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Physical Properties of PA6/PVA Blend Nanofibers via Electrospinning Technique

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Polyamide (PA) and polyvinyl alcohol (PVA) electrospun nanofibers for the removal of contaminated water with waste oils, were created in this study. In order to develop the nanofibers, PVA was selected as the substrate after being dissolved in formic acid with varying polymer concentration. In varying ratios of polyamide to polyvinyl alcohol, and varying electrospinning parameters of the polyvinyl alcohol and formic acid solution, the following were used: higher voltage (20 kV), pump distance (15 cm), and pump rate (1 ml/hour) were selected. Several solution parameters were measured and included viscosity, and surface tension and electrical conductivity. The results from scanning electron microscopy (SEM) and the differential scanning calorimetry (DSC) showed with increased concentrations of polyamide in the mixture, viscosity was decreases, and electrical conductivity of the solution was increased, which also resulted in a change in surface tension according to the results of the SEM. Increased electrical conductivity with decreased viscosity resulted in a decreased fiber diameter.

Keywords: Electrospinning; Nanofibers; Physical properties; Thermal behavior
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1. Introduction

Regarding the developments in modern society, nanotechnology is a major industrial revolution. Most medical, industrial, environmental, food, agricultural, sustainable development, and other industries use nanotechnology [1-5]. Nanotechnology, the lack of clean and fresh water is one of the most common problems facing the world, as 1.2 billion people in the world suffer from a lack of access to clean water or a shortage of it, and thousands of people become ill and die due to diseases transmitted through contaminated water [6,7]. Also, the development that occurred in the oil and gas industries led to pollution in wastewater. There are many traditional methods for purifying water as well as modern methods. Traditional methods include physical, chemical, and biological methods [8]. Modern methods include membrane filtration technology, which includes reverse osmosis, microfiltration, ultrafiltration, and nanofiltration [9-11]. Nanofiltration is one of the most efficient modern methods due to its very small porosity from the high surface area to volume ratio. It is also of great quality and ease of transition in manufacturing. The ability to operate membranes at ambient temperatures suitable for biologic and pharmaceutical applications [12]. There are many previous studies that have been conducted using the electrospinning technique for producing the nano-filters for cleaning the water.

In 2023, Darwish et al. [13] published a paper about using the electro spun PA/PVA nanofibers reinforced with CNPs for the filtration process. Some tests on the resultant filters were performed, including DSC, SEM, and AFM. According to the DSC data, adding CNPs to PA66 leads to an increase in T_g, a decrease in Tm, and an increase in T decomposition. In addition to that, the AFM results of electro spun filters proved that the electrospinning process produced a uniform porosity. As well as, the result also proved that the increasing CNP concentration leads to an increase in the bearing index of the surface. According to the SEM images, the electrospinning process generated uniformly shaped nanofibers with an average diameter of roughly 350 nm, which shrank as the concentration of CNP increased [13].

In 2024, a study presented by Rui Xin et al. [14] involved using PA-manufactured ultrafine nanofiltration membranes. He used dynamic adsorption and filtration efficiency of polyamide membranes by electrospinning to remove nanoparticles and 0.2-micron particles from wastewater. The morphology of nanoscale surfaces and fiber diameters varied with the concentration of polyamide solutions. The results of specific mechanical properties showed that at a concentration of 11% polyamide, the adsorption capacity of the membranes reached 204.5 and 133.3 mg/L, respectively, against cationic dyes (e.g., chrysoidine) and anionic dyes (e.g., metanil yellow), with the membrane rejection rate reaching 100%. Selective filtration was performed based on hydrogen bonding, hydrophobic interaction, and electrostatic interaction. A multi-adsorption separation was proposed based on hydrogen bonding, electrostatic interaction, and hydrophobic interaction, in addition to deep filtration [14].

2. Materials and Methods

Polyamide with 15,000 Mw as a weight powder and a weight powder of PVA with 20,000 Mw were used to prepare the nanofiber textile. Formic acid, with the chemical formula HCOOH, with a melting point of 84°C and a boiling point of 100.8°C, was used as a solvent for the PA/PVA blend. Table (1) shows the contents of the prepared samples.

Table (1) The contents of the prepared samples

Sample	Contents	PA6/PVA ratio
SA1	Pure PA6	100:00
SA2	Pure PVA	00:100
SA3	PA6/PVA	50:50
SA4	PA6/PVA	70:30
SA5	PA6/PVA	90:10

The electrospinning technique was used for preparing the nanofibers of PA/PVA blends, as shown in Fig. (1). Electrospinning solutions were determined by dissolving 1.5 g of PA6 in 10 ml of formic acid and 1.2 g of PVA in 10 ml of formic acid, each separately, and then they were mixed in different ratios, with the highest ratio being PA6 (50:50, 70:30, 90:10). They were then pumped with electrospinning for five hours under humidity and temperature at the following intervals: (28°C, 11°C, 18°C, 25°C, 18°C) (73, 81, 77, 76, 77).

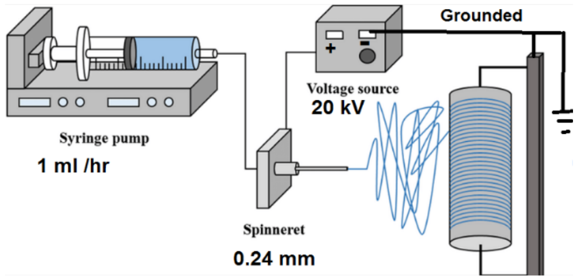


Fig. (1) Electrospinning setup

3. Results and discussion

As noted in table (2), the results of measuring the physical properties of PA/PVA polymer solutions show clear differences related to the mixing ratios of the two materials. The PVA solution recorded a higher viscosity value of 365 cp. This is attributed to the molecular structure of PVA, which depends on the density of molecular cross-links and strong hydrogen bonds, thus increasing viscosity [15]. According to my results, the best example was a 50:50 blend of polyamide (PA) and polyvinyl alcohol (PVA), where the fibers demonstrated structural uniformity, thermal stability, and a bubble-free structure. Its dense hydrogen lattice makes its viscosity very high. However, polyamide and samples with high polyamide content exhibited the opposite behavior in electrical conductivity, having the highest conductivity at 280 cp, compared to 270 cp for the 90:10 sample, and 150 cp for PVA. This is because polyamide contains numerous functional groups that increase the concentration of ion mobility within the solution, which corresponds to Huang's position that amide or carboxyl polymers possess higher conductivity due to the unhindered movement of ions [16].

Table (2) The properties of solutions prepared in this study

Sample	Surface tension	E.C. (μS/cm)	Viscosity (cp)
SA1	20.526	280	61.66
SA2	23.46	150	365.17
SA3	20.44	250	105.2
SA4	19.5	240	89.3
SA5	23.6	270	80.38

The surface tension ranges between 23.6 and 19.5. It was proven that pure PVA has the highest surface tension while samples that contain high percentage of PA exhibit lower surface tension because PA reduces the intermolecular attraction of surface molecules. This makes jet drawing during the electrospinning process much more stable and easier. Previous research suggests that lower surface tension is correlated with less drawing defects and more stable jets [17].

Table (3) Diameters of nanofibers

Sample	Contents	Diameter (nm)
SA1	Pure PA6	111
SA2	Pure PVA	152
SA3	PA6/PVA 50:50	152.36
SA4	PA6/PVA 70:30	140
SA5	PA6/PVA 90:10	120

Figure (2) shows the FTIR analysis of PA, PVA, and a blend of them with a 50:50 volume ratio. The main pattern of PA shows sharp peaks at 3347.18 and 3417.88 cm^{-1}, indicating the OH stretch, and at 1635.64 cm^{-1}, the C=OO bond. The PVA curve shows broad, strong peaks at 3417.8 and 3434 cm^{-1} due to the hydrogen bond. When the two materials are mixed, the same peaks appear clearly at 3417.80 and 3479.8 cm^{-1} due to the interaction of the H bond, and also at 1620 cm^{-1}, which is the C=O stretch bond.

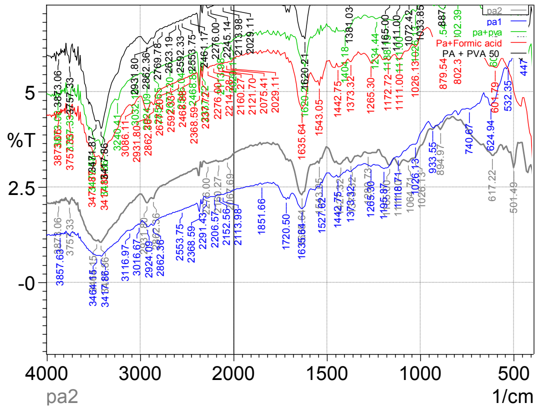
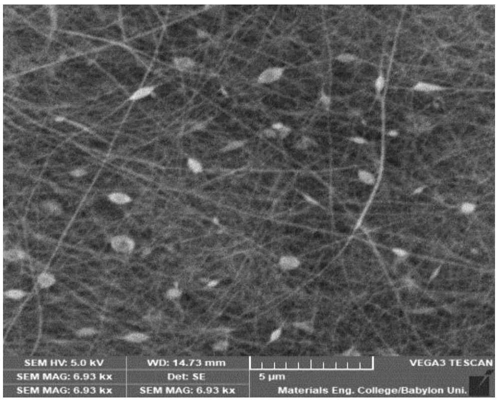


Fig. (2) FTIR analysis of samples

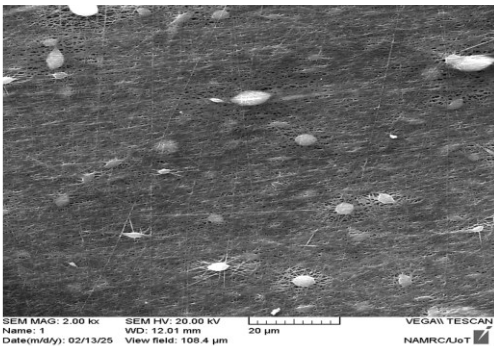
Using ImageJ software, fiber diameters were measured from SEM images, where 20 organic measurements were taken from different areas (SEM) with the model (VEGA 3 SBU). There is a close relationship between fiber diameter and solution properties. PVA is characterized by lower conductivity

(152 nm) and higher viscosity. This means that high viscosity reduces the elongation capacity of the extruder [18], which results in SA3 and SA4 samples with thicker diameters, with the diameter reduced to 140-152 nm due to the decreased viscosity of the resulting solution. This, in turn, enhances surface tension, which affects the extruder, leading to the production of more uniform and finer fibers. This is evident in the SA5 sample, which exhibited high conductivity and a viscosity of 80 nm. This was evident in the SA5 sample, which recorded a viscosity of 80 nm and the highest conductivity, yet had the smallest diameter. This is attributed to increased charge density combined with high conductivity, in addition to the ease of elongation of a low-viscosity solution. These factors contribute to reducing the fiber diameter [19]. Pure PA sample, on the other hand, showed a small diameter of 111 nm, resulting from a remarkable balance between high conductivity and medium viscosity, making the jet stable and easily stretchable. Furthermore, the low surface tension in SA3 and SA4 samples contributed to the production of finer fibers. Uniformity and fewer defects (i.e., granules)

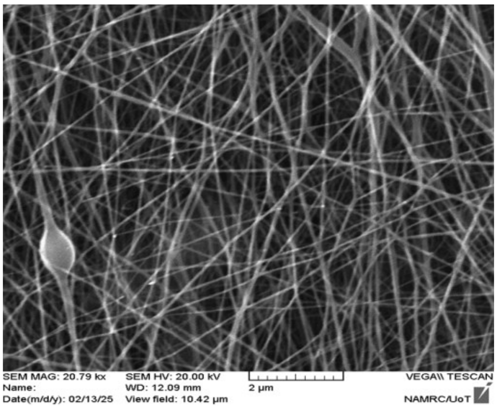
Figure (4) shows the contact angles of pure PVA (48.7°), pure PA6 (54°), PA50:PVA50 (58°), PA70:PVA30 (55°), and PA90:PVA10 (47°). As can be seen, all of the resulting nanofibers demonstrate hydrophilic behavior due to the contact angle having less than 90 o, as well as the PVA and PA6 structures containing OH groups. The appearance of OH groups in the structure of polymers leads to the interaction between OH groups and water, which leads to hydrophilic behavior [20,21]. On the other hand, the blending of PA6 and PVA leads to an increase in the contact angle of pure PVA from 48° to 55° and 58° with increasing blending ratios from 30 wt.% to 50 wt.% respectively, this is may be due to an interaction between the OH groups of PA6 and PVA was happened, in addition, it leads to a decrease in the number of OH groups through the resulting blend nanofibers and an increase in its contact angle [20-22].



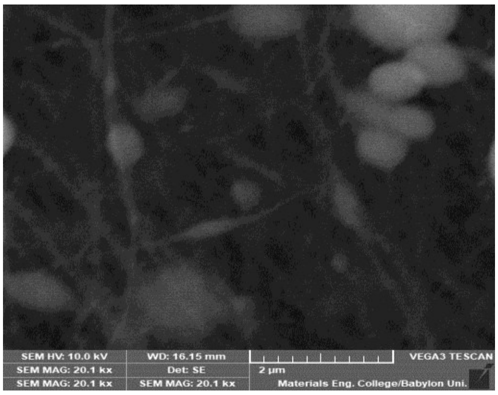
(a) PA nanofibers



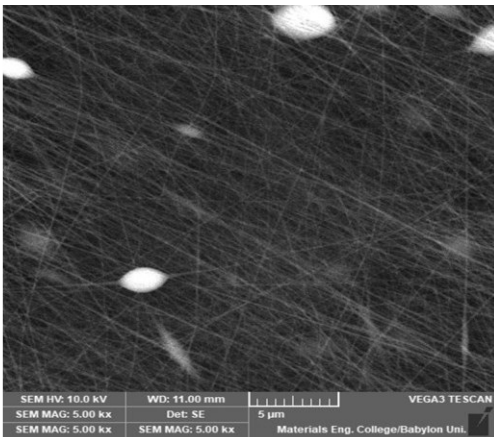
(b) PVA nanofibers



(c) 50:50 PA6:PVA blend nanofiber

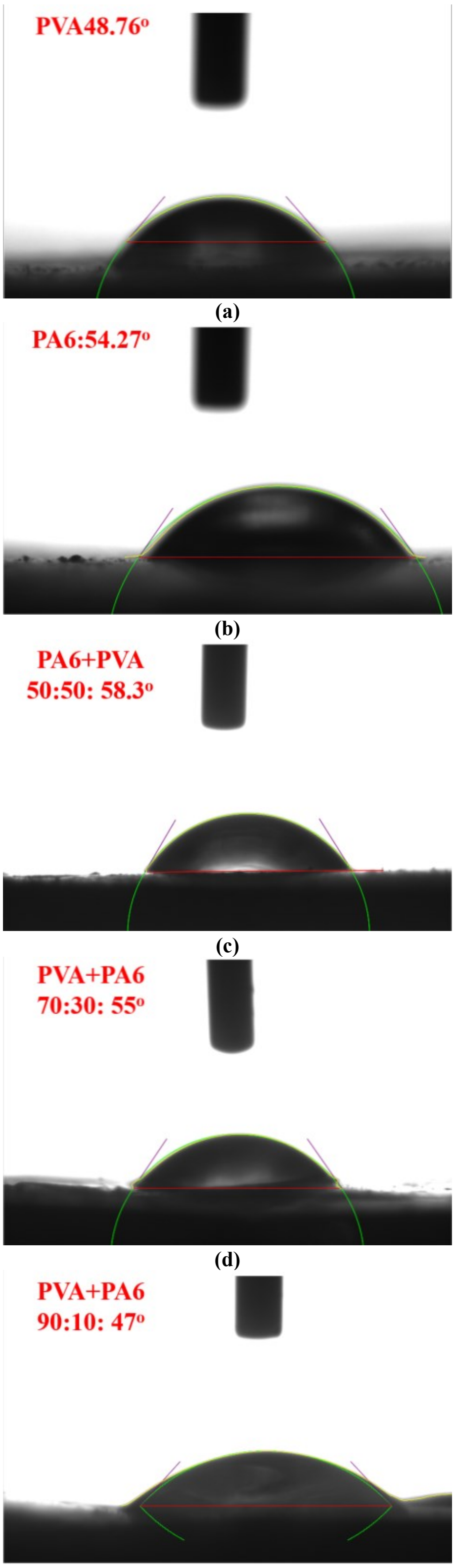


(d) 70:30 ratio of PA6:PVA blend nanofibers



(e) 90:10 PA6: PVA blend nanofiber

Fig. (3) SEM images of the samples prepared in this work



(a)

(b)

(c)

(d)

(e)

Fig. (4) The contact angle of the prepared nanofibers, PA6, PVA, and PA6:PVA with ratios of 50:50, 70:30, and 90:10

Figure (6) shows the DSC curves of nanofiber samples, including pure PA, pure PVA, PVA50:PA50, PVA30:PA70, and PVA10:PA90. A SHIMADZU DSC-60 differential scanning calorimeter has a temperature range of 20°-300°C, a flow rate of 30 ml/min, and a heating rate of 10°C.

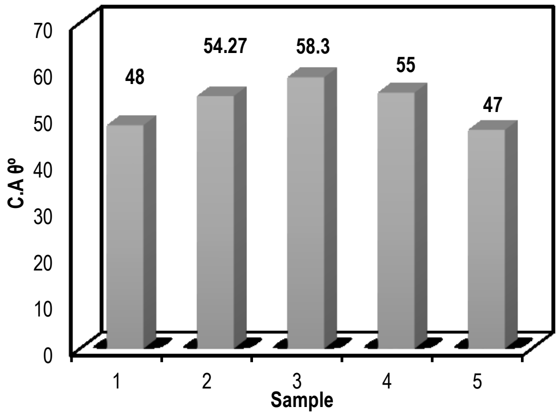
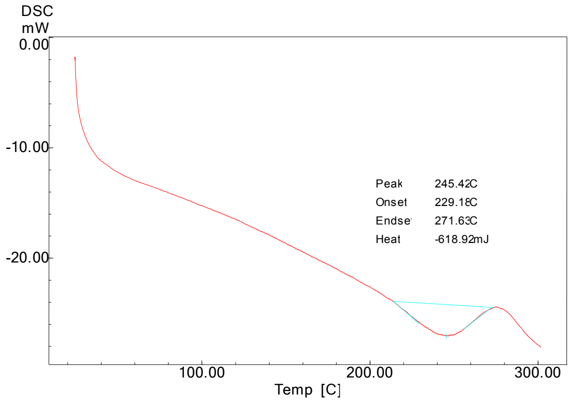


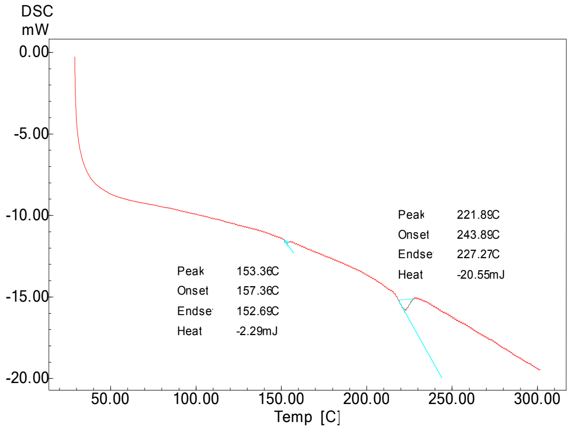
Fig. (5) Contact angle of all nanofiber samples

Table (4) Thermal analysis by DSC

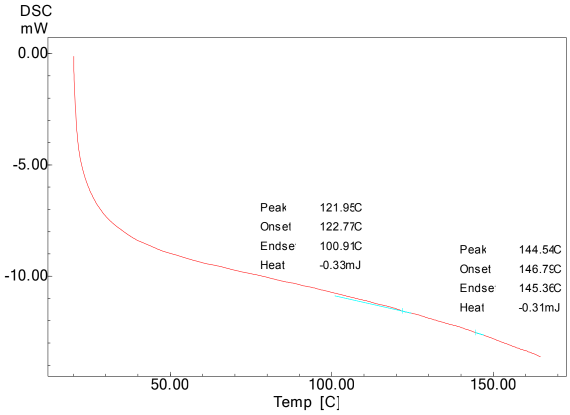
Sample	Tg (°C)	Tm (°C)
Pure PVA	65	245
Pure PA6	153	221
PA6/PVA50:50	87	221.8
PA6/PVA 70:30	121	221.12



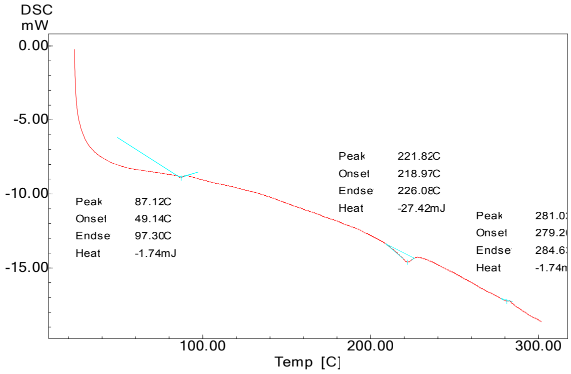
(a) Pure PVA



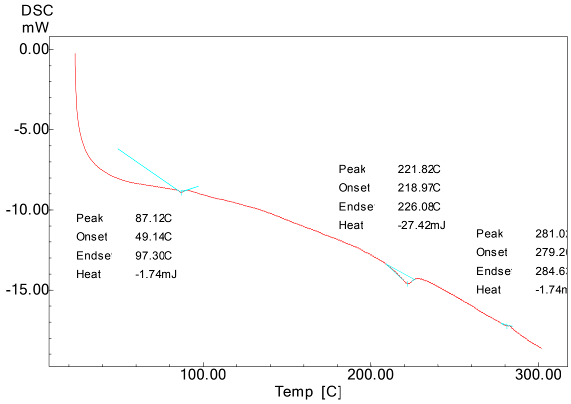
(b) Pure PA6



(c) PA6 30:PVA 70



(d) PA6 50:PVA 50



(e) PA6 90:PVA 10

Fig. (6) DSC curves of all the nanofiber samples

Thermal analysis using differential scanning calorimetry (DSC) revealed the distinctive thermal behavior of the electrically spun PA/PVA fibers, reflecting the compatibility between the two polymers. Pure PVA exhibited a low glass transition temperature (T_g) of 65 and a melting point of 245°C due to its high thermal flexibility and semi-crystalline structure. PA, on the other hand, showed a high glass transition temperature (T_g) of 135°C due to strong hydrogen bonds between its chains and a melting point of 221°C, indicating stable crystallinity. Changes in the glass transition temperatures were observed when the two polymers were blended. The T_g decreased from 50:50 to 87°C, indicating that PVA acts as a plasticizer for the PA chains, increasing their mobility. With an increase in the PA content to 70%, the T_g rose to 121°C. This increase is attributed to the influence of hydrogen bonds, with the PA group becoming dominant in the structure. The melting point remained close to 221°C. The melting point of pure PA indicates that the areas affected by fiber crystallization are PA with a small amount of PVA present, as is found in SA5 [23,24].

Conclusions

The current study concludes that adding polyvinyl alcohol to polyamide increases the spinnability of polyamide in the electrospinning system, resulting in the creation of fibers with superior morphology. Infrared analysis showed an important connection between polyvinyl alcohol and polyamide via the OH group. Furthermore, the addition of polyvinyl alcohol to polyamide acts as a plasticizer, resulting in a certain percentage drop in thermal characteristics. Furthermore, the contact angle remained within the hydrophilic range of materials, with minor variations in the results.

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