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SURFACE SCIENCE and TECHNOLOGY

Lab Work

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Research: Electrospinning Technology

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Master of Science in Materials Engineering for Industry 4.0

QUESTION 1

What is the electrospinning process? State and deeply describe the working principle of this technology, reporting and analyzing in detail its advantages and disadvantages. What are the parameters that affect the morphological features of final nanostructured samples?

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<u>What is the electrospinning process?</u>

Electrospinning is a voltage-driven fabrication process governed by a specific electrohydrodynamic phenomenon where small fibers are yielded from a polymer solution [1]. Electrospinning is a versatile and viable technique for generating ultrathin fibers (Xue et al, 2019). In order to obtain final nanofiber mats, starting from the polymeric solution, loaded in a syringe, an electrical potential must be applied between the tip of a needle, which is defined as the first electrode, and the counter electrode, where dried nanofibers are collected. (Massaglia et al, 2018)

<u>Description of the working principle of electrospinning process.</u>

The working principle of electrospinning involves the application of an electric field to a polymer solution or melt to create ultrafine fibers. The following is the breakdown of the process:

- Preparation: First, a polymer solution or melt is prepared. This solution typically consists of a polymer dissolved in a suitable solvent or a polymer melt at an elevated temperature (Keirouz et al, 2023).
- Setup: The polymer solution or melt is loaded into a syringe equipped with a metallic needle called a spinneret (Unnithan et al, 2015). The spinneret tip is where the electrospinning process occurs.
- Electric Field: A high voltage power supply is connected to the spinneret needle and a grounded collector plate or rotating drum placed at a certain distance away.
- Forces: When the voltage is applied, electrostatic forces are generated between the surface of the polymer solution/melt at the tip of the spinneret and the collector plate/drum. The surface of the solution/melt becomes charged due to the electric field (Keirouz et al, 2023).
- Taylor Cone: At high voltage, the solution forms a cone-shaped droplet (Taylor cone) at the needle tip (Palwai et al, 2023)
- Jet Formation: Once the electrostatic forces are strong enough, a jet of polymer solution/melt is ejected from the tip of the Taylor cone towards the collector plate/drum. The jet is accelerated due to the repulsive forces within the polymer solution/melt and the electrostatic forces acting upon it (Varesano et al)
- Solidification: As the jet travels, solvent evaporates (if solution) or melt cools (if melt), solidifying the polymer into nanofibers.
- Collection: The solidified nanofibers are collected on the grounded collector plate or rotating drum. Additionally, the arrangement of the collector plate or drum can influence the morphology and alignment of the collected nanofibers.

Advantages of electrospinning process:

- Nanofiber Production: Electrospinning produces nanofibers with diameters ranging from a few nanometers to several micrometers, offering high surface area-to-volume ratios and unique properties (Sharma et al, 2022)
- Versatility: It can work with various polymers and additives, allowing for the production of nanofibers with tailored properties such as porosity, surface chemistry, and mechanical strength (Senthamizhan et al, 2017)
- Controlled Morphology: The electrospinning process enables control over the morphology, alignment, and size distribution of the nanofibers, making it suitable for specific applications (García-Mateos et al, 2019).
- Scalability: Electrospinning setups can be scaled up for industrial production, making it feasible for large-scale manufacturing of nanofibrous materials.
- Applications: Electrospun nanofibers find applications in diverse fields such as tissue engineering, drug delivery, filtration, sensors, protective clothing, and energy storage (Haider et al, 2018).

Disadvantages of electrospinning process:

- Complexity: Setting up and optimizing electrospinning parameters can be complex, requiring expertise and careful control of variables such as polymer concentration, solvent selection, voltage, and distance between the spinneret and collector.
- Limited Production Rate: Electrospinning is typically a slow process compared to other fiber manufacturing techniques, limiting its throughput for large-scale production (Laudenslager et al, 2012).
- Solvent Concerns: The use of organic solvents in electrospinning can pose environmental and safety concerns (Hong et al, 2019). Additionally, the removal of residual solvent from the produced nanofibers may be necessary.
- Equipment Cost: High-voltage power supplies, precise syringe pumps, and other specialized equipment required for electrospinning can be expensive, particularly for research and small-scale applications (Flores-Rojas et al, 2023).
- Poor Mechanical Properties: Some Electrospun nanofibers may have lower mechanical properties compared to bulk materials, which can limit their application in load-bearing or structural components (Zulkifli et al, 2023).
- Overall, while electrospinning offers unique advantages in producing nanofibrous materials with tailored properties, it also comes with challenges related to process complexity, scalability, solvent usage, and material properties.

The parameters that affect the morphological features of final nanostructured samples.

There are several parameters that influence the morphological features of final nanostructured samples produced by electrospinning. These parameters include:

- **Polymer Solution/Melt Properties:**
  - Polymer concentration: Higher concentrations typically result in thicker fibers (Torfifard et al, 2019).
  - Molecular weight and viscosity: Higher molecular weight and viscosity lead to larger fiber diameters (Abdulhussain et al, 2023).
  - Type of polymer: Different polymers have varying electrospinnability and resulting fiber

morphology.

- **Solvent Properties:**
  - Type of solvent: Solvent polarity and volatility affect polymer solubility and jet stability (Luo et al, 2010).
  - Solvent/polymer ratio: Adjusting the ratio can influence fiber morphology and diameter.

- **Process Parameters:**
  - Applied voltage: The voltage applied in electrospinning has a significant impact on the fibers produced. Increasing the voltage leads to a smaller fiber diameter. This is because a higher voltage results in a greater electric force acting on the fibers, causing them to thin and elongate more [20]
  - Distance between spinneret and collector: Longer distances can lead to thinner fibers and vice versa (Phillemon et al, 2013).
  - Flow rate of polymer solution/melt: Faster flow rates generally result in thicker fibers.
  - Spinneret geometry: The shape and size of the spinneret affect the jet formation and fiber morphology (Keirouz et al, 2023).
  - Collector speed (if using a rotating collector): Rotation speed influences fiber alignment and deposition density.
- **Environmental Conditions:**
  - Relative humidity: Humidity can affect solvent evaporation rates, jet stability, and fiber morphology.
  - Temperature: Temperature influences the viscosity of the polymer solution/melt and solvent evaporation rate.

  The ambient parameters such as (i) higher humidity and (ii) increasing temperature produce thicker fibers and generate a thinner nanofiber, respectively. The controlled parameter through optimization decides the size and quality of the fibers (Chinnappan et al, 2022).

- **Additives and Processing Aids:**
  Incorporation of additives such as surfactants, crosslinkers, or nanoparticles can modify fiber properties and morphology (Abdulhussain et al, 2023).

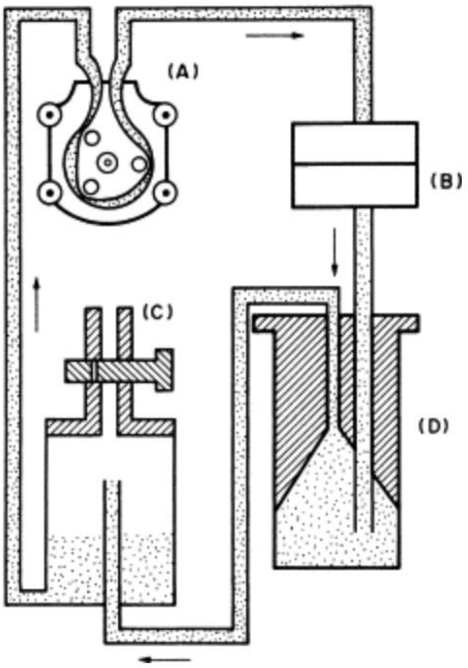
QUESTION 2: State a typical experimental procedure that must be followed to obtain final nanofibers mats. What are the possible post-process treatments to be implemented with the main aim to transform the polymeric nanofibers in other

material of nanofibers. By selecting one kind of this nanostructured material, state the experimental steps to be implemented.

Insert text (4000 characters, no spaces) and images

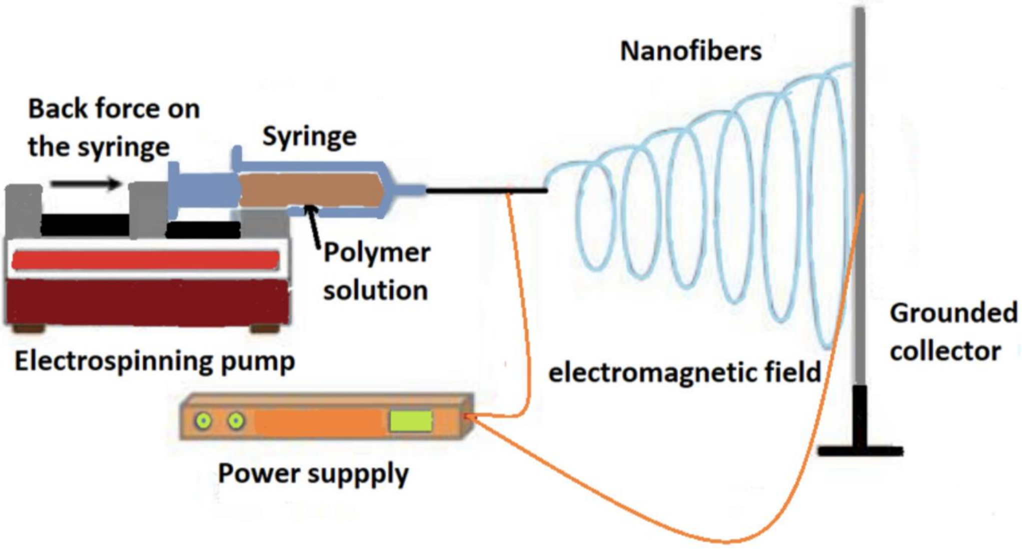
1- **Polymer Solution Preparation:**

Preparing polymer solutions for light-scattering measurements is crucial. Contaminants like dust particles can skew results, so it's vital to ensure the solution is free from foreign macromolecules and particles. To achieve this, thorough clarification of the solvent, light-scattering cell, and air above the solution is necessary, typically involving filtration and flushing with filtered air or inert gas. The polymer solution should be shielded from unfiltered atmosphere exposure, ideally filtered directly into a dust-free light-scattering cell. If needed, a closed circulation system can be used, though it may require larger quantities of the polymer solution.



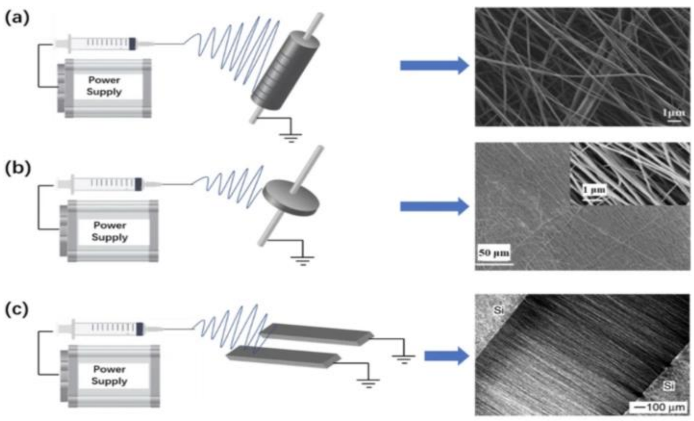
2-Electrospinning :

Electrospinning: Electrospinning is carried out at room temperature with atmosphere condition. There are basically two electrospinning set-ups; vertical and horizontal. Figure 2 shows the schematic of diagram for nanofibers produced by electrospinning process [1]. A high voltage is applied to create an electrically charged jet of polymer solution or melt. The jet undergoes stretching before it reaches the collector and it solidifies on the collector in the form of nanofibers by the evaporation of the solvent



2- **Fiber Collection:**

The ejected fibers travel towards the grounded collector plate, where they randomly deposit and accumulate to form a non-woven nanofiber mat.



Special collectors for obtaining aligned fibers: a high-speed rotating drum collectors; reproduced with permission from ref. b Tip collectors; reproduced with permission from ref. c Parallel plates collectors

3- **Drying/Heat Treatment:**

1.Drying

- **Purpose:** Removes any residual solvent trapped within the nanofibers after electrospinning.

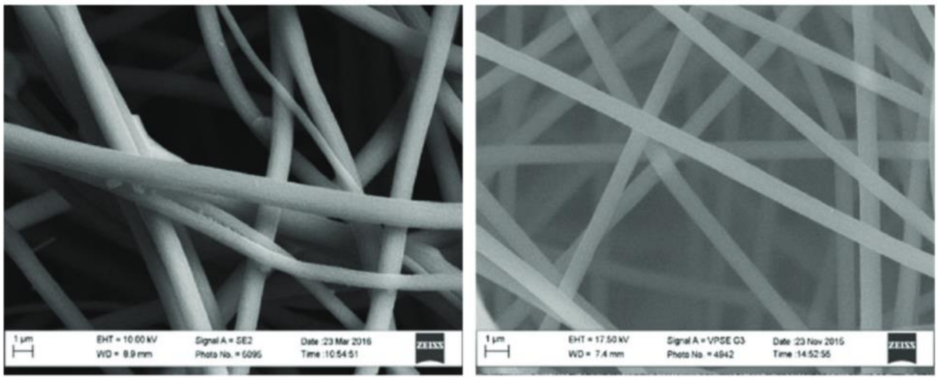
Residual solvent can negatively impact the nanofiber mat's:

- **Mechanical properties:** Solvent can act as a plasticizer, making the fibers less stiff and potentially weaker.

- **Thermal properties:** Residual solvent might boil off during subsequent processing or use, causing fiber shrinkage or collapse of the mat structure.
- **Surface properties:** Solvent presence can interfere with intended surface modifications or functionalities.

2.Heat Treatment

- **Purpose:** Modifies the physical and chemical properties of the nanofibers to achieve desired characteristics. Heat treatment can:
    - **Increase crystallinity:** For some polymers, heating can induce the polymer chains to arrange in a more ordered crystalline structure, enhancing strength, stiffness, and thermal stability.
    - **Stabilize morphology:** Heating can help solidify the fiber structure and prevent further shrinkage or collapse.
    - **Enhance specific functionalities:** Certain heat treatment protocols can activate specific chemical groups or promote cross-linking within the polymer for tailored functionalities.



(a) (b)

SEM images of the electrospun nanofiber mats: (a) Before heat treatment, and (b) After heat treatment.

Post-Processing Treatments for Nanofiber Transformation

Nanofiber mats can be further modified through various post-processing treatments to achieve specific functionalities:

- **Chemical Treatment**
- **Incorporation of Nanoparticles**
- **Composite Formation**

Chemical treatment:

Chemical Treatment of Nanofibers: Powering Up Tiny Tech

Nanofibers, with their incredibly small size and vast surface area, are already superstars in many fields. But chemical treatment takes them to the next level! It's like giving them a customized upgrade for specific jobs.

Here's how chemical treatment empowers nanofibers:

Example 1: Supercharged Filters: Imagine adding microscopic hooks to nanofibers. Chemical treatment can introduce functional groups that grab specific pollutants from water. It's like equipping the nanofibers with tiny filters to catch unwanted particles.

Example 2: Smart Fabrics: Chemical treatments can make nanofibers love water (hydrophilic) or hate it (hydrophobic). This is perfect for creating water-wicking sportswear or water-repellent raincoats. Think of it as a switch for water interaction built right into the fabric!

QUESTION 3: During the laboratory activities, you analyzed 3 different surfaces. What are the differences between them? How can you proceed to evaluate contact angle of all these surfaces qualitatively? Detail all the procedures followed.

Insert text (5000 characters, no spaces) and images

The three surfaces examined in this activity were PDMS, Nanofiber, and carbon paper.

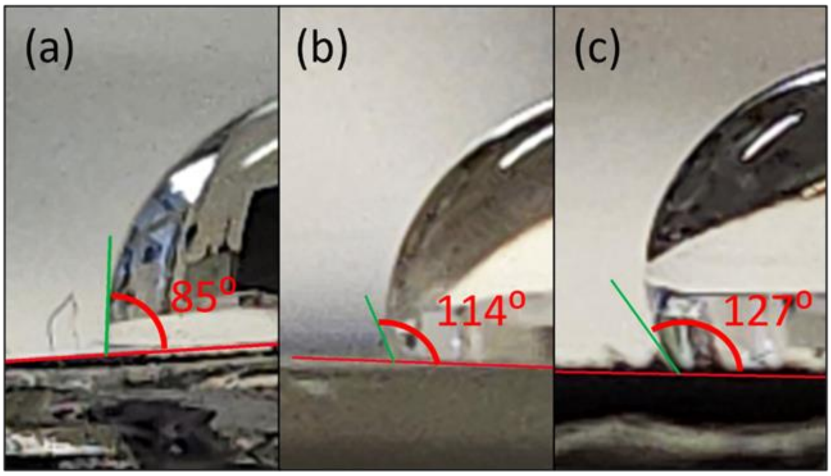
The interaction between the liquid and the solid phases influences the contact angle. Contact angles above 90° define a situation where the liquid is repelled by the solid; below 90° defines a situation of attraction where the liquid wets the surface. The magnitude above or below 90° shows the relative degree of repulsion or attraction between the two phases. (ISO REF : ISO/TS 21237:2020(en) Nanotechnologies — Air filter media containing polymeric nanofibres — Specification of characteristics and measurement methods)

A water droplet was dropped by a syringe to the horizontal planar surface of the three surfaces. Then we captured images of the samples with the camera and the desk positioned at a 90-degree angle to ensure the integrity of the water droplet angle relative to the different surfaces. Subsequently, we employed ImageJ software to quantify the contact angle of water droplets with the respective surfaces. The results are as follows:

a- PDMS with the contact angle of

b- Nanofiber with the contact angle of

c- Carbon paper with the contact angle of



Based on these images, PDMS shows a hydrophilic behaviour and nano fiber and carbon paper both show hydrophilic behavior with carbon paper being the most hydrophilic amongst the two.

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