Synthesis of Fibers and Particles from Polyvinyl Chloride (PVC) Waste Using Electrospinning

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Abstract. Synthesis of fibers from the waste of polyvinyl chloride (PVC) has been successfully done using electrospinning method. The PVC solutions were made with the solvents N, N dimethylformamide (DMF), tetrahydrofuran (THF) and dimethylacetamide (DMAc). The effects of PVC concentration on the morphology and the diameter of fibers were observed. The morphological change from particles to fibers took place along with the increasing concentration of PVC in DMF, DMAc, and THF. When DMF was used as the solvent, the average particle diameter was 2.91 μm for the concentration of 5 wt%. As the concentration was increased to 10 wt%, 15 wt%, and 20 wt%, fibers were obtained, and their mean diameters were 1.12, 1.47, and 1.63 μm, respectively. When DMAc was used, the average particle made of the precursor solutions of concentrations of 5 wt%, 10 wt%, and 15 wt% were 2.79 μm, 3.03 μm, and 3.31 μm, respectively. If the concentration was further increased to 20 wt%, then fibers were formed with the average diameter of 1.35 μm. Finally, with the use of THF solvent, the average particle diameters were 6.83 μm and 8.38 μm for the solution concentrations of 5 wt% and 10 wt%, respectively. When the concentrations were 15 wt% and 20 wt%, fibers were finally achieved with the average diameter of 1.38 μm and 2.83 μm, respectively. It was clearly found that there is a PVC critical concentration for a morphological transition from PVC particles to fibers.

Keyword. Electrospinning, fibers, and polyvinyl chloride (PVC)

1. Introduction
The development of nanotechnology has grown tremendously in recent years and has produced many applications in various fields. One of the most interesting developments in nanotechnology, due to its vast applications, is nanofiber. Nanofiber is a one-dimensional material whose form is like fine threads with a diameter of one to several hundred nanometers. Nanofiber is proven to possess many advantages such as having a large ratio of surface area over volume, high flexibility, tensile strength, and porosity, which can then be utilized in many applications [1], [2]. Some of the areas where nanofiber has been extensively researched as drug delivery medium, sensors, catalysts, filters, protective clothing, etc. [3]–[6]
One of the techniques to produce nanofibers is electrospinning. A high-voltage source, a collector, a syringe pump, and a nozzle are the major components of this technique [6]. This technique utilizes electrostatic forces to form fibers, and it has the ability to produce fibers with different characteristics. The characteristics of resulting fibers can be controlled by adjusting the electrospinning parameters such as concentration of the solution, viscosity, charge density, net charge, charge neutralization, surface tension, flowrate and voltage [4], [7].

Polyvinyl chloride (PVC) is one of the most commonly used thermoplastic materials and is the second most produced thermoplastic after polyethylene. Due to its physical and chemical advantage as well as its anti-degradable properties and low production costs, PVC is widely applied as pipes, packaging materials, window frames, cables, bottles, and even in medical devices [8]–[10]. The flexibility of the PVC application then causes an accumulation of waste due to the growth of the plastic industry. As far as we know, there is still no study of electrospinning associated with PVC waste. The purpose of this study is then to provide alternative solutions in the processing of PVC waste into fibers by means electrospinning method. The solvents used for the precursor solutions were N, N dimethylformamide (DMF), tetrahydrofuran (THF) and dimethylacetamide (DMA). In this research, the relationship between the concentration of PVC in the precursor solution to the morphology of the fibers and particles as the product of electrospinning was investigated.

2. Materials and methods

2.1. Materials

PVC waste was collected from the PVC pipe waste. The main solvents used were N, N dimethylformamide (DMF), tetrahydrofuran (THF), and dimethylacetamide (DMA), which were purchased from Sigma-Aldrich.

2.2. Synthesis of fiber

To observe the effect of polymer concentration to the fibers, several concentrations of PVC solution (5, 10, 15 and 20) wt% were prepared by dissolving PVC in different solvents (DMF, DMAc, and THF).

The fiber membranes were fabricated using electrospinning apparatus (Nachriebe 600) that consists of a syringe pump and its controller, a high voltage (HV) power supply, a drum collector, and a synthesis chamber with a controlled environment, as schematically shown in Figure 1. The prepared precursor solution was filled in the plastic syringe with needle diameter of 0.8 mm. The filled syringe was mounted on the syringe pump to control the flow rate of the ejected solution. The metal needle tip was then connected to the positive terminal of the HV power supply. The grounded drum collector was coated with aluminum foil. The ejected solution from the metal needle tip, which forms a Taylor cone, was monitored using a CCD camera in the synthesis chamber.

The characterization of the fibers was done by using a digital microscope (National, DC3-163) with the magnifications of 400 times and 1000 times. The microscope images were then processed to obtain the average and the distribution of the diameter of fibers. The measurement of diameter was taken at different locations on the fibers up to 100 points. The average diameter of fibers was calculated using Equation 1.

\[ CV = \frac{\sigma}{\mu} \]  

where \( \sigma \) is the standard deviation and \( \mu \) is the average diameter of fibers [14].
3. Results and discussion

It is widely known that the morphology of the fibers produced from electrospinning is affected by the polymer properties (the type and molecular weight), solution properties (polymer concentration, viscosity, conductivity and surface tension), processing parameters (voltage, distance the needle tip to collector, flow rate, and the diameter of the tip of the needle), and environmental factors (temperature and humidity). By adjusting the parameters, the microstructure of the nanofibers can be altered [6], [11]. In this research, the effects of the concentration of polymer in a DMF, DMAc, and THF solvent were studied as follows.

PVC solutions with various concentrations of (5, 10, 15, and 20) wt% were created using a DMF solvent. Figure 2 shows microscope images of the PVC fibers with DMF solvent under the digital microscope with magnifications of 400 times and 1000 times and their respected histograms showing the size distribution of fibers. During synthesis, the process parameters were kept constant. The flow rate was set at 0.5 mL∙h⁻¹, the applied voltage was 12 kV, and the distance from the collector to the tip of the needle was kept at 15 cm.

Figure 2. Microscope images of the PVC fibers produced by employing DMF solvent under the digital microscope with the magnification of 400 times and 1000 times and their corresponding histograms: (a) PVC 5 wt%, (b) PVC 10 wt%, (c) PVC 15 wt%, and (d) PVC 20 wt%
From Figure 2, it can be seen that the gradual change of microstructures from particles to fibers does exist along with the increase of the concentration of PVC waste solution. In Figure 2.a, the morphology of the particles made of the precursor solution with the concentration of 5 wt% was particles with average diameter of 2.91 μm. The fibers started to appear when the concentration of the precursor solution was 10 wt% as shown in Figure 2.b with an average diameter of fibers of 1.12 μm. Furthermore, in Figures 2.c to Figure 2.d, as the polymer concentration increased, more fibers were formed, and the diameter of the resulting fibers increased as well. The average diameter of produced fibers for the 15 wt% concentration was 1.47 μm while the average diameter made of 20 wt% concentration was 1.63 μm.

Figure 3 gives the microscope images of the PVC fibers with DMAc solvent under the digital microscope with magnifications of 400 times and 1000 times, and their respected histograms are showing the size distribution of fibers. During the synthesis, the process parameters were again kept to be the same as before.

Figure 3. Microscope images of PVC fibers produced by employing DMAc solvent under the digital microscope with magnifications of 400 times and 1000 times and their corresponding histograms: (a) PVC 5 wt%, (b) PVC 10 wt%, (c) PVC 15 wt%, and (d) PVC 20 wt%

From Figure 3, there was also a gradual morphological change from particles to fibers along with increasing the concentration of PVC waste in the solution. However, in this case, the fibers started to appear at a higher concentration (20 wt%) as a contrast to DMF where the fibers were formed at lower concentration (10 wt%). The average diameters of obtained particles were (2.79, 3.03, and 3.31) μm for the concentrations of (5, 10, and 15) wt%, respectively, while the average diameter of fibers was 1.35 μm (Figure 3.d).

Figure 4 depicts microscope images of the PVC fibers obtained by using THF solvent under the digital microscope with magnifications of 400 times and 1000 times, and their respected histograms showing the size distribution of fibers. During the synthesis, the process parameters were kept constant as before.

Figure 4 shows microscope images of the PVC fibers the use of THF solvent resulting in a morphological change from particles to fibers similar to the two prior solvents (DMF and DMAc). Submicron particles were produced with average sizes of particles of 6.83 and 8.38 μm as depicted in Figures 4.a and Figure 4.b. In Figure 4.c, it can be seen that the fibers began to form with an average diameter of fibers of 1.38 μm. Finally, in Figure 4.d, the particles disappeared and were replaced by fibers with an average diameter of 2.83 μm.

Based on the obtained results, it is clearly found that the concentration of PVC in the solutions greatly affects the morphology. Particles will be formed if the concentration of the precursor solution is low for all types of solvents used since the interaction between solvent molecules with other solvent molecules is more dominant as compared to the interaction between the polymer molecules with other polymer molecules. This causes a tendency to form spherical particles aiming to reduce the surface
area. Additionally, for low polymer concentrations, the surface tension is likely to have a large value. When the surface tension is much higher than the force due to the interaction of charge and also the force due to the interaction between the molecular chains of the polymer, then particles will be formed [6], [11]. Furthermore, fibers will be generated as the concentration of polymer is increased for all solvents. As the concentration is further increased, the diameter of the fibers will increase as well. If the interaction between polymer molecules is higher than the solvent molecules, the fibers will be formed when the liquid jet is attracted to the collector due to electrical force. The diameter of fibers will increase due to the high viscosity as a result of high polymer concentration. The high viscosity will restrict the deformation of a liquid jet into the fiber, and thus the stretching process will be longer. Consequently, if other parameters are kept constant, the diameter of the fibers will be larger as a result of higher polymer concentration [6], [11]–[14].

![Image](image1.png)

Figure 4. Microscope images of PVC fibers produced by using THF solvent under the digital microscope with the magnifications of 400 times and 1000 times and their corresponding histograms: (a) PVC 5 wt%, (b) PVC 10 wt%, (c) PVC 15 wt%, and (d) PVC 20 wt%

4. Conclusions
The morphological change of PVC particles to fibers has been successfully produced by electrospinning using PVC waste diluted in DMF, DMAc and THF solvents. From the obtained microscope images, there was a critical concentration of PVC solution in which electrospun PVC experienced a morphological change from particles to fibers. In general, PVC particles were formed for PVC concentrations lower than its critical concentration, and the average size of particles increased with the PVC concentration. For the concentrations higher than its critical PVC concentration, PVC fibers were obtained, and the average size of fibers increased with the PVC concentration.

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