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Review Article

Titanium dioxide electrospun nanofibers for dye removal- A review

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Abstract
Due to rapid urbanization and industrialization, water demand has increased worldwide. The availability of potable water is becoming more difficult in the global scenario. Hazardous pollution disposal by the industries to the nearest stream and search for the facile environmentally friendly technologies capable of treating these pollutants become more challenging. Effluent disposal consisting of the dyes without proper pre-treatment adversely affects the aquatic life and ecological system as they are carcinogenic and highly toxic. Dyes in the water are becoming a significant problem in the current scenario and attracted many researchers to research the current topic. Even though the conventional treatment options are available for treating polluted water, still they are not enough for the demand and supply. Thus, new state-of-the-art technologies are required to meet the demand and supply. Titanium dioxide nanofibers synthesized by electrospinning techniques have proven to be new nanomaterials gaining prominence in science. Several researchers are using these fibres by fabricating them into a thin film for pollutant removal and water treatment. They are gaining much importance as they perform best in treating water containing both organic and inorganic loads. The present review provides insights into the background and the origin of the electrospun nanofibers and preparation mechanisms. Further, we identified 25 widely used titanium dioxide electrospun nanofibers with various combinations in removing the dyes from the aqueous medium.

Keywords: Electrospinning, Nanofibers, Titanium dioxide, Wastewater treatment

INTRODUCTION

Surface water pollution by the organic dyes is commonly found due to the partially treated effluents released from the various industries (Khan *et al.,* 2019). Dyes are stable synthetic compounds resistant to photo light, microbial degradation and extreme temperature, and further, these are toxic and carcinogenic (Jose *et al.,* 2021; Khan et *al.,* 2022; Bölgen and Vaseashta, 2021). Several techniques have been employed to treat water and wastewater, including coagulation/ electrocoagulation, biological processes, oxidation, membrane technologies, phytoremediation, extraction techniques, etc. Still, none of them is proven efficient in removing all the pollutant parameters. (Nidheesh and Singh, 2017; Marinho *et al*., 2021). During recent decades metal oxides of titanium were utilized chiefly in applying photocatalytic activity. The critical factor for widespread is its chemical stability and low cost of operation. However, using these substances in suspension to eliminate the catalyst in water is complicated, making usage of these oxides not economically viable

(Matsuzaw *et al*.,2008; Qi *et al*.,2021; Marinho *et al*.,2021); To deal with this problem, one of the alternatives is to functionalize the metal oxides with the catalyst nanoparticles, making the separation of water from pollutants easy as it is highly stable. Various techniques have been adopted to fabricate the different types of titanium oxide and catalyst films (Vella *et al*.,2010); nevertheless, the nanofibers fabricated by the electrospun technique are good examples of the next generation nanocatalysts (Altaf *et al*.,2020). As this method is versatile, it can fabricate polymers, composites, and inorganic materials from nanomaterials with controlled diameters (Armstrong *et al*.,2020). The nanofibers produced by the electrospun techniques have larger surface areas and high pore volume with interconnectivity favouring methods like water remediation, energy storage, and conversion (Song *et al*., 2021; Marinho *et al*., 2021). The research works related to titanium oxide nanofibers are increasing. These substances are highly potential in environmental applications compared to conventional catalysts due to their unique parameters, and these nanofibers help overcome water pollution-related problems (Xu *et al*.,2018; Pascariu *et al*.,2019; Marinho *et al*., 2021). Indeed, the utilization of nanofibers fabricated by the oxides of titanium is already proven to be best in removing the pollutants than the nanoparticles produced (Altaf *et al*.,2020; Ehsani and Aroujalian, 2020). In this connection, the current review presents a history of electrospinning, techniques and applications of titanium dioxide nanofibers in dye removal.

BACKGROUND

Marinho *et al*. (2021) stated that before developing titanium dioxide nanofibers, several researchers conducted many studies for more than four centuries. Fig, 1 depicts the brief timeline of evolving electrospinning techniques until the first research publications regarding titanium dioxide nanofibers by an electrospun method in 2002. William Gilbert, in 1628 observed the changes in the shape of the water droplet when it encountered the external electrical field, and it was found to be the first record of electrospinning. Primarily, the spherical water drops attracted the amber piece and changed its shape to a cone, and this progression is called as "Taylor cone" (Gugulothu *et al*.,2018; Barhoum *et al*.,2019). Further in the next two centuries, variations in these shapes in contact with electrical charges were studied by many researchers. A researcher named Charles V. Boys in the year 1887 found that fibres can be fabricated by using a viscid liquid with the help of a dish connected with an electrical charge (Xue *et al*.,2019). After a few years, JF Cooley, in 1902, filed a patent in the USA titled "Apparatus for electrically dispersing fibres" with his observations and description about electrospinning (Cooley, 1902). Later in the twentieth century, electrospinning techniques were spread around the globe, primarily in the production of water filters and industrial applications. Doshi and Reneker 1995 reported electrospinning techniques with the usage of various polymers (Xue *et al*.,2019). These techniques are popularized and led to modern electrospinning concepts, which ushered in the production of ultrathin fibres having a diameter at a nanoscale level (Tucker *et al*., 2012). The research rapidly disseminated the incorporation of metal oxides and metal nanoparticles into the fibres fabricated by electrospun methods. Many researchers described incorporating titanium dioxide nanofibers in 2001-2002. After that, the publications on electrospinning techniques have increased each year exponentially. It was

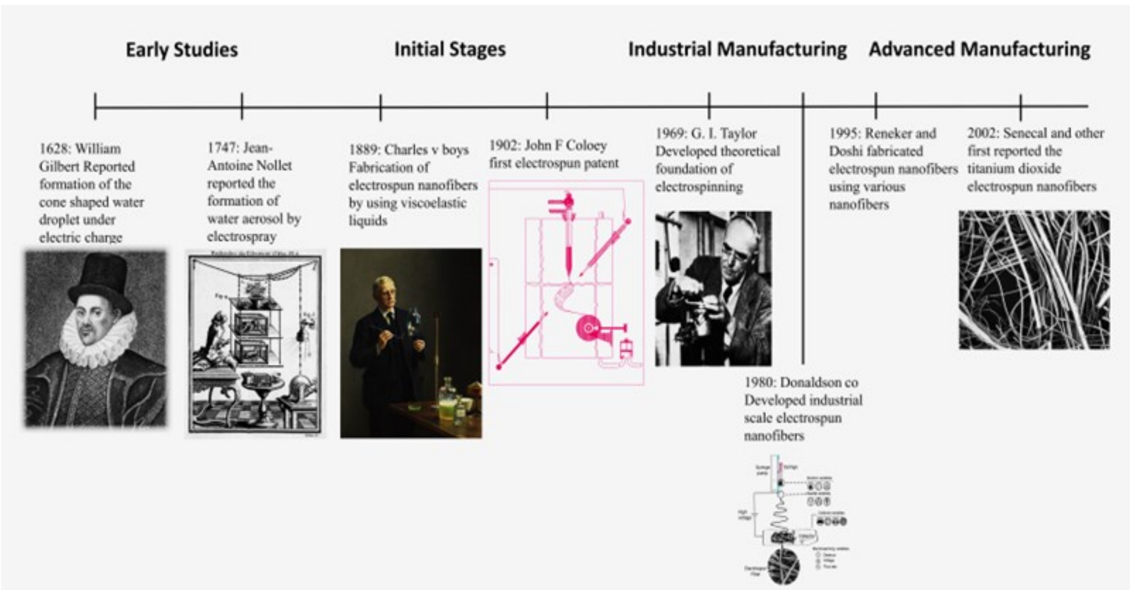

Fig. 1. *A brief timeline of electrospinning*

estimated that more than two hundred types of polymers had been fabricated into nanofibers by using electrospun techniques for several applications (Zhu *et al*.,2020). In addition, these nanofibers can be produced in larger volumes, allowing the manufacturing of more recent commercial goods; the present uses on-air/water filtration, biomedical products, and facial masks. In the meantime, the research organizations are developing innovative methods and compositions that provide specific functions in advanced applications (Barhoum *et al*.,2019; Marinho *et al*.,2021).

Process of Electrospinning

Electrospinning techniques are simple, have low operation costs, and continuously produce nanofibers in bulk production (Kim *et al*.,2021). The significant benefits of electrospinning are required simple equipment compositions and have high flexibility in nanofiber orientations (Liao *et al*.,2018). Thus, the electrospinning technique has gained much prominence in research worldwide (Istirohah *et al*., 2019). Electrospraying is a technique that depends on the ejection of liquids from the jets under high voltage, and it is an electrohydrodynamic process. In the electrospraying technique, jets will break down the droplets to produce a particle, whereas, in the electrospinning process, the jet continuously makes the nanofibers. The liquids' viscosity and elasticity are the main features that determine the behaviour of the jets (Xue *et al*.,2019). The setup of the electrospinning process is simple and accessible by all laboratories, as shown in Fig. 2 (Marinho *et al*.,2021). The equipment configuration includes a power supply, an injection pump, and a sharp tip and collector. Nevertheless, different electrospinning designs were found worldwide, which provide for multiple needles with a jet (Démuth *et al*.,2016; SalehHudin *et al*.,2018), coaxial shape needles (Prado-Prone *et al*., 2018), without needles (Ali *et al*.,2017), and liquid bath connected to a solid collector (Zhou *et al*., 2009; Wu and Hong, 2016).

Different types of materials were used to prepare nanofibers by the electrospinning technique. However, organic polymer solutions were primarily used in electrospinning methods (Liao *et al*., 2018). Alternative methods are introducing nanomaterial into the polymer solutions to produce nano functionalized fibres. This process has created many opportunities for researchers to test several combinations of nanoparticle-solvents-polymers to fabricate nanofibers for varied applications. One of the first reported research for the fabrication of titanium dioxide nanofibers used a composition of ethanol dissolved polyvinyl pyrrolidone and titanium tetraisopropoxide (Marinho *et al*., 2021).

Titanium dioxide nanofibers in dye removal

Titanium dioxide materials have more significant advantages than other materials as they show higher photoactivity and higher stability (Wang *et al*.,2019). Indeed, titanium dioxides are the most used semiconductors for photocatalytic applications (Greenstein *et al*.,2021). Even though many researchers have studied these materials, most of the information related to titanium dioxide is related to its production in suspension or powder forms. Nevertheless, recycling these materials is a complex and costly process, as separating catalyst powder from liquids is difficult (Ananpattarachai and Kajitvichyanukul, 2016). This problem can be avoided by functionalizing titanium dioxide with active semiconductor nanoparticles; it eliminates the post-filtration step and allows the catalyst for reuse with more excellent stability. Several methods like chemical vapour deposition, sol-gel techniques, sputtering, physical vapour deposition, and sputtering, have fabricated titanium dioxide and catalyst supports (Sonawane *et al*.,2003). Moreover, materials like paper, ceramics, pumice stones, glass, and stainless steel are often tested as catalyst supports (Vella *et al*., 2010). Electrospinning techniques are simple and economical methods for fabricating nanofibers continuously with even diameters with various compositions (Someswararao *et al*., 2018). One dimensional nanomaterials also gained much prominence in research as titanium dioxide nanofibers due to their larger surface areas and photocatalytic activity (Pascariu *et al*., 2019; Marinho *et al*., 2021). Conventionally, the titanium oxide nanofibers are fabricated by electrospinning technique by incorporating titanium dioxide as a precursor with a polymer.

Further, the nanofibers are calcinated to change their amorphous phase to crystalline (Mahltig *et al*.,2007). Although titanium dioxide is used for calcination and precursors, nanofibers can be fabricated even by electrospinning methods using blended semiconductor fibres alone from them. The coaxial and dual electrospinning techniques are more effective for new nanofiber production. Luo *et al*. (2016) used the coaxial design to spin the titanium dioxide and polyvinyl alcohol nanoparticles concurrently.

Nevertheless, it is essential to note that nanofibers fabricated by polymeric solutions containing titanium dioxides might show instabilities, and both materials demixing can occur (Grothe *et al*., 2018). However, with more excellent stabilities, titanium dioxide nanofibers by mixing polyetherimide and titanium dioxide nanopowder in dimethylformamide and tetrahydrofuran. The nanofibers fabricated are subjected to the cold plasma in nitrogen atmospheres to enhance their photocatalytic and adhesion properties. The material showed excellent photocatalytic activities for the discolouration of methylene blue with higher stability, and the same was tested for five cyclic performances. The alternative approach is to coat the purest electrospun nanofibers to the titanium dioxide after the electrospun process. Indeed, it is one of the first approaches for

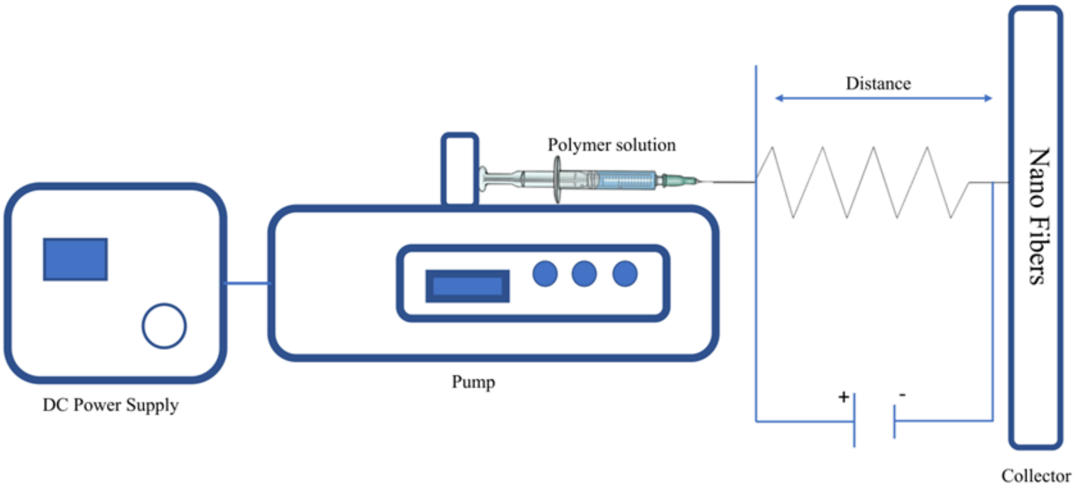

Fig. 2. *Diagrammatic representation of electrospinning device*

fabricating titanium dioxide nanofibers. Drew *et al*. (2003), synthesized the nanofibers using polyacrylonitrile by immersing them in a solution consisting of titanium dioxide. The structural properties of the pristine titanium dioxide nanofibers can be modified by adding transition-metal/nonmetals to form composites (Pascariu *et al*.,2019). Kudhier *et al*. (2018) compared the pristine and silver doped titanium nanofibers; the material's bandgap was decreased by adding silver to the titanium dioxide nanofibers, which enhanced the antibacterial activity. Correspondingly, titanium dioxide nanofibers were fabricated by doping with the graphitic carbon nitride to titanium dioxide in polymer solution adding urea. Heterojunctions are formed between the semiconductor materials due to graphitic carbon nitride with titanium dioxide enhancing the photocatalytic activity by suppressing the recombination charge (Tang *et al*., 2018).

The properties of the nanofibers are mainly dependent on the polymer solutions utilized, working procedures and laboratory conditions (Pascariu *et al*., 2019). Kim *et al*. (2018)stated that modified aluminium collectors would help in the unidirectional growth of nanofibers. The unidirectional nanofibers show higher crystallinity, act as electron transport, and enhance the nanofibers' optical/mechanical properties.

In recent years many techniques have been developed to treat water and wastewater. Several technologies have been examined to find better treatment options at lower costs (Chen *et al*.,2020). Generally, conventional technologies like coagulation, adsorption and biological processes are easily operated and involve lesser costs. Nevertheless, they have a disadvantage like sludge disposal and a longer duration of the water treatment (Bora *et al*.,2016). In contrast, ultrafiltration, photocatalytic degradation, and electrochemical methods involve initial high capital investment and energy; however, these methods have long-term advantages (Ortega *et al*., 2017). Water and wastewater treatment methods are never unique or straightforward and need continuous improvement and different approaches (Song *et al*.,2017). In this regard, the titanium dioxide nanofibers fabricated by the electrospinning technique have more significant advantages for removing pollutants by the methods like membrane filtration, photocatalytic activity, and adsorption (Li *et al*., 2014; Marinho *et al*., 2021).

Titanium dioxide nanofibers are suitable adsorbents for eliminating heavy metals from the water as their surfaces consist of carboxyl, hydroxyl groups, etc. (Zhu *et al*.,2020). As hydrophilicity of the nanofibers is enhanced by titanium dioxide; further it also increases the stabilities, mechanical strength, and anti-smudge properties when they are utilized in the process of membrane filtration (Chen *et al*., 2020). The surface roughness of the nanofibers is improved, which helps for the desalination of water (Pan *et al*., 2019). Furthermore, the titanium dioxide photocatalytic properties also helped regenerate electrospun nanofibers. Li *et al*. (2014) conducted studies on the titanium dioxide electrospun nanofibers for dye removal and achieved removal percentages of 92 for methylene blue, 95 for the congo red and 52 for methyl orange. The pH of the solutions affects the charge of the titanium dioxide particles significantly. The zero-point charge is termed pH, where the surface of the particles is not charged. Singh *et al*.,2003 reported that the zero-point charge's commercial titanium dioxide nanoparticles pH is 6.2; beyond this value might negatively affect the catalysts and attract the other molecules.

On the other hand, the reactant adsorption phenomena are negatively affected and limit the reactions at higher temperatures, i.e., above 80^{o}C; further, the oxygen concentration in the water decrease by increasing the temperatures. Thus, optimum temperatures for the reactions range from 20 to 80^{o}C (Malato *et al*.,2016). Even though these methods seem to be promising as they have a greater agglomeration tendency, they are challenging to separate from the aqueous medium; thus, usage of these methods is limited (Kumar *et al*., 2014). However, doping of photocatalytic nanoparticles to the electrospun nanofibers is found to be an alternative for these limitations (Peng *et al*., 2016). Comparing heterogeneous photocatalytic activity with membrane filtration to remove the toxic pollutants found that the titanium nanofibers show many advantages as they have the potency to degrade the contaminants compared to the membrane filtration and separate the solution phase easily. Li *et al*., 2017 used Heteropolyacids and titanium dioxide nanoparticles for the composite and removed the methyl orange dye with an efficiency of 94%. Ramasundaram *et al*., 2015 used Polyvinylidene fluoride and titanium dioxide nanoparticles to remove the Bisphenol A and removed the pollutants from the aqueous medium completely. Park *et al*. (2011) used silver, titanium dioxide and polyvinylpyrrolidone nanoparticles to prepare electrospun nanofibers and removed the methyl orange with an efficiency of up to 80%. In addition, traditional membrane separation methods only separate the pollutants concentrate; this method requires high energy for the operation to reduce the fouling of the membrane to maintain the constant flow (Gao *et al*., 2020). Wang et al., 2018 prepared electrospun nanofibers by doping carbon nanofibers and removing the methylene blue dye with an efficiency greater than 57 %. Khan et al., 2022 synthesized titanium dioxide nanofibers by doping them with zinc and cadmium and tested them to remove the organic dyes. These electrospun titanium dioxide nano-

Table 1: Application of electrospun titanium dioxide nanofibers for dye removal

S. no	Nanofiber constituents	Dye	Dye removal (%)	References
1	Heteropolyacids and titanium dioxide	Methyl orange	94	Li *et al*., 2017
2	Metal organic frame works, titanium dioxide and zinc	Rhodamine	92	Hou *et al*., 2019
3	Polymethyl methacrylate and titanium dioxide	Methylene blue	100	Vild *et al*., 2016
4	Polyvinylidene fluoride and titanium dioxide	Cimetidine	100	Ramasundaram *et al*., 2015
5	Polyvinylidene fluoride and titanium dioxide	4- Chlorophenol	100	Ramasundaram *et al*., 2015
6	Polyvinylidene fluoride and titanium dioxide	Bisphenol A	100	Ramasundaram *et al*., 2015
7	Silver, titanium dioxide and polyvinylpyrrolidone	Methylene blue	100	Chang *et al*.,2009
8	Silver, titanium dioxide and polyvinylpyrrolidone	Methylene blue	80	Park *et al*.,2011
9	Titanium dioxide and bio-glass nanofibers	Methylene blue	60	Lian *et al*.,2018
10	Titanium dioxide and carbon nanofibers	Rhodamine B	80	Xu *et al*., 2016
11	Titanium dioxide and cyanide nitrogen	RhB	96	Wang *et al*., 2018
12	Titanium dioxide and graphene oxide	Propranolol	100	Gao *et al*., 2020
13	Titanium dioxide and peroxyl acetyl nitrate	Isoproturon	90	Xie *et al*., 2017
14	Titanium dioxide and polyvinylpyrrolidone	Methylene blue	90	Aghasiloo *et al*.,2019
15	Titanium dioxide and polyvinylpyrrolidone	Rhodamine	92	Wang *et al*.,2019
16	Titanium dioxide, zinc oxide and polyvinyl alcohol	Methyl orange	60	Ramos *et al*.,2020
17	Titanium dioxide, graphene oxide and polyvinyl acetate	Rhodamine	90	Seong *et al*.,2018
18	Titanium dioxide, graphitic carbon nitride and polyvinyl acetate	Rhodamine	90	Adhikari *et al*.,2016
19	Titanium dioxide, polyaniline and polyacrylonitrile	Methyl orange	90	Sedghi *et al*.,2017
20	Titanium dioxide, silver and peroxyl acetyl nitrate	Methylene Blue	99	Shi *et al*., 2017
21	Titanium dioxide, silver and peroxyl acetyl nitrate	Methylene Blue	100	Panthi *et al*., 2017
22	Zinc ferrite, titanium dioxide and polyvinylpyrrolidone	Methylene blue	100	Nada *et al*.,2017
23	Titanium dioxide, Zinc and Cadmium	Methylene blue	94	Khan *et al.,* 2022
23	Titanium dioxide, Zinc and Cadmium	Methyl orange	96	Khan *et al.,* 2022
25	Titanium dioxide and carbon nanofibers	Methylene blue	57	Wang *et al.,* 2018

fibers removed the methylene blue with an efficiency of 94% within 2 hours. Further, they have achieved 96 % removal of methyl orange with the same nanocomposites within 100 minutes.

In contrast, the utilization of titanium dioxide nanofiber proved that they are efficient in photocatalytic activities compared to the titanium dioxide nanoparticles.Table 1 depicts the list of titanium doped electrospun nanofibers to remove the dye from the aqueous medium. 1. Still, there is a need to conduct further research on these nanofibers for industrial and large-scale applications. Integrating academic research with the industries will help move the bench-scale operations to the industries that will benefit both. Further researchers should concentrate on the disadvantages of the solvents and fabrications procedure, and alternative green chemistry methods should be implemented to make the complete process sustainable.

Conclusion

The evolution in treating the water with various nanomaterials showed how the current research advances science. The literature showed that electrospun nanofiber's application in water and wastewater is efficient, and researchers have gained much prominence. Even though several bench-scale studies have proven that nanofibers are efficient in removing the organic and inorganic pollutants from the aqueous medium, further scaling up of studies is required to move these technologies to the water industries for larger-scale production and utilization. The cost expenditures in producing the electrospun nanofibers hinder their usage in developing countries like India. However, adopting these technologies in the water industry is reliable, reducing secondary unit treatment processes. Thus, these technologies must be adopted and utilized for water treatment to replace the associated costs linked with conventional water treatment units.

Conflict of interest

The authors declare that they have no conflict of interest.

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