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The Effects of Different Concentrations of Nanoclay in Polyvinyl Alcohol (PVA) nanofibres- Polyester Bilayer on Morphological Structure, Mechanical Properties, and Filtration of Direct Red Dye

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ABSTRACT

Nanofibres produced by electrospinning have high porosity, low basis weight, high surface area, and controllable pore size, which can be applied in various applications such as solar cells, supercapacitors, tissue engineering and regenerative medicine, wound dressing, chemical and gas sensing and photocatalysis. However, nanofibres have poor filtration properties, particularly in textile wastewater due to the poor colour adsorption by the fibres. Hence, the study aimed to improve the filtration properties of the nanofibres by developing a bi-layer membrane of polyvinyl alcohol (PVA) nanofibres-polyester woven with different concentrations of nanoclay. In this study, the nanoclay was added to polyvinyl alcohol (PVA) solution, which was electrospun using electrospinning to make PVA/nanoclay with polyester bi-layer. The analysis shows that the addition of nanoclay resulted in good adsorption of direct red dyes since direct dyes are often used in textile industry, indicating an improvement in the filtration properties. The tensile strength and Young's modulus of samples decreased with an increase in the nanoclay concentration due to the fibre becoming stiffer in the bi-layer sample.

1. Introduction

Numerous chemical sectors work with dyes, but the textile industries are responsible for a significant portion of dye consumption and post-process effluent discharge, [1] making up two-thirds of the global dyestuff market due to the wet processing of textiles [2]. In addition, due to the widespread discharge of wastewater with high dye concentrations, the textile dying industry has become one of the primary sources of environmental contamination [3]. Dissolved particles, colour, toxic metals (chromium), printing gums (pentachlorophenol), detergents, sequestering agents

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(trisodium polyphosphate and sodium hexametaphosphate, chlorine, azo dyes), and stain removers are all present in the water discharged after the fabric preparation (CCl_4, residual chlorine, fixing agents like formaldehyde and benzidine) [1]. The wastewater discharge contains colourants, which are detrimental to humans, animals, and the environment [3]. Dye effluents in water sources are unacceptable since living creatures need water for daily activities, including drinking, cooking, bathing, and washing [4]. The decreasing water quality impacts human health and has drawn worldwide concern [5]. Therefore, removing harmful materials from industrial wastewater, such as textile waste products, is essential to protect the environment. Several conventional methods for removing contaminants from wastewater include chemical coagulation, oxidation, and flotation [6].

As compared to the conventional methods mentioned above, filtering using a membrane to separate dispersed particles from the liquid is possible more simply and efficiently [6]. Electrospinning has gained popularity for the past two decades due to its ability to produce the micro or nanoscale flexible nanofibre membrane from synthetic or natural polymers [7]. Nanofibre-based membranes have many benefits, such as high porosity, low basis weight, high surface area, controllable pore size, submicron pore size, and continuous-interconnected pores [5]. These characters draw interest and become an attractive choice for advanced application because it has a large surface collection area and low air resistance [8]. Although it has many advantages, the nanofibre has weak mechanical properties. It is unsuitable for filtering coarse particles due to its ultrafine size, which causes structural damage to the fibres [9]. Electrospinning is a unique and simple way to produce nanofibre structures [10]. It uses a high voltage field or an electrostatic force to create ultrafine nanofibres from the liquid polymer or melt [10]. Electrospinning operates using the concept of electrostatic that imparts toward polymer solution to produce fine fibres with the range of 100 – 500 nm. According to Fazal *et al.,* [11], the electrospinning process happens when the polymer solution is heated to a specific temperature and forced to form a droplet at the tip of the syringe. Then, the high voltage is applied to the polymer solution to help the polymer acceleration on the surface of the collector to form nanofibres [12]. This method recently gained widespread popularity in manufacturing a continuous nano-scale fibre due to its versatility, which gives it the potential for application in various fields [13, 14].

Nanofibres also exhibit excellent filtration of finer particles [15], but they have a drawback of a lower flux as an accumulation of solid start blocks the surface [16]. Typical membrane filtration, particularly pressure-driven liquid filtration requires mechanical strength and chemical and thermal stability since it will often expose to a broad range of temperature, humidity, hydraulic pressure, mechanical vibration, and abrasive particulates in the fluid flow [17]. Due to this, a bi-layer structure is needed since, in the previous study, the bi-layer of nylon 6 nanofibres/cellulose membrane with nylon 6 nanofibrous/PET nonwoven membrane shows improvement in flux, mechanical strength, and high rejection of polystyrene latex (PSL) [5]. To produce water filtration using a bi-layer membrane, it is advisable to have hydrophilic and hydrophobic components or layers, as each can complement the other. Hydrophilic membrane surface has been usually favoured due to its advantages, such as it helps to reduce protein adhesion and bio-fouling in micro and ultrafiltration, improving the wettability of porous support material, and improving water flux [17]. However, it inclines to plasticize in an aqueous environment and swell, which increases the fibre size and changes membrane dimensions, which can lead to a reduction in fibre strength in the swollen state [17]. Hydrophobic, however, does not swell but is likely to absorb foulants [17].

Emerging science known as nanotechnology works with materials that are nanoscale in size. The unique structural, chemical, mechanical, magnetic, electrical, and biological characteristics of nanoparticles are of tremendous interest and are applied across all scientific disciplines [18]. While the use of nanoparticles dates back a century, they have lately acquired appeal due to their simplicity,

environmental friendliness, lack of toxicity, and low cost for wastewater treatment applications [19]. Nanoclays are nanoparticles of layered mineral silicates with layered structural units that can form complex clay crystallites by stacking these layers [20]. An individual layer unit is composed of octahedral and/or tetrahedral sheets. Polymer/clay composites can deliver a high adsorption capacity and an excellent life cycle for water treatment/remediation due to their easy processability, effective cation exchange, large surface area, and relatively low cost and toxicity [21]. By introducing nanoclay into the nanofibre, we can add the nanoclay's advantages to the nanofibre for filtration purposes.

This study aimed to develop a bi-layer polyvinyl alcohol (PVA) membrane incorporating different concentrations of nanoclay with a polyester woven. Through this development, the effects of different concentrations of nanoclay on the nanofibre morphological structure and its filtration of direct red dyes were studied. introduction of nanoclay to nanofibre bi-layer membranes can improve the direct red dye filtration process. The nanoclay bi layer PVA composite can be used as an alternative way of treating textile wastewater produced by the textile industry will be developed.

2. Methodology

2.1 Materials

Woven polyester fabric, and polyvinyl alcohol (PVA) pallets were purchased from Sigma Aldrich, nanoclay and distilled water were used in the experiments.

2.2 Preparation of PVA and PVA/Nanoclay Spinning Dope

To prepare PVA spinning dope, 10 wt% of PVA pallets were dissolved in distilled water with a hot plate and magnetic stirrer at a temperature of 80 °C for 1 hour. The PVA/nanoclay spinning dope was prepared by dissolving 10 wt% polyvinyl alcohol (PVA) pallets from Sigma Aldrich and different concentrations of nanoclay (0.25 wt%, 0.5 wt%, and 0.75 wt%) in distilled water using a hot plate and was constantly stirred using a magnetic stirrer at the temperature of 80 °C for 1 hour.

2.3 Electrospinning

The nanofibrous bilayer was prepared using an electrospinning machine Spinbox equipped with a collector plate. The collector was covered with polyester woven fabric to collect the PVA or PVA/nanoclay nanofibrous mat. The process parameter for the electrospinning machine for the PVA was an acceleration voltage of 12 kV, flowrate 0.15 ml/hr, a spinning distance of 16 cm, and was spun for 10 minutes. For the PVA/nanoclay (0.25 wt%, 0.5 wt%, and 0.75 wt%), the process parameter was acceleration voltage 16 kV, flowrate 0.25 ml/hr, spinning distance 16 cm, and was spun for 10 minutes. The produced membranes were named as Table 1:

Table 1
Name of membranes

Membrane	Name of membrane
PVA nanofiber with woven polyester	PVA-NF
PVA/nanoclay (0.25 %wt) with woven polyester	PVA-NC-NF-1
PVA/nanoclay (0.5 %wt) with woven polyester	PVA-NC-NF-2
PVA/nanoclay (0.75 %wt) with woven polyester	PVA-NC-NF-3

2.4 Scanning Electron Microscopy

The morphology of PVA nanofibre with PVA and PVA/nanoclay (with different concentrations) nanofibre with PVA were examined using a scanning electron microscope (Phenom Microscope, FEI Company, Hillsboro, OR USA) at a voltage of 1 kV. The surface of the samples was coated with argon gas.

2.5 Filtration Test and Spectrophotometer

The filtration test was used to determine the effectiveness of samples in filtering direct red dyes. 0.1 g/L direct red dye solution was filtered using filter glass and bi-layer membrane samples. After filtration, the filtrate was put right into a spectrophotometer cell. On a spectrophotometer (Zuzi 4251/50), the dye concentration was determined for direct red dye with wavelength 535 nm. The methodology is somewhat similar to that of a previous study by M. Pasichnyk *et al.,* [22], except for the type of filtrate used.

2.6 Mechanical Properties

The tensile test was conducted based on ISO 13934-1, with minor modifications. All the bilayer samples were tested using a universal testing machine (Zwick/Roell) with an extension rate of 10 mm/min at room temperature. The samples were 100 mm long and 25 mm wide, and the distance between the two clamps was 50 mm. Both warp direction and weft direction were tested. Three measurements were taken for each sample.

3. Results

3.1 Membrane Fibre Size and Morphology

The polyester's morphological structures of electrospun PVA and PVA-NC-NF (1-3) were observed using SEM images, shown in Figure 1. The electrospun PVA-NF had more cylindrical surfaces than those incorporated with nanoclay. This is consistent with the results reported by Liu *et al.,* [23]. By incorporating nanoclay in nanofibre, the fibres appeared to have beads and fibre swelling. Fibre swelling and fibre irregularities continued to increase with an increase in nanoclay concentration from 0.25 wt% to 0.75 wt%. Due to the fibre swelling and irregularities, the fibres were measured for diameter, and the results are presented in Figure 2 and Table 2. It shows that by increasing the amount of nanoclay from 0.25 wt% to 0.75 wt% in the PVA solution, the fibre diameter increased to 11 %. The sample with the largest diameter is the PVA-NC-NF-3 (233 ± 82.2 nm), while the smallest is the PVA-NF (186 ± 47.63 nm). This is due to the addition of nanoclay to the PVA solution. Increasing solution viscosity and conductivity causes the average fibre diameter to increase by adding nanoclay [23,24]. In addition, the parameters of the PVA NF are different from the PVA introduced with nanoclay at different concentrations.

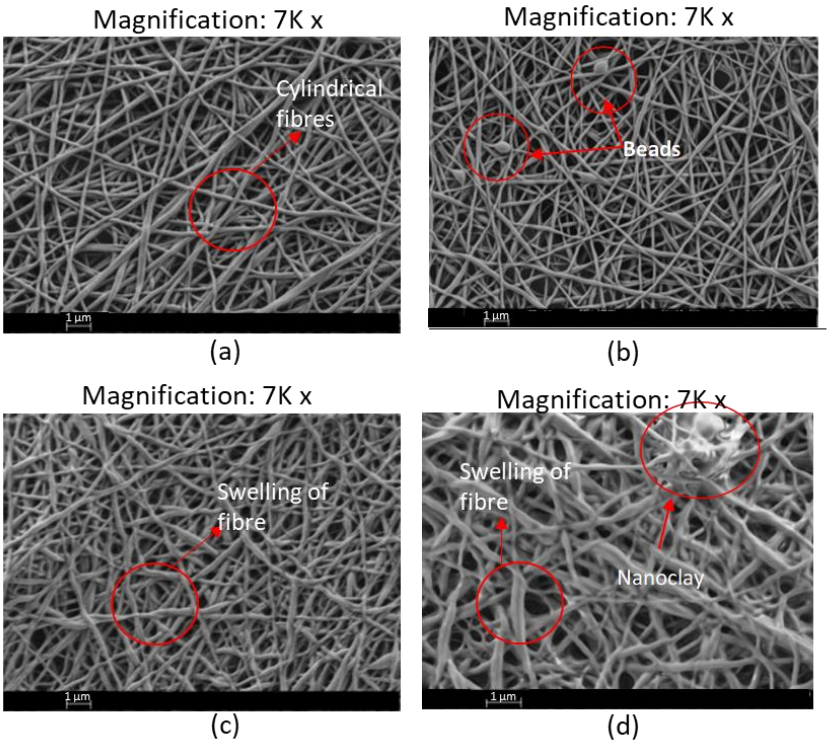

Fig. 1. SEM images of (a) PVA-NF, (b) PVA-NC-NF-1, (c) PVA-NC-NF-2, and (d) PVA-NC-NF-3

Table 2
The nanofibre size

Sample	Nanofibre size (nm)
PVA-NF	186±47.6
PVA-NC-NF-1	206±56.8
PVA-NC-NF-2	220±34.9
PVA-NC-NF-3	233±82.2

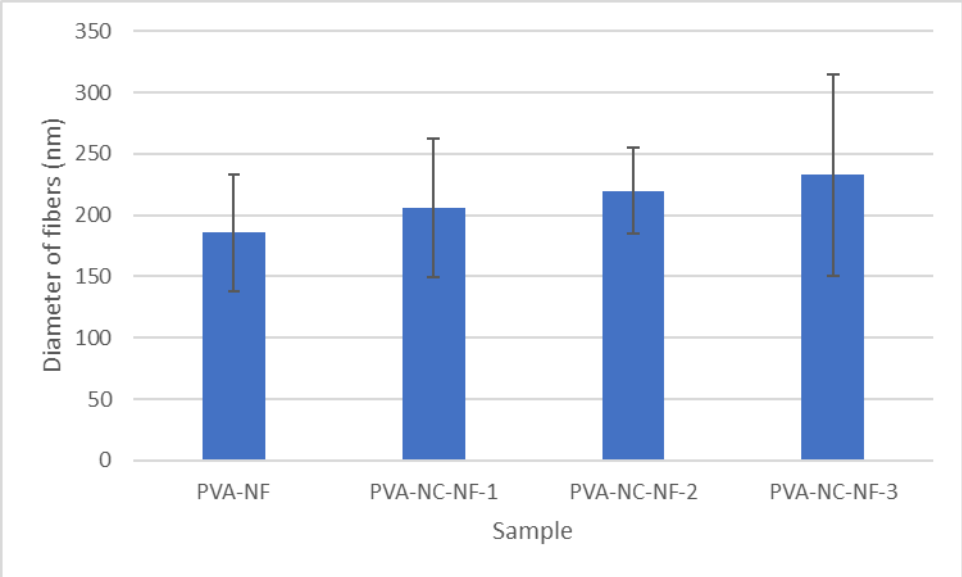

Fig. 2. Diameter of PVA-NF, and PVA with different nanoclay concentrations (PVA-NC-NF-1, PVA-NC-NF-2, and PVA-NC-NF-3)

3.2 Spectrophotometer Analysis

The condition of samples after filtration of direct red dye is shown in Figure 3. It shows the nanofibres on polyester fabric rupture after filtration of direct red dye solution (shown with red arrows). This is due to the low mechanical properties of PVA-NF, which was also observed in a previous study [9]. It is also shown that the filter glass membrane absorbed the red dyes as the colour of the membrane is much more concentrated than that of polyester fabric. This is due to the hydrophobicity nature of polyester.

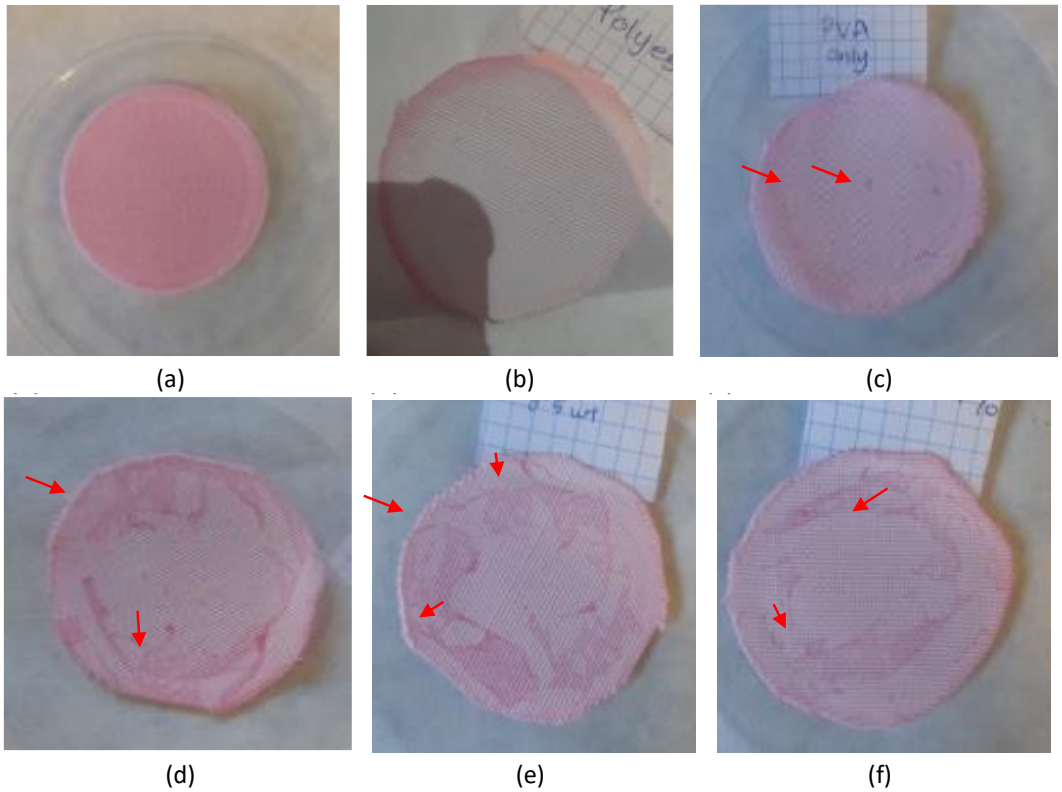

Fig. 3. The conditions of samples (a) filter glass membrane, (b) polyester fabric, (c) PVA NF, (d) PVA-NC-NF-1 (e) PVA-NC-NF-2, and (f) PVA-NC-NF-3 after filtration of direct red dye solution

Spectrophotometry is a method to measure how much a chemical substance absorbs light by measuring the intensity of light as a beam of light passes through a sample solution. In this case, the spectrophotometry measures the absorbance of light of filtrate direct red dye solution. As illustrated in Table 3 and Figure 4, the absorbency of filtrated relatively decreases with the increasing concentration of nanoclay in the bilayer membrane. Hence, this indicates a decrease in the concentration of direct red dye in the solution. The filtrate that has the highest dye concentration is the filter glass membrane (0.113 mg/L), while the lowest is the filtrate of PVA-NC-NF-3 with polyester fabric (0.0463 mg/L). The gap dye concentration between the unfiltered and the filtrate is 59 %. This is because nanoclay has an effective cation exchange and a large surface area [21]. The increment of dye adsorption with increasing nanoclay is parallel with the study conducted by Dalaran *et al.,* [25], where they stated that by increasing the nanoclay concentration, the adsorption capacity of acidic dye increases.

Table 3
Absorbency and concentration of filtrated

Sample name	Absorbency	Concentration (mg/L)
Not filtered	2.1260	0.1130
Filter glass membrane	2.0510	0.1090
Polyester	1.9518	0.1037
PVA-NF	1.9086	0.1015
PVA-NC-NF-1	1.1372	0.0605
PVA-NC-NF-2	1.4116	0.0750
PVA-NC-NF-3	0.8702	0.0463

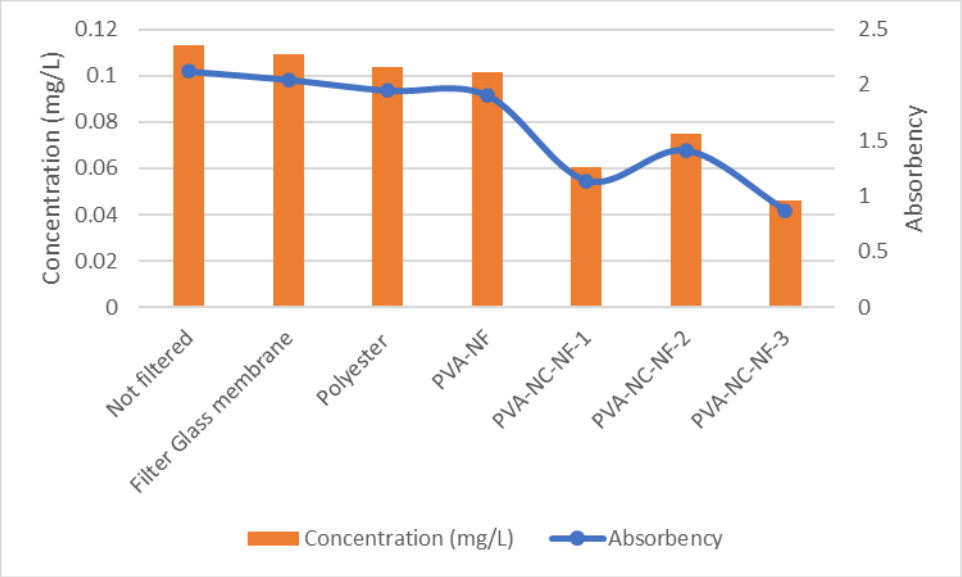

Fig. 4. Absorbency and concentration of filtrate direct red dyes

3.3 The Mechanical Properties of The Bilayer Membrane

Table 4 tabulates the tensile strength and Young's modulus of samples in the warp and weft direction. In the warp direction, the tensile strength of the samples relatively decreased with an increase in the nanoclay concentration. This shows that the concentration of nanoclay did influence the tensile strength of the bi-layer PVA-NC-NF samples, particularly in the warp direction of the polyester. In addition, the authors also noticed a high tensile strength of PVA-NC-NF-2. For Young's modulus, the PVA-NF has a high Young's modulus, approximately 41.41 % higher than the polyester fabric. However, an increase of the nanoclay in the PVA reduced Young's modulus of the sample. This indicates that the application of nanoclay in the PVA influenced the stiffness of the sample.

For the weft direction of the polyester, the highest tensile strength of bi-layer samples is PVA-NC-NF-1, which is about 3.3539 N/cm^2, while the lowest is PVA-NC-NF-2 was approximately 3.1224 N/cm^2. An increase in the nanoclay concentration from 0.25 wt% to 0.75 wt% reduced Young's modulus of the sample by as much as 26 %. The nanoclay was expected to affect the stiffness of the bi-layer samples

Table 4
Tensile strength and young's modulus of samples at warp and weft direction

Sample	Tensile Strength (N/cm^2)		Young's Modulus (N/m^2)	
	warp	weft	warp	weft
Polyester Fabric	6.7660	3.3525	1.3288	0.8212
PVA-NF	6.1279	3.3288	2.2696	2.3655
PVA-NC-NF-1	5.5390	3.3539	1.5722	1.8786
PVA-NC-NF-2	6.8528	3.1224	1.7022	1.0795
PVA-NC-NF-3	5.1731	3.2394	1.5547	1.3923

4. Conclusions

In conclusion, the development of PVA/nanoclay with polyester fabric was achieved in the study. The surface morphology of fibre in the bi-layer membranes became swollen and had several irregularities with increased nanoclay in the fibre. The mechanical strength of the bilayer membrane at both the warp and weft directions was relatively decreased with increasing concentration of nanoclay, probably due to the fibre becoming stiffer in the bi-layer sample. The current study also shows that the PVA-NC-NF could filter direct red during the filtration process.

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