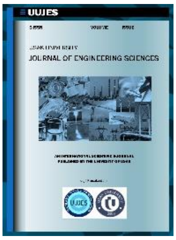

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Review article

NANOFIBERS: EXCELLENT ADSORBENTS FOR THE REMOVAL OF METHYLENE BLUE

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Abstract

As in every field, nanofibers continue to play an increasing role in environmental applications. Thanks to the unique features due to their dimensions, they enable the design and creation of indispensable products for separation, filtration, adsorption, and sensor applications over time. Adsorption, which is one of the most important of these areas, is defined as the accumulation of the adsorbates in a liquid/gas medium onto a solid adsorbent. The adsorbent is a solid material that adsorbs the dissolved compounds in liquid/gas medium. The importance of adsorbents in terms of wastewater treatment is related to the pollutant removal efficiency of adsorbents. Their performance depends on the surface area, morphology, and chemical structure. When these properties are considered, the advantages of nanofibers are again revealed. For years, research has been carried out by obtaining nanofiber surfaces from a wide variety of polymers with different nanofiber production techniques to generate creative solutions in the adsorption of various substances. The number of studies on this subject is so large that this review is limited to only methylene blue adsorption. The potentials of nanofibers for removal of methylene blue from wastewater, and some of their applications in the literature are highlighted in this review.

Keywords: *nanofibers, adsorption, methylene blue, textile wastewater, electrospinning*

1. Introduction

Developing technology to respond to increasing customer needs more quickly harms the environment in all respects. Gases released into the atmosphere, solid wastes that are uncontrollably casted away and not recycled, and factory wastewaters are among these

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damages. Wastewater is defined as the used mixture released from houses, workplaces, industries, commercial processes, or institutions subjected to treatment plants (1). Textile industry is one of the most abundant industries comprising wastewater. Nowadays, lots of obligatory rules are available and industrial mills strictly obey them. There are problems with the adsorptions of the chemical compounds that are much more dangerous or hazardous to the nature and environment. According to the compounds that should be adsorbed; physical, biological, and chemical treatments are conducted before releasing the wastewater considering the national or international wastewater standards (2). One of these hazardous compounds frequently met in textile wastewater is methylene blue (MB) which is also called methylthioninium chloride.

MB is a compound with heterocyclic aromatic structure. The chemical structure of MB is given in Fig. 1. When it dissolves in water, it results a dark blue color. Its molar absorption coefficient is high. Thus, it causes a decrease in the transfer of sunlight and decomposes the aquatic habitat. Additionally, MB has the disadvantage of producing single oxygen by photoactivation. These oxygen atoms are known to have considerably high cytotoxic levels.

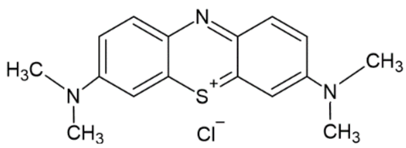

Fig. 1 Chemical structure of methylene blue

Textile wastewaters include residuals of MB, a cationic and nondegradable dye, that has a toxic effect in nature. It is important to eliminate this compound to protect both aquatic ecosystems and water bodies (3, 4).

When MB is mixed with water, it makes water sources (that should be colorless) dark blue (5). Blocking sunlight and affecting aquatic organisms are some disadvantages. It is stated in the literature for MB has maximum absorption between 665-670 nm in the visible light region (6). The UV-vis spectrum of MB was recorded by a UV-vis spectrophotometer (Biochrom Libra S70) and it was observed that the maximum absorption peak was at 670 nm (Fig. 2).

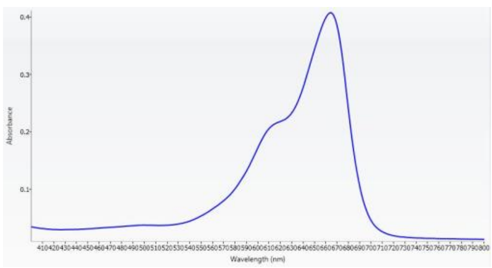

Fig. 2 UV-vis absorption spectra of MB

Since the removal of the colorful, environmentally and ecologically harmful aromatic component by the use of nanofibers as adsorbents is discussed in this review, nanofibers are also mentioned here. The first studies on nanofibers date back even before the term "nanotechnology" emerged. A series of patents that Anthon Formhals received starting from 1934 offer a nano-sized fiber production method (7). Although they were discovered so early, the reason why the industrial development rates remained low was the low output amount in the methods. With the academic interest that started in the 1900s, the possibilities of using nanofibers for a wide variety of application areas have been studied by researchers around the world and remained as a hot topic. In this process, besides laboratory scale production methods, industrial scale production methods have also been proposed. The main reason why nanofibers attract so much attention and allow the production of successful products, whether they are produced on a lab-scale or industrial scale, is that, as with all nano-structured materials (0D, 1D, 2D and 3D), the effectiveness of nanofiber products enhances with the increase in the surface area due to their characteristic size. Besides, adsorption is one of the potential uses of nanofibers. The positive effect of surface area on the adsorption process and the advantages of nanofibers with high surface areas are obvious.

The aim of this study is to present the utilization of nanofibers in MB adsorption from textile wastewater. The general concept of nanofibers is first given and the studies in the literature conducted on adsorption of nanofibers are presented in a methodical manner.

2. Nanofibers, Properties and Productions

In textile science, the term "fiber" is used to describe natural or synthetic, thin, and flexible matter that has a high aspect ratio. It is generally defined as the base unit of a textile material. The textile fibers are in the diameter range of 10 to 50 μm in practice (8). If the prefix nano is introduced, the fiber diameters decrease to the submicron level (9).

Low areal density of nanofibers provides advantages in terms of applications where the weight is important such as aerospace, transport, and energy. For a conventional textile fiber with a diameter of 10 μm (10000 nm), consider the weight per unit area as 1. When we reduce the diameter to 100 nm (for the same raw material) the unit area weight becomes 10^4 times less (10). This makes nanofibers indispensable in the production of composites for wind turbine blades, or sound insulation products for aerospace materials, where weight is important.

When diameter decreases to nanoscales bulk atoms become surface atoms and the number of active sites is increased. These active sites make nanofibers more effective in terms of several physical and chemical properties. An increase in the number of active sites (by reducing the unit size at nanoscale and making surface area to volume ratio higher) results in an increase in the probability of bonding or interacting between the unit structures of the material or the media (11, 12). So, adsorption, absorption, filtering, separation, and sensing abilities enhance.

There are various techniques for producing nanofibres. Electrospinning (13), centrifugal spinning (14), template synthesis (15), and self-assembly (16) are some of them. The fiber formation principle in each method is quite different from the others. The fiber structures and morphologies obtained with each method, and sometimes even with modifications made in the same methods, may differ. Finer/coarser and continuous/discontinuous fibers may be got. Figure 3 shows the scanning electron microscope image of the randomly oriented nanofibers obtained by using a stationary plate.

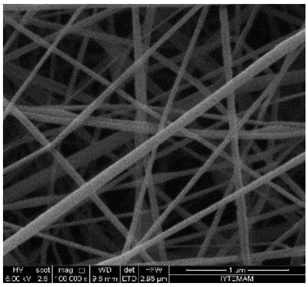

Fig. 3 SEM image of nanofibers (X100K)

Electrospinning utilizes electrostatic field forces acting on the jet that forms nano-structured fibers (17). In centrifugal spinning, air frictional and centrifugal forces act on the jet and thins the polymer solution to form nanofibers (18). A template is used in template synthesis method. It requires the preparation step of a template with nanopores that polymer solution/melt is extruded. The diameter distributions of the nanofibers formed are lesser in this method when compared to others as expected. Self-assembly is a bottom-up method unlike the methods mentioned above (molecular recognition). Self-assembly is a restricted method because not all materials show self-assembling tendency. It occurs by the intermolecular interactions of the functional groups on the polymer backbone.

Nanofibers specific to various applications have been produced by these various techniques and their effectiveness in numerous applications has been investigated by several researchers for years. Since the focus of this study is MB adsorption by nanofibers, the next section deals with the roles and potentials of nanofibers in MB adsorption.

3. Nanofibers as Methylene Blue Adsorbents

Adsorption is the adhering or accumulation of substances in a gas or liquid medium onto the interface of two phases (generally onto solids which they contact) (19). The adsorbent is defined as the mainly solid material that performs the adsorption of dissolved molecules and adsorbate is called the compound that binds/ adheres on the adsorbent. In that case, nanofibers are adsorbents, and MB is the adsorbate that should be removed from wastewater.

Adsorption process is utilized in wastewater treatment for the aim of removing dissolved impurities. Nanofibers are the best adsorbents succeeding activated carbon (20). Numerous studies have been conducted on usage of nanofibers in adsorption. The earlier studies about MB adsorption by nanofibers date back to the 2000s. Afterwards, there has been a high increase in the number of articles about the topic.

Numerous polymers and materials have been used and various nanofiber production methods have been utilized in the literature to obtain the adsorbent nanofibers. Cellulose nanofibers have been utilized in adsorption of MB as in the form of graphene and silica nanocomposites. Two step production process (ionic reaction + sol-gel) has been followed. Pseudo-second-order kinetic model has been observed for the adsorption and maximum adsorption capacity has been found as 625 mg/g. Adsorption capacity has had a slight decrease in four cycles (21). Another group studying with graphene oxide and cellulose is Hussain et al. They have prepared nanofibers by self-assembly technique and have stated that MB dye adsorption followed pseudo-second-order kinetics model with maximum adsorption of 227 mg/g (22). Keratin nanofibers with a surface area of 13.59 m^2/g and with a porosity of 90% have been produced by electrospinning. The initial concentration of MB, contact time with adsorbent, adsorbent dosage, pH, and temperature have been the parameters tested. High adsorption performances have been observed with the keratin nanofibers. Batch adsorptions tests have been performed by preparation of MB solutions in distilled water and sensitively adjusting the parameters tested. Determined amount of adsorbents (hereby keratin nanofibers) have been added to the solutions and final concentrations have been measured and compared with initial ones via a spectrophotometer to calculate the removal of MB (23).

ZnO and TiO_2 were the other materials that attracted attention too much in this area. Adsorption of MB from aqueous solutions has been studied by a group and ZnO nanoparticle containing polystyrene nanofibers and nanofibers composited with a resin have been produced and compared. The ZnO particles have given better adsorption results. Effects of different variables on the adsorption ability have been determined (24). ZnO nanoparticles have been incorporated to carbon nanofibers during electrospinning. Homogeneously distributed ZnO have created micropores on the nanofibers and these pores have had a positive effect of adsorption on MB (25).

TiO_2 has been electrospun with poly (l-lactic acid) to observe the MB removing efficiency. Nanofibers have shown 100% removal capacity when the pollutant concentration has been 100 ppm and adsorbent dosage has been 1 mg/ml (4). In another study, TiO_2 nanofibers have been electrospun with different silver (Ag) concentrations. Thus, photocatalytic decolorization rates have been determined. The rate constants increased with the increase in Ag concentration (26). Besides, TiO_2 nanoparticles have been used together with lignin and poly (ethylene oxide). Electrospun and carbonized nanofibers have resulted in a 91.5% MB adsorption rate with 200 rpm stirring (27).

Polyethylene terephthalate (PET) electrospun nanofibers have been composited with TiO_2 nanoparticles by dipping. %88 of MB has been removed in 10 minutes. It has been emphasized that the effectiveness of TiO_2 incorporated PET nanofibers might be attributed to the more active binding sites formed by TiO_2 on the surface of nanofibers (28).

Various studies have been carried out with modifications such as surface modification, hollow or coaxial nanofiber production, and the adsorption efficiencies of traditional nanofibers have been tried to be increased. Chang and Zeng are the group working on MB adsorption by producing coaxial nanofibers. Electrospinning aided with step-by-step seeding and hydrothermal processes have been selected for production. They have concluded that excellent MB adsorption performances were acquired (29). In Cheng et al.'s study (30) it has been observed that the surface modified nanofibers gave 8.6 times better MB adsorption results than unmodified nanofibers. Their materials have been deacetylated cellulose acetate (DA), and polydopamine (PDA). They have explained that the success of the modified nanofibres might be attributed to the electrostatic and π–π stacking interactions that take place between the nanofibers and MB molecules. Another surface modification has been studied by He et al (31). Poly (vinylidene difluoride) (PVDF) methacrylic acid (MAA) -co- trifluoroethyl acrylate (TFA) was some of the examples. ZnS particles have been loaded on nanofibers by immersing and thermal treating. Three mechanisms have been proposed for the degradation of MB as adsorption, migration and photodegradation. The authors have stated that the effect of surface modification on adsorption was obvious, and they suggested long thermal treatment at 140 °C Hollow TiO_2 nanofibers fabricated by template synthesis method have been in the focus of Jafri et al. (32). Calcination has also applied to the nanofibers. Specific surface area of the nanofibers has been reported as 81.2776 m^2/g. Another hollow nanofiber study has been conducted by Jung et al. TiO_2 hollow nanofibers have been obtained via electrospinning, impregnation and calcination using multiwalled carbon nanotubes (CNTs). SEM, TGA, BET and XRD analyses have been performed. Pure hollow nanofibers and CNTs embedded nanofibers have been compared. It has been stated that the increased performance of the CNTs loaded nanofibers could be related to the high adsorption capability of the CNTs and electron transfer that occurred between the nanofibers and the nanotubes (33). To create porous structures thermal modification has been preferred for ZnO nanofibers (34).

Phytic acid has been a material that has created phosphoric groups on PANI nanofibers and so PANI nanofibers have been improved for MB dye adsorption. Batch adsorption tests have been conducted for determination of MB removal efficiency. 80 mg of nanofibers have been inserted to a 40 ml of MB solution with an agitation of 250 rpm. Pre- and post-process MB concentrations have been recorded by UV-vis spectrophotometer at 664 nm (35). For removing the MB dye within seconds, sulfated cellulose nanofibers have been suggested by Harris and McNeil. The maximum adsorption capacity of nanofibers has been reported as 340 ± 40 mg/g (36).

Activated carbon (AC) is another attractive material in MB adsorption. AC obtained from poly(acrylonitrile) has been examined for removal of MB in an aqueous medium. In that study 72.46 mg/g maximum adsorption capacity has been calculated and pseudo second order kinetic model has been found. In addition, researchers have tested the reusability as 3 cycles (37). Carbonaceous materials (such as graphite and CNTs) incorporated polystyrene-co-acrylonitrile electrospun nanofibers have been investigated in terms of MB adsorption. It has been revealed that the incorporation of carbonaceous materials improved the adsorption ability (38).

Considerably fine boron nitride nanofibers have been produced by a two-step method (freeze drying and post pyrolysis) (20 and 60 nm). The positive impact of nanoscale ultrafast MB adsorption has been observed (39).

Crosslinking of electrospun nanofibers generally makes the nanofibrous structure insoluble in water. Wang et al. (40) studied three types of crosslinking agents and their effect on MB adsorption with sodium alginate (SA) nanofibers. SA nanofibers crosslinked with trifluoroacetic acid have resulted in a minimum adsorption capacity in an acidic medium and SA nanofibers crosslinked with glutaraldehyde have resulted in a maximum adsorption capacity in an alkaline medium. β-cyclodextrin electrospun nanofibers have been thermally crosslinked in another study. BET surface area has been measured as 19.49 m^2/g and the maximum adsorption capacity has been calculated as 826.45 mg/g (41).

Dipping, calcination, and etching methods have been followed to fabricate carbon nanofibers having a surface area of 973.4 m^2/g, and a pore volume of 0.875 cm^3/g. The MB adsorption exhibiting pseudo-second-order kinetic model has been observed with a maximum capacity of 405.23 mg/g (42).

Moreover, some new raw materials have been studied in this field. One of them is the waste biomass. A research group converted waste biomass (horse manure) to carbon nanofibers. Hydrothermal treatment and carbonization methods were applied. Application of an environmentally friendly and cheap material in fabrication of nanofibers for adsorption have been the importance of the study. They have also used Fe to get magnetic carbon nanofibers. Proposed nanofibers have exhibited high performance in adsorption. Since they have been magnetic, they have easily separated from water (43).

Table 1. Some studies about the adsorption of MB via nanofibers

Material(s) used	Production methods	Characterization tests	Adsorption test results	Ref.
Keratin	Electrospinning	Surface area, porosity, SEM, batch adsorption tests	Nf prop: d: 220 nm, ssa: 13.59 m^2/g, p: 90% Kinetic data model: pseudo second-order q: 170 mg/g (at 20 ℃, pH 6, ads. dosage 1 g/l)	(23)
PS, ZnO, resin	Electrospinning	FTIR, SEM, XRD	Nf prop: d: 220 nm Adsorption isotherm model: Langmuir q: 40 mg/g (initial conc: 0.25 mg/l)	(24)
Carbon, ZnO	Electrospinning	SEM, TEM, TGA, XRD	Nf prop: d: 100 nm, ssa: 447.707 m^2/g Kinetic data model: pseudo second-order and Freundlich q: 68.225 mg/g (400 min, 50 °C)	(25)
TiO_2, Ag	Electrospinning	SEM, XRD, FTIR, Raman, UV-vis	Nf prop: d: 52-134 nm Kinetic data model: pseudo first-order q: 90.5%	(26)
PVDF, MAA-co-TFA, ZnS	Electrospinning, Immersing, Thermal treatment	SEM, XRD, FTIR, XPS, surface area	Nf prop: d: 100-300 nm ssa: ~50-70 g/m^2 photocatalytic stability: 140 ℃, 6 h UV irradiation (repeated 10 times), the remaining MB: below 1.0%	(31)
PANI, phytic acid	Radical polymerization	SEM, HRTEM, XRD, FTIR, XPS, Zeta potential, BET surface area, batch release	Nf prop: d: 60 nm, ssa: 33 m^2/g, Kinetic data model: pseudo second-order, Freundlich isotherm q: 43.4 mg/g. (Mb initial conc: 7 mg/l, 60 min, pH: 8.5, 35 °C)	(35)
AC	Electrospinning, Thermal treatment	SEM, EDX, FTIR	Nf prop: d: 240-280 nm, saa: 108 m^2/g Kinetic data model: pseudo second-order, Langmuir isotherm q: 72.46 mg/g	(37)
Boron nitride	Freeze drying, Post pyrolysis	SEM, XRD, FTIR	Nf prop: d: 20-60 nm, ssa: 515 m^2/g q: 107.3 mg/g	(39)
Biomass (Horse manure)	Hydrothermal treatment, Carbonization	SEM, FTIR, XRD, VSM (Vibrating sample magnetometer)	q: 98% (100 mg/l initial MB conc., 15 minutes)	(43)
PVA, starch	Electrospinning, Crosslinking, Thermal treatment	SEM, FTIR, batch adsorption tests	Nf prop: d: 350-450 nm ssa: 25-45 m^2/g Kinetic data model: Langmuir q: 400 mg/g	(45)
MnO_2, cellulose	In situ synthesis	TEM, FTIR, XRD	q: 99.8% (1 g/l adsorbent dosage, 25 mL volume, MB initial conc. 80 mg/l, 2 minutes)	(48)
PAA, SiO_2	Electrospinning, Sol-gel	SEM, TEM, FTIR, BET	Nf prop: d: 245-380 nm, saa: 108 m^2/g Adsorption isotherm model: Langmuir, pseudo second-order q: 437.78 mg/g, (at pH 10, 120 min)	(49)
CA, CS, SWCNT, FE_3O_4, TiO_2	Electrospinning	SEM, TEM, FTIR, XRD, TGA, BET surface area, batch mode adsorption tests	Nf prop: d: 150-340 nm Efficient removal up to 100 mg/l Mb conc, min 15 minutes. Max q: nfs with 1:20 SWCNT/ Fe3O4: TiO2 ratio,	(50)

Nf prop: nanofiber properties, d: diameter, ssa: specific surface area, q: adsorbate amount or ratio,

Some of the research have been conducted not only with MB but also with other hazardous materials that should be adsorbed. Annealing method has been chosen by Li et al. to obtain mesoporous carbon nanofibres. Ferric-nitrilotriacetate has been the precursor produced by hydrothermal method. MB and anionic methylene orange (MO) adsorption have been studied. Both experimental equilibrium data have been fitted to the Langmuir isotherm model (44) In the study of Moradi et al., working with MO and MB, an MB adsorption capacity of 400 mg/g was reported with poly (vinyl alcohol) and starch hydrogel nanofibers (45). Bromophenol blue and Coomassie brilliant blue have been the other materials tested with MB in the same study (46). In addition to MB, reactive black 5 and reactive orange 16 have also been evaluated (47).

While the productions of nanofibers investigated for adsorption are carried out by existing methods, there are also studies on adsorbent nanofibers that are fabricated with new nanofiber production techniques in the literature. In 2014, Wang et al. (48) proposed a method for the production of manganese dioxide/cellulose hybrid nanofibers. The reducing agent has been cellulose and MnO_2 nanosheet formation has been supported by ultralight. By this means, 99.8% of the MB has been removed in two minutes. The list of the studies encountered in the literature is given in Table 1 in summary form.

4. Conclusion

It is aimed to present a review including nanofiber adsorbents used for the removal of MB from wastewater. While the rapid change in technology responds to human needs very quickly, the destruction of nature also occurs rapidly. To minimize this destruction, new technologies are used and studies involving environmentalist approaches are revealed. Studies in the literature show that nanofibers have a high ability to adsorb hazardous compounds like MB. Nanofibers as adsorbents provide diversified advantages in wastewater treatment since they have high surface area to volume ratio, finer unit structure, and increased number of functional groups. These all enhance the efficiency of nanofiber adsorbents and eliminate the adverse impacts made on environment. These unfavorable effects not only affect environment and habitat, but also cause health problems in human beings because of the toxicities of pollutants. Researchers working on nanofibers are clearly interested in the subject and it is thought that the number of published articles on the subject will continue to increase for a while over the years. Moreover, some of the excellent nanofiber adsorbents obtained by setting of different polymers by doping with different active substances have started to be used on industrial scales out of laboratory scales. This review systematically presents various studies that nanofibers may be used effectively in the removal of MB, which is especially abundant in textile wastewater, and emphasizes the importance of nanofibers in this field. In conclusion, it is expected that the study will shed light on the researchers who will work on this subject.

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