Production of Nanofibers from Waste Poly (Ethylene Terephthalate) Using Electrospinning Method

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Abstract

Nanofiber is a broad terminology referring to a fiber with a diameter less than one micrometer. In this paper waste Poly(ethylene-terphthalate) (PET) was electrospun by using solution electrospun method and using a combination of solvents with different Trifluoroacetic acid (TFA) and Dichlormethae (DCM) solvent ratios. The effect of TFA: DCM solvent ratios and polymer concentrations on the fiber formation and fiber diameter were investigated. The synthesized nanofibers were examined using Scanning Electron Microscopy (SEM) to evaluate the fiber diameters. The obtained physical characteristics of nanofiber resulting from the Scanning Electron Microscopy showed nanofibers obtained under various conditions, which will be discussed in more details in main paper text. The obtained results considered as promising results for better properties and thus new application of recycled PET

Keywords: Electrospinning, Nanofiber, SEM, Ploy (ethylene terephthalete)

Introduction

Nanofibers can be produced by a number of techniques such as drawing, template synthesis, phase separation, self-assembly and electrospinning [1]. The fibers synthesized by electrospinning are smaller in diameter than those synthesized by traditional fiber production methods such as melting or solution spinning. The average fiber diameters of these polymeric nanofibers are in the range of 10 nm to 1 μm [2, 3]. Potential applications of such nanofibres include filtration and composite materials, catalyst supports, optical and chemical sensors, drug delivery, and electrospun non-woven biodegradable fabrics that can be used as adhesion barriers, for wound dressing and tissue engineering. Several reviews summarized important developments in the field of electrospinning [4-7].

A conventional electrospinning device includes a spindle, a high-voltage power source, and an assembly. During electrospinning, the polymeric solution is filled into the capillaries. A high electrical voltage was applied across the polymer solution. To overcome the surface tension of the polymer solution, the electric field induces repulsive force charging in the polymer solution. When the applied voltage reaches a critical value, the polymer comes out from the tip of the needle and forms fibers due to the electric field, and these fibers will be thinner as they travel in the air due to elongation and evaporation of the solvent. The fibers are finally collected on the collector electrode as a fibrous mesh [8, 9].

There are many parameters impact fiber formation and their morphology, during the electrospinning process. These include concentration, viscosity and conductivity of the solution, as well as the applied voltage, tip to collector distance, initial jet radius, and flow rate of the solution and other environmental conditions. These different processing parameters can be used to control the fabrication of fibers [10-12].

In electrospinning method, at low polymer concentrations, due to the effect of applied voltage and surface tension of the solution, the charged jet fragments into discrete droplets before reaching the ground collector. At an increased polymer concentration, the solution viscosity is increased and chain entanglements between the polymer chains are improved and smooth nanofibers are formed. The polymer concentration of the solution to be electrospun has an effect on both the viscosity and surface tension which ultimately decides the electrospinnability of the solution into nanofibers. Under the same electrospinning conditions, increasing the polymer concentration will increase the diameter of the electrospun fibers. However, Deitzel et al.[12] found that a non-linear relationship between the solution concentration and the fiber diameter usually forms. The reason for this non-linear relationship can be attributed to the non-linear relationship between the polymer concentration and the solution viscosity [13]. The fiber diameter will increase with an increase in polymer concentration until a certain concentration limit is reached where the solution viscosity will be too high, disrupting the flow of the polymer solution through the capillary [14]. Reneker et al. investigated the effect of viscosity on the electrospinning of aqueous PEO solutions. They observed that nanofibers were formed from solutions with viscosities between 800 and 4000 cp. Below 800 cp the jet garmented into droplets and above 4000 cp nanofiber formation was difficult because the solution dehydrated at the tip of the capillary [2]. Thus, Nanofiber formation occurred at an optimum range of solution viscosity as dictated by an optimum polymer concentration.

PET is a thermoplastic made by condensation polymerization and has many applications due to its resistant to excellent mechanical properties. Due to the single-use, PET polymer regarded as one of the most common causes of environmental pollution. Electrospinning of PET waste is one of the most promising ways to benefit from its waste accumulation. Gergely A et al [15] produces PET fibers with 200-600 nm diameter, and free-standing fiber mats that could potentially be used in filtration applications.

The most effective solvents for PET are Trifluoroacetic acid (TFA) and Dichloromethane (DCM) [16]. Therefore, main objective of this research is to investigated the effect of variation of the solution parameters namely concentration, viscosity and solvents volume percentage, on the fiber formation and the obtained nanofiber characterization.

Experimental Work

The waste PET (i.e. PET bottles) were collected and cleaned properly, then some properties of these samples were determined as shown in the table 1, polymer solution was obtained at several concentrations in mixture of (TFA) and (DCM), which were used as solvents, and the solutions were continuously stirred for adequate time (2 to 3 hr) at room temperature to insure dissolving the polymer.

Type of sample	M_w (g/mol)	Mn (g/mol)	PDI	T_g (k)	T_m (k)
Used PET	62,000	40,000	1.52	350	525

Table 1: Characterization and Properties of the used PET

Electrospinning was performed according to our previous work [17]. Figure 1 shows the electrospinning device used to spin na-nofibers. The setup of the experiment consists of the following: syringe usually (10-ml) to contain the polymer solution. Stainless steel needle that were positioned horizontally, a syringe pump to regulate flow rate, volume of the polymer solution and thus controlling fiber diameter. A DC voltage supply in the kV range up to 50 kV. A voltage source is connected to the syringe pump to pull the fibers from the solution into the syringe. A voltage source also connects the syringe to the collector plate. Since the collector plate carries a positive charge, this will force the negatively charged polymer solution toward the collector.

Once the applied electric field reaches a critical value, the surface tension force of the polymer solution is disrupted. This will cause a charged jet of the solution to be expelled from the tip of the Taylor cone. As the polymer solution travels through the air, the solvent evaporates and this leads to the formation of fine solid fibres on the collector. The fibre charge will eventually dissipates into the surrounding environment [18].

The distance between collector and needle tip was 15 cm for all experimental runs. The flow rate of polymer solution was also constant at about 0.01 ml/min.
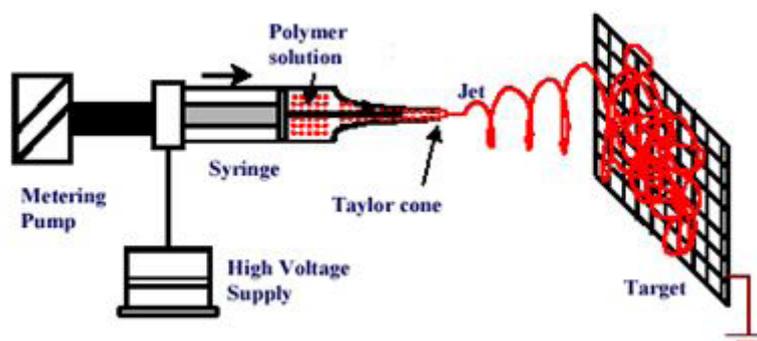

Figure 1: Schematic representation of the applied electrospinning process

To investigate the effect of the polymer concentration on fibre properties, five different PET polymer concentrations ranges from 5, 8, 12, 15, and 20 % wt the solvent used here was a mixture of (DCM and TFA) with 50:50 volume percent.

Scanning Electron Microscope (SEM) analysis was performed to obtain the morphology of the electrospun nanofibers and to confirm the formation of nanofibers. The instrument used was a Zeiss Merlin Field Emission Scanning Electron Microscope (FE-SEM. The samples were coated using an Edward S150A Gold Sputter Coater. The coated samples were loaded into the SEM instrument and images were recorded at 7 kV voltage with 250 pA beam current under vacuum.

Results and Discussion

The electrospinning of PET samples was carried out using various experimental conditions, in order to identify the optimum electrospinning conditions. Figure (2) shows SEM images of PET nanofibers obtained at various polymer solvent concentrations. It is clear from Figure (2) that electrospinning solution of various PET concentration using TFA: DCM 20:80 solvent ratio does not resulted in a stable and smooth electrospinning nanofibers. This is most probably due the high evaporation rate of DCM solvent which represents the highest part of the solvent mixture
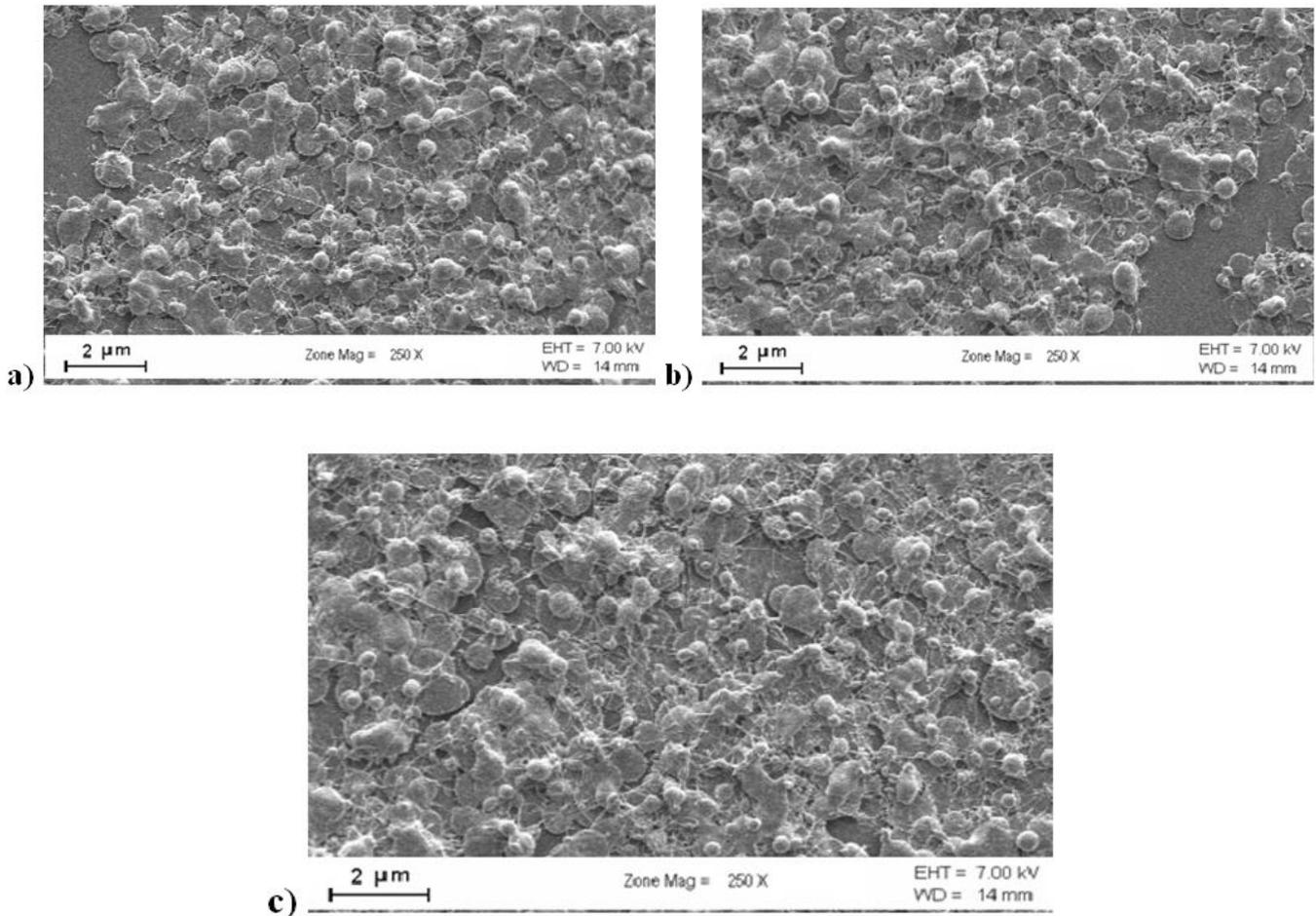

Figure 2: SEM images of PET nanofibers at a) 10 wt %, b) 20 wt% and c) 30 wt %

PET concentration in 20:80 vol % of TFA and DCM solvents

Due to of the fast evaporation of solvent and insufficient time for the polymer chain entanglement and overlap, will lead to the formation of polymer droplets. Other researcher [15] has observed similar results earlier. Figure (2) shows some limited fibre formation occurred in the form of very thin fibres that were connected by polymer beads. However, these beaded fibers were discontinuous with an overall length of no more than 1 μm.

However, different results were obtained when different solvent ratios are used. Figure (3) shows SEM images of the obtained PET nanofibers and the corresponding diameter distribution diagrams using three different PET concentrations, a) 10 wt. %, b) 20 wt% and c) 30 wt % and 40:60 vol % of TFA and DCM solvents. Nanofibers are observed for all the polymer solution concentrations and a few numbers of distributed beads can be seen on several nanofibers. The diameters of these beads were about 1 μm to 2 μm
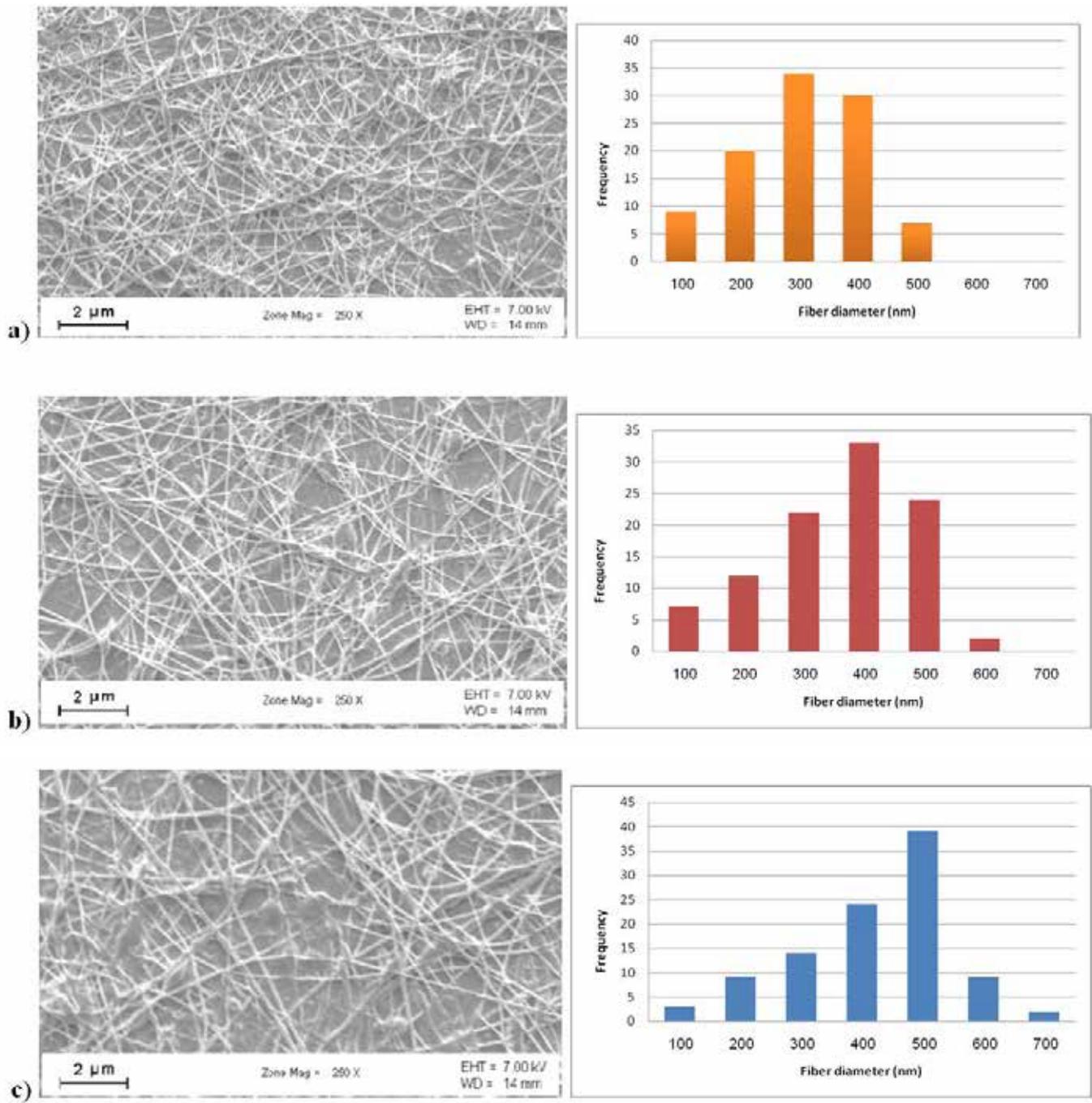
Figure 3: SEM images and corresponding diameter distribution diagrams of PET nanofibers at a) 10 wt %, b) 20 wt% and c) 30 wt % PET concentration in 40:60 vol % of TFA and DCM solvents

Furthermore, from the diameter distribution diagrams of PET nanofibers in Figure (3) one can observe that increasing PET polymer concentrations results in an increase in the diameter of the nanofibers. This can be due to the increasing in the chain entanglement. The higher concentration of PET polymer solution leads to increasing the viscosity of the electrospun solution, which will lead to increase the entanglement of PET polymer chains and it is well known that the chain entanglement is one of the factors, which contributed to viscosity. Therefore, the higher concentration of polymer solution tends to increase the diameter of nanofibers.

Similar results were obtained as the percent of DCM solvent increases in the TFA: DCM solvents ratio to 60%. From Figures 4, it can be seen that with the increase of the TFA: DCM solvent ratio, fibers with fewer beads, more uniform and smoother were observed. The Figure also shows that as the PET polymer concentrations increases the diameter of obtained nanofibers increases and more beads were observed.
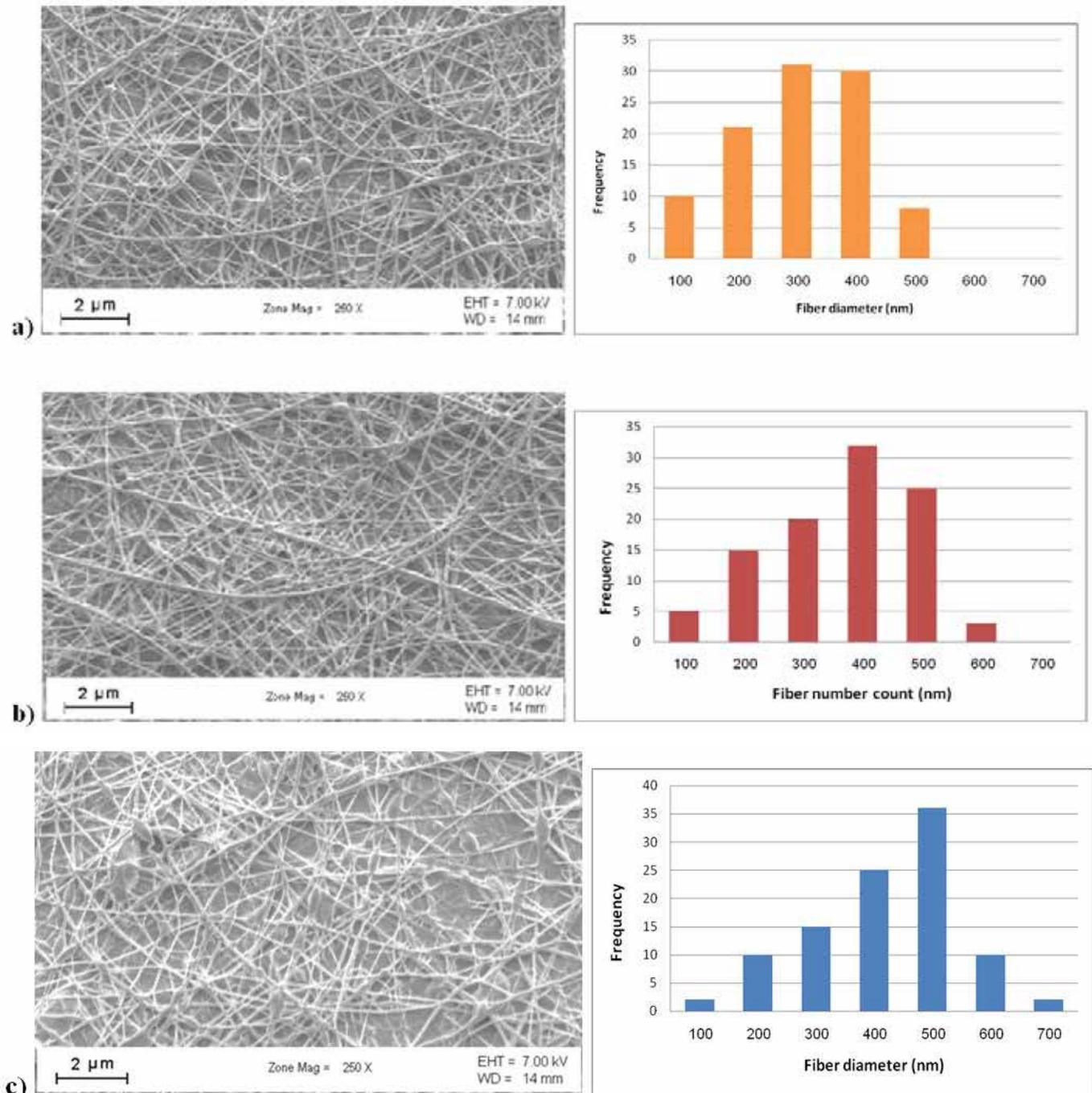

Figure 4: SEM images and corresponding diameter distribution diagrams of PET nanofibers at a) 10 wt %, b) 20 wt% and c) 30 wt % PET concentration in 60:40 vol % of TFA and DCM solvents

The polymer concentration of the solution to be electrospun has an effect on both the viscosity and surface tension, which ultimately decides the electrospinnability of the solution into nanofibers. The fiber diameter will increase with an increase in polymer concentration until a certain concentration limit is reached where the solution viscosity will be too high, disrupting the flow of the polymer solution through the capillary [14].

Conclusions

In this study, nanofibers were produced from waste PET using electrospinning method. From the obtained results it is observed that the polymer concentration has an effect on both the viscosity and surface tension which ultimately effect the electrospinnability of the solution into nanofibers. The results showed with increasing of PET polymer concentration derive to increase the diameter of nanofibers. This is attributed to the increase in viscosity with increasing concentration of PET and this is a result of the increase in chain entanglements. The results also showed that by using a mixture of solvents with different boiling points, the specification and morphology of the nanofibers could be controlled.

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