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# Production of Fibre Mats from Recycled PET Bottles

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## Abstract

The excessive use of polymer materials in the packaging industry and improper waste management greatly contribute to environmental pollution. The recycling of this plastic waste is key to improving sustainability. In this paper the production of non-woven fibre mats from PET bottles was studied, which can be used in air filtration. The fibres were generated using centrifugal spinning. The potential to substitute the common solvents of PET with less harmful ones was also investigated.

**Keywords:** *PET, centrifugal spinning, microfibre, recycling.*

## 1. Intorduction

Polyethylene terephthalate (PET) is the sixth most produced polymer with ~56 million tons being synthesized and manufactured into goods each year. Roughly 40% of this amount is used in the packaging industry (foils, bottles, boxes), and another 45% in the textile industry. Considering the nature of its uses, the amount of PET waste produced yearly is also high **[1]**.

There are various ways to recycle PET, one of them is the production of nano- and microfibres. The most common technique to produce non-woven nanofibre mats is electrospinning. Other possible methods are melt blowing and centrifugal spinning. One of the primary aims during fibre production is to minimize the fibre size **[2]**. Melt spinning does not require the use of solvents at all, however the equipment is more complex, and the in the case of PET the average fibre diameter of the produced fibres is around an order of magnitude larger **[3]**.

During centrifugal spinning a chamber that contains the polymer solution is rotated at 4000-15 000 1/min. Due to the centrifugal force, the polymer solution ejects from the chamber through radial capillaries, creating liquid jets. As the jets travel through the air the solvent evaporates, and the dry fibres deposit on the collector. Despite a simple setup, centrifugal spinning has a high production rate even under laboratory conditions. In addition, the electrical conductivity of the solution does not influence the process, and there is no need for a high voltage source, as in the case of electrospinning **[2]**.

Vo et al. . successfully produced PET fibres with centrifugal spinning from PET solutions in a mixture of trifuloroacetic acid (TFA) and dichloromethane (DCM) in a 70/30 mass ratio. The experimental setup involved various capillary diameters (160-340 μm), rotational speeds (6000-15 000 1/min), and solution concentrations (5-13% PET). The average diameter of the fibres that did not contain beads were between 0.66±0.27 and 4.26±2.32 μm, depending on the process parameter **[4]**.

Since PET is a semi-crystalline polymer, it doesn't readily dissolve in most organic solvents. In general PET solutions are prepared with acidic solvents, most often with TFA. In some published studies the mixture of TFA and DCM is featured in 70/30 and 50/50 mass ratios **[4, 5]**. The problem with fluorinated acids is that they are toxic and persist for a long time in the environment. The half-life of TFA in aquatic ecosystems is on the scale of around a hundred years **[6]**. CTherefore, it is crucial to look for alternatives.

The solubility of polymers can be studied with the help of the Hansen solubility parameters (HSP). According to the model, the solubility of every chemical compound is determined by 3 interaction parameters, that represent the potency of dispersion ($\delta D$), polar ($\delta P$) and hydrogen bonding ($\delta H$) intermolecular forces. The closer the HSPs of the polymer are to that of the solvent, the better the solubility [7].

## 2. Experimental

### 2.1. Materials

Polyethylene terephthalate (PET) was acquired from a mineral water bottle. Trifuoroacetic acid (TFA, Sigma Aldrich, 99%), phenol (Thermo Scientific, 99%), dichloromethane (DCM, VWR Chemicals, 99.8%), dimethyl formamide (DMF, VWR Chemicals, 98%), and toluene (VWR Chemicals, 99.9%) were used as received.

### 2.2. Polymer solution preparation

15% PET solution was prepared with 15/85 mass ratio TFA/DCM solvent mixture, and 10, 12.7, 15, and 20% solutions with 30/70 TFA/DCM mixture in 20 ml vials. The contents of the vials were stirred with a magnetic stirrer for 24 hours at room temperature. For solubility studies 10% PET was added to 50/50 mixture of phenol and DCM, as well as 55/45 mixture of toluene and DMF.

### 2.3. Centrifugal spinning

Centrifugal spinning was performed with a custom-built setup [8]. A chamber (spinneret) with two radial capillaries (25G needles) was rotated by an electric motor at 4000-8000 1/min. The polymer solution was fed into the spinneret through a teflon tube at 60 mL/h with the help of a syringe pump. Fibres were collected on 8 radially positioned steel rods with a needle-collector distance of 100 mm. All experiments were performed at 22°C and 40% relative humidity.

### 2.4. Scanning Electron Microscopy (SEM)

SEM was performed using a JEOL JSM-5200 scanning electron microscope at 15 kV accelerating voltage on uncoated samples. The PET fibre diameters were measured with the open-source ImageJ software, and 50 measurements were taken on each image.

### 2.5. Solubility calculations

The Hansen solubility parameters (HSP) of PET and the solvents can be found in the literature [7].

The HSP distance ($R_a$) can be interpreted as the radius of a sphere, that is calculated from the difference of the position vectors of the polymer and the solvent in the HSP space, as follows:

$$R_a = \sqrt{4 \cdot (\delta D_2 - \delta D_1)^2 + (\delta P_2 - \delta P_1)^2 + (\delta H_2 - \delta H_1)^2} \quad (1)$$

where a $\delta D$, $\delta P$ and $\delta H$ denote the dispersion, polar, and hydrogen bond interactions, while the lower indices 1 and 2 refer to the polymer and the solvent, respectively. The dispersion term is multiplied by 4, based on empirical data [7].

## 3. Results and Discussion

The 15% PET in 15/85 TFA/DCM did not dissolve even after 24 hours of constant stirring. For this reason, fibres were not produced at this concentration. In order to increase the solubility of PET the amount of TFA was increased, resulting in a 30/70 TFA/DCM solvent ratio. 10, 12.7, and 15% PET were fully dissolved in the solvent mixture, however at 20% only partial dissolution was achieved.

Centrifugal spinning was performed with the 10, 12.7, and 15% solutions, each time at 3 different rotational speeds. Out of the nine attempts only the spinning of the 10% solution at 6000 and 8000 1/min and the 12.7% solution at 4000 and 6000 1/min resulted in fibre generation. The 10% solution at 4000 1/min produced mostly droplets, while the 12.7% solution at 8000 1/min, and the 15% solution at all rotational speeds produced only short fibre segments that could not be captured by the collector rods.

The SEM images are shown in Fig. 1. The images show that the generated fibres contained beads at all conditions. According to the literature beading can be reduced with the increase of the polymer concentration, which is due to the increased solution viscosity, and thus the higher viscoelastic tensile forces that oppose the surface tension [9]. In our case increasing the PET concentration from 10 to 12.7% did not result in a lower bead density, and at 15% the formation of continuous fibres ceased.

The fibre diameters were measured from the SEM images, and the results can be seen in Table 1. The results show that there was a slight increase in average fibre diameter and standard deviation as the rotational speed increased. The most noticeable increase in fibre diameter, approximately 28%, was observed in the case of the 12.7% solution. Based on the SEM data the most favorable conditions in regards of fibre size were the 10% solution concentration and 6000 1/min rotational speed, resulting in both the lowest average size and lowest deviation. The fiber mats showed a slight orientation perpendicular to the axis of the spinneret.

The PET solution preparation experiments showed that the TFA and DCM solvent ratio greatly affected the solubility of PET. In the majority of the published literature PET solutions are prepared either with a 70/30 or 50/50 mass ratio of TFA and DCM. In this study complete dissolution and fibre formation was achieved with a 30/70 TFA/DCM solvent mixture, which is a significant reduction in acid usage. On the other hand, the average diameter of fibres produced under similar conditions, but from a 70/30 TFA/DCM mixture was smaller (0.66±0.27 µm) [4].

In order to study the dissolution phenomenon of PET in more detail, the Hansen solubility parameters can be used. Fig. 2 shows the solubility parameters of PET and some other chemicals that may be given consideration as a potential solvent. For reference, water was also added to the plot. It can be seen, that DCM is located close to PET ($R_a = 2.06$), which would indicate that it is a good solvent, however our experiments showed that pure DCM does not in fact dissolve PET. In contrast, TFA is further away on the chart ($R_a = 6.71$), and yet it dissolves PET. Our hypothesis is that this is due to the semi-crystalline nature of PET, and the key to the dissolution is the acidity of the TFA, which is not accounted for in the Hansen solubility model.

**Table 1.** *Average fibre diameters and standard deviations*

| Concentration | Rotational speed (1/min) | Fibre diameter (µm) |
|---|---|---|
| 10% | 6000 | 1.72±0.63 |
| | 8000 | 1.94±0.98 |
| 12.7% | 4000 | 1.89±0.84 |
| | 6000 | 2.42±1.37 |

Experiments with other solvent mixtures resulted in the PET dissolving in 50/50 mixture of DCM and phenol ($R_a = 2.88$), which is a weak acid. It should be noted that phenol-based solutions are unfit for fibre production, as phenol is a crystalline solid at room temperature, and it does not evaporate during fibre generation. PET did not dissolve in 55/45 DMF/toluene (not acidic), albeit the HSP distance was particularly low in this case ($R_a = 1.57$). As a continuation of the project, solu-

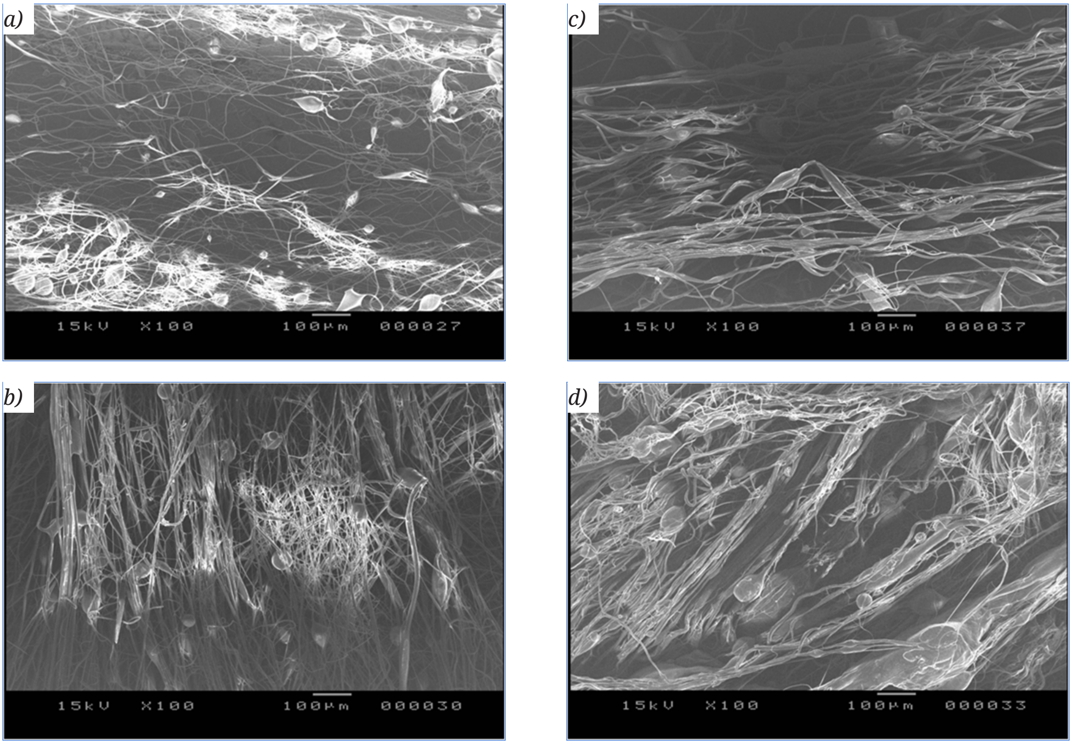

**Fig. 1.** *SEM micrographs of the PET fibre mats at 100× magnification. a) 10% , 6000 1/min, b) 10%, 8000 1/min, c) 12.7%, 4000 1/min, d) 12.7%, 6000 1/min.*

bility experiments with amorphous PET can be attempted, in which case the acidity of the solvents might not play a significant role.

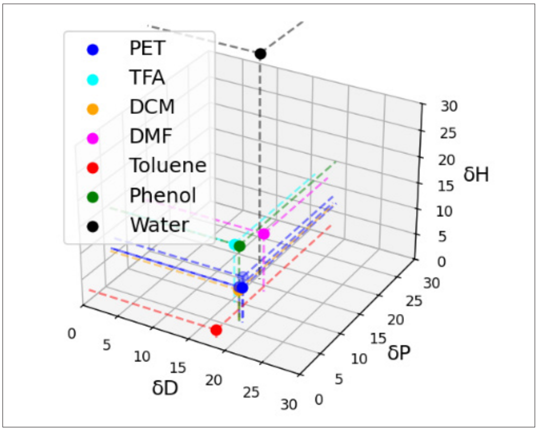

**Fig. 2.** *Hansen solubility parameters of PET and various solvents. The labels on the axes represent the δD – dispersion, δP – polar, δH – hydrogen bond components.*

## 4. Conclusions

PET-based non-woven fibre mats were successfully created with centrifugal spinning from recycled water bottles. 10% PET concentration, 30/70 TFA/DCM solvent ratio, and 6000 1/min rotational speed proved to be the optimal conditions, resulting in fibres with 1.72±0.63 µm in diameter. Under all conditions beads were present in the fiber mat, so further optimization of the production parameters is necessary. In addition, we studied the solubility of PET with the Hansen solubility model. According to the model, multiple substances should act as a solvent, however the results indicate that an acidic medium is required for the dissolution of semi-crystalline PET.

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