ELECTROSPUN NANOFIBERS FORMATION

THE EFFECT OF ADDING Al ON THE MORPHOLOGY OF Fe-Al-O SPINEL NANOFIBERS

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INTRODUCTION

RESEARCH GOAL

The aim of our experiment was to synthesise (by electrospinning) different types of Fe-Al-O nanofibers for their potential application as catalysts for the production of liquid fuel out of CO_2 and H_2.

Why electrospinning? Electrospinning is the simplest and most cost-efficient method of producing nanofibers (even at large scales).[1]

Why liquid fuel out of CO_2 and H_2? This kind of fuel is 'green' and can help reduce the effects of climate change. Liquid fuel is also the most effective way of energy storage and transport.[2]

CASUALLY EXPLAINED:

NANOFIBERS

Nanofibers are fibers with diameters in the nano scale (between 1nm and 100nm). One nanometre (nm) is equal to 1 billionth ($1x10^{-9}$) of a metre.

The feature that separates one nanofiber from another is its morphology (which includes its structure, features and components).

APPLICATIONS

Nanofibers have a wide range of potential uses, including for cell adhesion, tissue engineering, filtration, insulation, as sensors for energy storage and for catalysis.

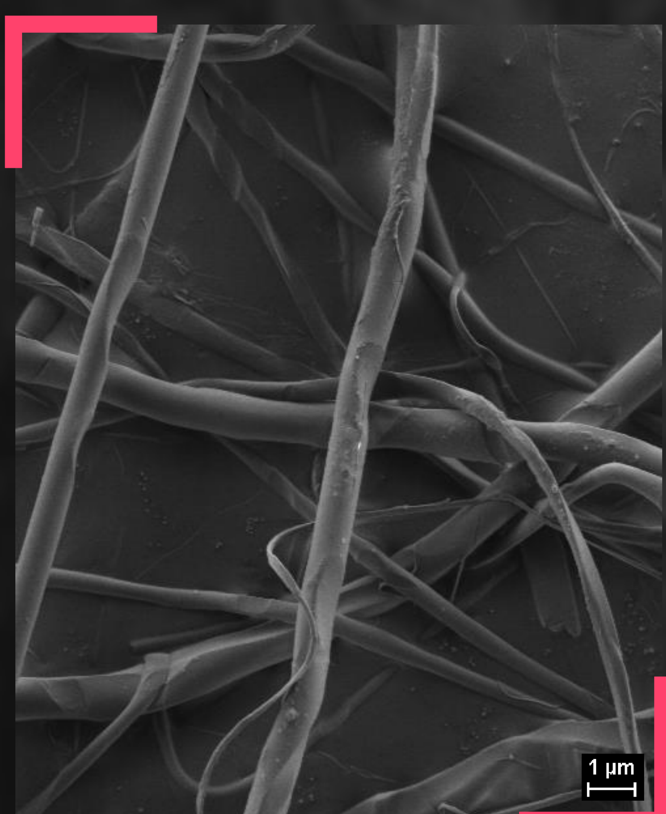


Figure 1. Fe/PVP Nanofibers under Scanning Electron Microscope (SEM) before thermal treatment

EXPERIMENTAL

PREPARE PRECURSOR 1.

The precursors are composed of ~70%wt. of solvents (Ethanol and Acetic acid) and 30% wt. of the polymer (Polyvinylpyrrolidone or PVP) and organic metal/s complex of $Fe(acac)_3$ and/or $Al(acac)_3$.

Different composition were used in order to test the influence on final morphology.

The solution is stirred until homogeneous, and its viscosity is measured.

The solution is now ready for electrospinning.

ELECTROSPINNING 2.

Electrodes generate an electric field between the needle and the collector.

The electric force generated overcomes the solution's surface; forming a liquid jet. The repelling forces due to like charges between molecules cause instabilities which reduce the diameter of the jet. At the same time, the solvents evaporate.

The jet is attracted to and collected at the rotating collector, where it forms the nanofiber mat.

Modifying certain parameters of the setup can change the morphology of the produced mat (these include the distance between the needle and collector, the rotation speed, voltage, type of needle and pumping rate).

Other components which can affect the morphology include the temperature, humidity, viscosity of the solution and the chemicals it incorporates.

The electrospun mat (Fig.1) is ready for thermal treatment.

THERMAL TREATMENT 3.

The mat is placed in a vacuum chamber to dry it.

When dry, the mat is removed from the collector and placed inside a furnace which is heated in a specific profile based on thermal analysis.

After the thermal treatment, the organic materials in the fibers are burnt and the mat turns ceramic.

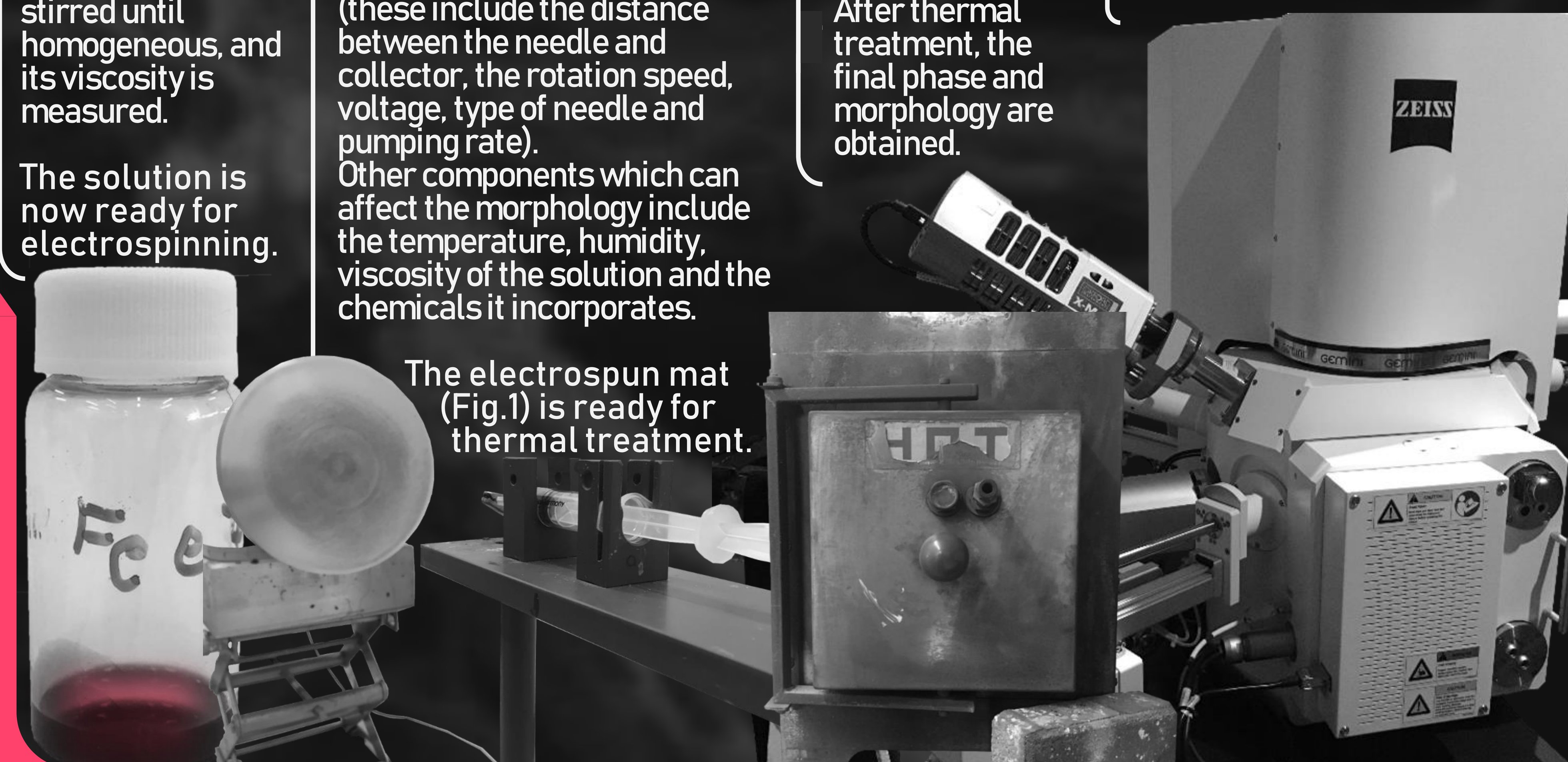
After thermal treatment, the final phase and morphology are obtained.

FINAL MORPHOLOGY 4.

The Scanning Electron Microscope (SEM) is used to observed the fibers' morphology. The phase is determined using X-ray diffraction (XRD).

Fiber morphology refers to the structure (length, diameter) and the shape (porous/smooth), as well as the shape of the pores.

The morphology of the fiber affects its physical and chemical properties.



RESULTS

The following are close-up images taken under the Scanning Electron Microscope (SEM) of the different fibers we created.

Al-O

Figure 2
0.8 Al/PVP

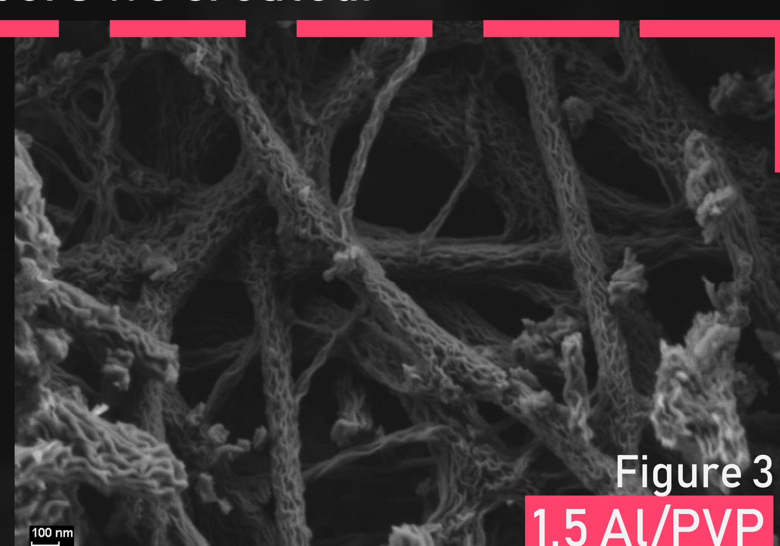


Figure 3
1.5 Al/PVP

Ratios 0.5, 0.8 and 1 (of Al to PVP) all showed similar morphologies (Fig.2). A higher concentration of Al led to a lamellar-like morphology. Solubility limit is reached at higher ratios (such as 1.8 Al/PVP) – therefore electrospinning cannot be preformed. XRD results showed the amorphous phase.

Fe-O

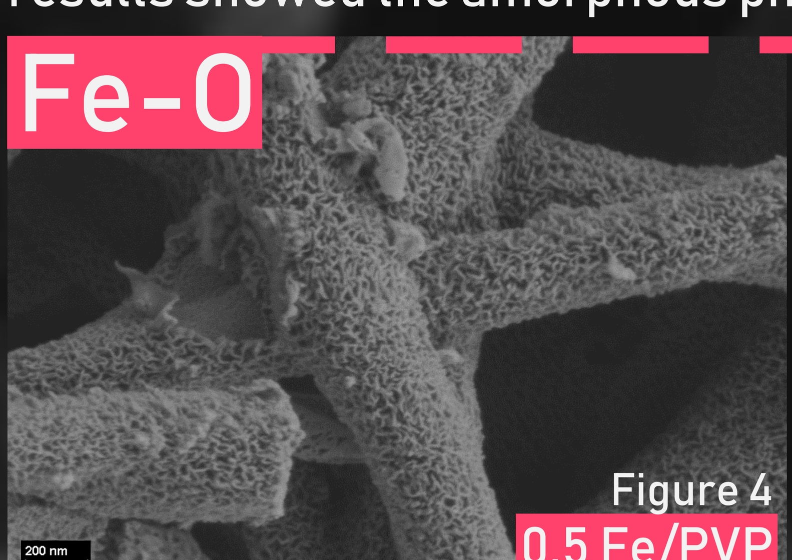


Figure 4
0.5 Fe/PVP

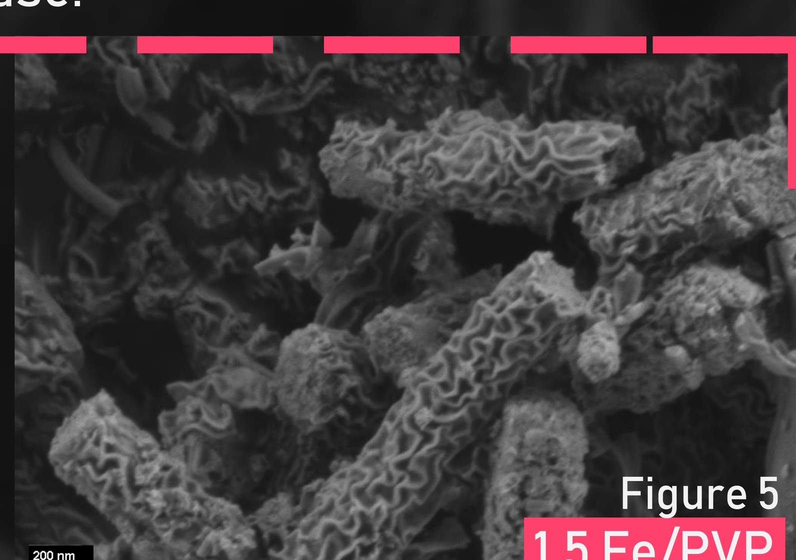


Figure 5
1.5 Fe/PVP

All ratios of Fe/PVP produced lamellar-like morphology, however, increasing Fe concentration showed to cause a less dense lamellar structure, with more spaces (Fig.4 and Fig.5). Ratios of at least 1.8 produce a less stable electrospinning and produce fibers with more impurities. XRD results showed phases of Iron Oxide (either Fe_2O_3 or Fe_3O_4).

Fe-Al-O

Figure 6
1:4 Fe/Al

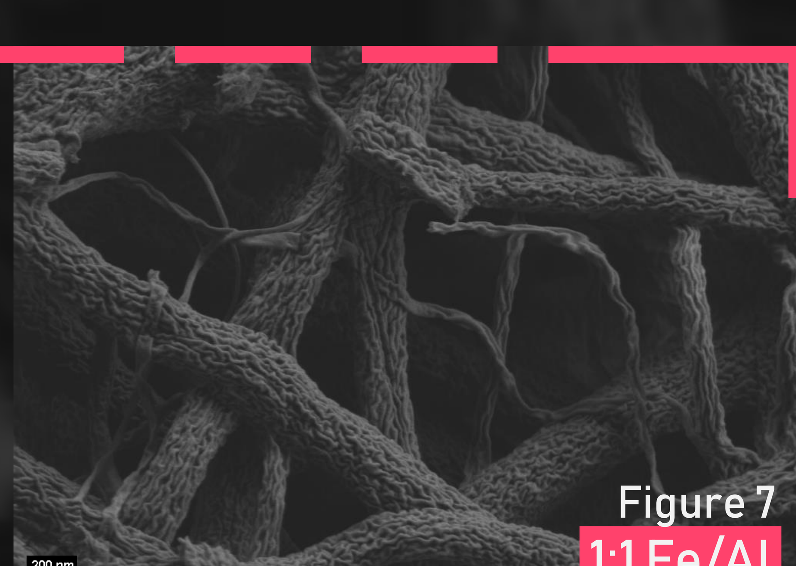


Figure 7
1:1 Fe/Al

In the Fe-Al solutions, the ratio between the Fe+Al to PVP was kept constant at 1.5 (optimal ratio from previous knowledge). Changing the Fe/Al ratio did not make a major difference to the morphology of the fibers (Fig.6 and Fig.7). Lamellar-like structure was achieved in all Fe-Al-O fibers. Note the long fibers and small number of impurities. Previous experiments showed that this kind of fiber has a spinel structure and the chemical composition of Fe $(Fe_xAl_{1-x})_2O_4$.

SUMMARY

- ✓ Three different types of nanofibers (Fe-O, Al-O and Fe-Al-O) were produced by electrospinning.
- ✓ Changes in the fibers' morphology caused by modifying chemical composition were studied (ratios of Fe/PVP, Al/PVP and Fe/Al).
- ✓ Low ratio of Al to PVP (1 or less) produces fibers with a plain surface, while a higher ratio of Al to PVP (1.5, but <1.8) produces a lamellar-like surface (Al-O fibers).
- ✓ Higher Fe concentration causes larger spaces in lamellar-like surface (Fe-O fibers).
- ✓ Adding Al to Fe-O nanofibers does not affect the morphology of the Fe-Al-O nanofibers, meaning that the structure is always lamellar-like (as long as Fe+Al/PVP ratio is kept constant at 1.5). However, it affects the fibers' chemical and physical properties.
- ✓ Future research could test the fibers' potential usage as catalysts in the Fischer-Tropsch reaction for the purpose of creating liquid fuel out of CO_2 and H_2.

REFERENCES

[1] Lu, Xiaofeng, Ce Wang, and Yen Wei. "One-dimensional composite nanomaterials: synthesis by electrospinning and their applications." *Small* 5.21 (2009): 2349-2370.

[2] Landau, Miron V., Roxana Vidruk, and Moti Herskowitz. "Sustainable production of green feed from carbon dioxide and hydrogen." ChemSusChem 7.3 (2014): 785-794.

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