00	97.63	Pascariu et al. (2019)
			Methylene Blue	12.00	8.50	0.40	-	0.40	225.00	-	Samadi et al. (2014)
			Methylene Blue	10.00	17.50	0.20	10.00	0.40	360.00	>80.00	Liu et al. (2018)
			Methylene Blue	10.00	8.00	0.18	-	0.10	30.00	-	Han et al. (2013)
			Methylene Blue	17.00	25.00	0.10	-	-	60.00	-	Busuioc et al. (2015)

Furthermore, comparison of the dye degradation efficiencies of the TiO_2 and ZnO nanofibers prepared in similar conditions have been conducted. Mapukata and Nyokong reported that under the same experimental conditions, anatase TiO_2 nanofibers showed better photodegradation of MO than the wurzite ZnO nanofibers (Mapukata and Nyokong, 2020). Arshad et al. conducted a similar study wherein they fabricated TiO_2 and ZnO nanofibers followed by calcination at 450°C. They reported that under the same fabrication and processing conditions, the TiO_2 nanofibers had better photoactivity and thus a higher MO degradation efficiency. They attributed this to the TiO_2 nanofibers being more aligned and electron-supportive for conduction as compared to ZnO nanofibers, which were dense and agglomerated at some point. Moreover, their Hall Effect measurements showed that the TiO_2 nanofibers had a higher conductivity of 1.38×10^{-04} $\Omega.cm^{-1}$, compared to ZnO nanofibers with 1.08×10^{-04} $\Omega.cm^{-1}$ (Arshad et al., 2023).

Some researchers also studied the photodegradation of dyes using composites of the two nanofibers, i.e., TiO_2/ZnO nanofibers. For instance, Pei and Leung evaluated the photocatalytic activities of TiO_2/ZnO nanofibers in the degradation of RhB under the 420 nm visible-light irradiation. The nanofibers' diameters ranging between 80 and 130 nm were obtained by varying the concentrations of zinc acetate in the precursor solutions between 0.15% and 0.6% (Pei and Leung, 2013). 2g L^{-1} of TiO_2/ZnO was found to be the optimal loading for efficient removal of RhB. Similarly, Leet et al. fabricated TiO_2/ZnO nanofibers, which showed higher photocatalytic properties when compared to TiO_2–based nanofibers in the degradation of MB. The higher activity of the composite nanofibers has been attributed to the transfer of electrons from ZnO to the TiO_2 and transfer of holes from the TiO_2 to the ZnO. This enhances the generation of $HO^{\bullet}$ and $O_2^{\bullet -}$, thereby increasing the degradation efficiency (Lee et al., 2019).

4.2 Pharmaceuticals

Pharmaceutical products have been found to be present in sewage treatment plants, surface water, ground water and drinking water due to reckless disposal of drugs and direct release from the manufacturing industries (Waleng and Nomngongo, 2022). Additionally, they can only be partly metabolized during therapeutic use, resulting in the excretion and release of residual fractions through human and animal waste (Frascaroli et al., 2021). This accelerates the spread of therapeutic resistance, thus causing a threat to human health and ecological systems (Serwecińska, 2020). Moreover, the occurrence of pharmaceuticals in potable water has become pervasive, presenting life-threatening issues because amongst other things, they are potential endocrine disruptors (Massima Mouele et al., 2021). Complete mineralization of these pharmaceuticals has proven to be a challenge using many conventional methods, thereby prompting research efforts in other fields (Papagiannaki et al., 2022). As a result, numerous researchers have proposed using TiO_2 nanofibers as efficient materials for the treatment of various pharmaceuticals as discussed next.

4.2.1 Treatment of pharmaceuticals using TiO_2 nanofibers

Javid et al. evaluated the performance of TiO_2 nanofibers calcined at 560°C in the oxidation of the antibiotic tetracycline. At optimum conditions (tetracycline concentration: 50 mg/L, pH = 8.3, time = 15 min), they reported a maximum degradation efficiency of around 95% (Javid et al., 2013). Dhal et al. fabricated TiO_2 nanofibers with mixed phases (anatase and rutile) which were efficiently applied in the degradation of tetracycline hydrochloride among other contaminants. The excellent photocatalytic activity of the nanofibers was attributed to the heterojunction at anatase–rutile interface and immediate charge transfer due to 1D structure, which inhibit the electron–hole recombination (Dhal et al., 2019). Moreover, Lin et al. supported TiO_2 nanofibers onto boron-nitride sheets and applied them in the photocatalytic destruction of ibuprofen; a nonsteroidal anti-inflammatory drug. Relative to the pure TiO_2 nanofibers, the supported ones had reduced recombination of charge carriers and higher photocatalytic efficiency under visible light (Lin et al., 2019).

Additionally, Pascariu et al. studied the effect of doping TiO_2 nanofibers on their photocatalytic efficiency against pharmaceuticals. They prepared pure TiO_2 and La^{3+}-doped TiO_2 nanofibers for the treatment of the antibiotic ciprofloxacin amongst others pollutants. They optimised the La^{3+}-dopant content and calcination temperature to 0.1% La^{3+} and 600°C, respectively to achieve 99.5% degradation efficiency after 300 min irradiation under visible light (Pascariu et al., 2022a). In another study, Pascariu et al. doped the TiO_2 nanofibers with Sm^{3+} and Er^{3+} at doping levels tuned in the range of 0.05%–1.0% for the mineralisation of ciprofloxacin (Pascariu et al., 2022b). They reported that TiO_2:Sm (0.1%) calcined at 600°C showed superior catalytic activity towards ciprofloxacin, exhibiting a removal efficiency of ~99.6%. Similarly, Liao et al. prepared hierarchical BiOI/TiO_2 composite nanofibers for the degradation of tetracycline under visible light irradiation (Liao et al., 2022). They obtained a degradation efficiency of ~99%, attributing it to the large surface area, interconnected interfaces and readily exposed active sites on the BiOI/TiO_2 composite nanofibers. Moreover, a recent study by Ruiz-Ramírez et al. reported on the preparation of bare and Ag-doped SiO_2/TiO_2 composite nanofibers for the photocatalytic degradation of the antibiotic oxytetracycline. They found that the undoped composite nanofibers had a degradation percentage of 65%, which improved to 90% upon doping with Ag (Ruiz-Ramírez et al., 2023).

The degradation of pharmaceuticals using ZnO nanomaterials has been reported (Sabouni and Gomaa, 2019; Tanveer et al., 2019; Al-Khadhuri et al., 2023). However, to the best of our knowledge, there are no reports on the use of electrospun ZnO nanofibers for the treatment of pharmaceuticals, an area that can be explored in future research endeavors.

4.3 Treatment of other EOPs using TiO_2 and ZnO nanofibers

When released into the environment, wastewater containing polycyclic aromatic hydrocarbons (PAHs) can disrupt the ecosystem and degrade the quality of water bodies (Pathak et al., 2023). Exposure to wastewater with PAH like naphthalene can cause headaches, vomiting, diarrhea, abdominal pain, fever and alter mental status (Volney et al., 2018). Mondal et al. proposed the use of TiO_2 nanofibers for the photocatalytic treatment of these contaminants. They fabricated partially aligned free-standing mesoporous pure anatase TiO_2 nanofiber mats for the photocatalytic degradation of naphthalene. Doping the nanofibers with carbon residue (2.54%) doubled the efficiency of the pristine TiO_2 nanofibers in the photodegradation of the PAH, but the effectiveness declined at higher carbon content (6.45% and 10.91%) (Mondal et al., 2014). On the other hand, Sekar et al. also successfully degraded naphthalene using Fe-doped ZnO nanofibers (Sekar et al., 2018).

Wastewater containing phenols can also be very toxic, even at low concentrations (Fseha et al., 2023). Phenolic compounds are widely used in various industries including fertilizers, paints, explosives, rubbers, plastics, cosmetics, and antioxidants (Balasundram et al., 2006; Singh and Yadav, 2022). The ingestion of water contaminated with phenols can lead to severe health effects including ailments of the liver, kidney, gastrointestinal and central nervous system (Chand Meena et al., 2015; Panigrahy et al., 2022). Electrospun TiO_2 nanofibers have been proposed as efficient materials for the photocatalytic treatment of these contaminants. Norouzi et al. prepared Ag-doped TiO_2 nanofibers and applied them for the photodegadation of phenol under the visible light (Norouzi et al., 2020). They varied the phenol concentration, pH, dopant loading and catalyst dosage to get optimum degradation efficiencies. A maximum degradation of 93% was achieved when the phenol content was 5.62 ppm, the catalyst loading was 2.06 g L^{-1}, and the pH value was 8. Rongan et al. prepared composite Bi_2O_3/TiO_2 nanofibers with an S-scheme heterojunction, which exhibited superior photocatalytic activity for removal of phenol under visible light irradiation in comparison to pristine Bi_2O_3 and TiO_2 nanofibers (Rongan et al., 2020). Similarly, Nada et al. fabricated composite WS_2/TiO_2 nanofibers, which showed a remarkable performance in phenol degradation (83%) under visible light (Nada et al., 2022).

The release of bisphenol A (BPA) into water bodies; which is a common monomer in a variety of industries, including the manufacturing of food containers, plastic bottles, and electronic equipment can have adverse human and animal health effects. Devastatingly, BPA has estrogen-like and anti-androgen effects causing damages to different tissues and organs, including reproductive system, immune system and neuroendocrine system (Michałowicz, 2014; Ma et al., 2019). To eradicate such a contaminant, Jafri et al. proposed the use of free-standing TiO_2 hollow nanofibers to photocatalytically mineralise BPA (Jafri et al., 2022). The process entails the attack of electron-rich C3 in the phenyl group of the BPA by $HO^{\bullet}$ from the excited photocatalyst (see Figure 10). Subsequently, the cleavage of the phenyl groups, forming 4-isopropanolphenol and phenol occurs. Hydroquinone is produced from the oxidation of 4-isopropanolphenol. These compounds are further degraded into short-chain aliphatic compounds through ring-opening reactions by the •OH radicals, eventually forming carbon dioxide and water.

Another industry that generates waste with high organic content is the dairy industry. Dairy effluent is rich in organic substances including lactose, whey proteins, minerals, and lipids (Das et al., 2016; Usmani et al., 2022). The complexity of this waste has made it challenging for researchers to treat it efficiently. Electrospun ZnO nanofibers have been proposed as suitable candidates for the photocatalytic treatment of dairy effluent. Kanjwal and coworkers fabricated ZnO nanofibers and

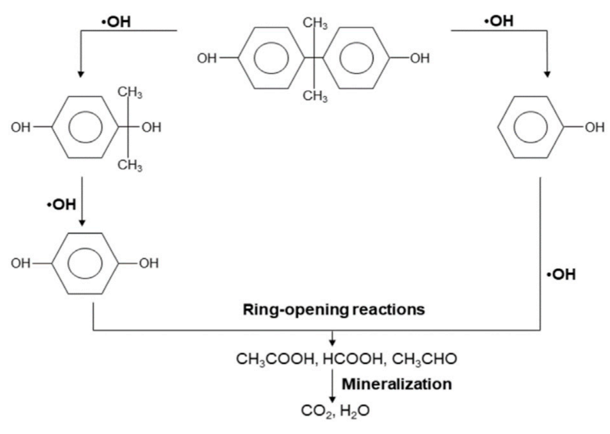

FIGURE 10
Proposed photocatalytic degradation pathway of BPA. Reprinted with permission (Jafri et al., 2022).

composite ZnO/NiO nanofibers amongst other materials and applied them in a range of pollutants including dairy effluents (Kanjwal et al., 2015). The ZnO nanofibers showed a maximum degradation of 75% for the dairy effluents, while the composite ZnO/NiO nanofibers eliminated 80% within 3 h.

Lastly, aside from photocatalytic decomposition of wastewater contaminants, TiO_2 nanofibers have also been applied for oil-water separation. This is because oil pollution is an ever present threat in the marine environment with a large numbers of spills, being recorded every year (Asif et al., 2022). Zhang et al. electrospun pine-branch-like TiO_2 nanofibrous membranes for oil-water separations. The resultant hierarchical TiO_2 membrane displayed superhydrophilicity and underwater superoleophobicity. The hierarchical TiO_2 membrane could separate various highly corrosive acidic, basic, and salty oil-in-water emulsions with high separation efficiencies (>99%) and stable flux (Zhang et al., 2017).

5 Conclusion and outlook

In summary, TiO_2 and ZnO nanostructures have been used for the elimination of EOPs in wastewater. Their photocatalytic properties coupled with other properties such as good chemical and optical stability, relatively low cost, abundance, corrosion resistance and non-toxicity have publicized their extensive usage in water treatment applications. Their effective use relies on their morphological properties and in this instance, the fiber morphology that offers several advantageous characteristics including high surface-area-to-volume ratio, high porosity, superior mechanical strength, versatile surface functionalization and tuneable properties. Synthesized through electrospinning technique, these characteristics can further be adjusted by varying the techniques parameters to offer fibers with a range of sizes and porous structures. The electrospun nanofibers are also favorable for their easy regeneration and reusability. Modifications to enhance the photocatalytic performance of the TiO_2 and ZnO nanofibers have been achieved using different strategies, such as doping, dye photosensitization and through the creation of heterogeneous nanofibers. Extensive research studies on the application of TiO_2 and ZnO nanofibers as well as their modified versions have demonstrated the effectiveness of using these materials for the photocatalytic decomposition of wastewater contaminants, such as pesticides, pharmaceuticals, dyes and other pollutants. The need to protect the aquatic environment from these contaminants have seen the growth of research in development of water treatment technologies. Not only are the contaminants posing a threat to human health, but to the aquatic environment and life as well. The use of TiO_2 and ZnO nanofibers and their modified versions in water treatment is promising, owing to the advances in research and innovation towards improving their efficiency, thereby ensuring access to safe and clean water. The reported literature shows that the TiO_2 nanofibers have a higher photocatalytic activity relative to the commonly used commercial Degussa P-25. Moreover, the properties of the TiO_2 nanofibers are highly dependent on the calcination temperatures due to the different polymorphs, wherein the purely anatase nanofibers are predominantly favored. The ZnO nanofibers still need to be explored for photodegradation of emerging pollutants, such as pharmaceuticals. It is however worth mentioning that the fabrication and modification of TiO_2 and ZnO nanofibers results in photocatalysts with exceptional properties and potential in wastewater treatment applications.

Author contributions

SM: Conceptualization, Formal Analysis, Writing–original draft, Writing–review and editing. KS: Writing–review and editing. TM: Writing–review and editing.

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Conflict of interest

The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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