Bin Yuan[1],
Jun Liu[1],*,
Lei Qiu[1],
Qian Hui[1],
Meng-Jie Chang[1],
Ya-Qing Li[2]

**[1] Xi'an University of Science and Technology,
Department of Materials Science and Engineering,**
Xi'an 710054, People's Republic of China,
* e-mail: jun_liu@xust.edu.cn

**[2] Xi'an University of Science and Technology,
School of Safety Science and Engineering,**
Xi'an 710054, China

Effect of MoS$_2$ Nanosheet Fillers on Poly(vinyl alcohol) Nanofibre Composites Obtained by the Electrospinning Method

Abstract

The graphene-like two dimensional (2D) inorganic materials have been been shown great interest for a variety of applications. In this work, polymer composite nanofibres containing molybdenum disulfide (MoS$_2$) nanosheets were obtained by electrospinning. The MoS$_2$ nanosheets were well dispersed inside the fibres, and the nanofibres maintained the fibre morphology well with the MoS$_2$ nanosheets embedded. The incorporation of MoS$_2$ nanoshe-ets changes polymer nanofibre morphology from round to ribbon-like. Moreover, through thermogravimetric (TG) analysis and dynamic mechanical thermal analysis (DMTA) measu-rements, it was found that the MoS$_2$ nanosheets as an additive material led to an increase in thermal stability and in the storage modulus. This work comprises an extensive approach to producing a novel 2D inorganic-organic composite structure, which should be applicable for membrane engineering with enhanced thermal and mechanical stability.

Key words: *electrospinning, MoS$_2$ nanosheet, poly(vinyl alcohol), composite, nanofibres.*

Introduction

Electrospinning has been demonstrated to be a simple and versatile technique for producing fibres and networks from polymer and composite materials. Such fibres and porous polymer membranes allow wide engineering applications, e.g., tissue engineering, water filtration, and energy and environment-related. In particular, many inorganic materials, such as carbon nanotube and graphene, have been used as nanofillers to improve the performance of fibre composites. Accordingly, it is desired that some new materials with attractive properties be prepared as nanofillers in fibres by electrospinning.

The molybdenum disulfide (MoS$_2$) nanosheet is an attractive inorganic graphene analogue (IGA) among the various 2D materials. The basic unit of MoS$_2$ is comprised of two layers of sulfur atoms, forming a sandwich structure, with a layer of molybdenum atoms in the middle. Usually, the bulk MoS$_2$ material is made of 2D layers stacked together by weak van der Waals interaction, which allows to obtain MoS$_2$ layers via chemical or mechanical exfoliation of bulk material. The MoS$_2$ layers exhibit unique properties like optical, luminance, mechanical and electrical, making them attractive for the application of optics, sensors, electronics, catalysers and transistors. The fascinating properties of the MoS$_2$ layers make it desirable to investigate whether MoS$_2$ would be good a nanofiller for electrospun nanofibres. Previously,

many reports have shown the compatibility of MoS$_2$ nanosheets with polymers by incorporating them into a variety of polymer matrices. For example, composites of MoS$_2$ and polyanilines or polythiophene were fabricated to study their electronic properties . Recently, Coleman and other researchers prepared MoS$_2$ nanosheets by liquid exfoliation and obtained composites with different polymers. It has been suggested that MoS$_2$ nanosheets could enhance the thermal, mechanical and fire safety properties of the composites. To date, most of the aforementioned researches on MoS$_2$-polymer composites have been based on dense film composites prepared by the casting process. Fabric nanocomposites containing MoS$_2$ nanosheets have hardly been reported.

In this work, we successfully prepared 1D poly(vinyl alcohol) (PVA) nanofibres containing 2D MoS$_2$ nanosheet materials via the electrospinning process. We mainly focused on what effect the 2D MoS$_2$ nanosheets would have on the nanofibre. PVA was chosen as the electrospun polymer for its biocompatibility, biodegradability and water-solubility. We undertook MoS$_2$ nanosheet dispersion in water, and then the PVA/MoS$_2$ composite solution was electrospun into nanofibres. The morphology and structure of the fibres are strongly dependent on the electrospinning parameters and the properties of the spinning solution. The influence of process conditions on the nanofibre morphology were systematically investigated, including PVA concentrations of the spinning solution and the MoS$_2$ filler concentration, as well as the applied voltage. More-

over, thermal and mechanical properties of the composite fibres were studied.

Experimental

Materials

Molybdenum disulfide (MoS$_2$) powder and n-butyllithium (n-BuLi) were purchased from Sigma Inc. Polyvinyl alcohol (PVA, 87-89% hydrolysed, M.W = 88,000-97,000) was obtained from Alfa Aesar Inc. The high voltage supply for electrospinning was from Dongwen Inc. (Tianjing, China).

Preparation of MoS$_2$ nanosheets

MoS$_2$ nanosheets were prepared by the Li$^+$ intercalation and exfoliation process. Typically, 100 mg of dry MoS$_2$ powder was added into 5 ml of dry hexane in a nitrogen atmosphere. Then 10 ml of n-BuLi was gradually dropped into the mixture at -78 °C. Then the solution was slowly heated to reflux and reacted for 72 h. The intercalated samples were washed with hexane several times to remove any unreacted n-BuLi. The intercalated sample was exfoliated by ultrasonication with DI water in a closed vial, during which process profuse gas was released and an opaque suspension of the nanosheets was formed. The suspension was dialysed for 72 h with DI water in a dialysed bag (450 nm).

Nanofibre fabrication by electrospinning

The spinning solution for electrospinning was prepared by dissolving PVA in water at 80 °C and subsequent mixing

with different amounts of MoS_2 solution. The solution obtained was transparent with a light brown colour, indicating good dispersion of the MoS_2 nanosheets in the PVA solution. For the electrospinning process, the needle of a solution loaded syringe was connected to a high-voltage supply. The distance between the tip of the needle and the collector was set at 10 cm. The feed rate of the spinning solution was set at $5 \cdot L\ min^{-1}$ by a syringe pump. The fibres were collected on an Al foil negative electrode under certain applied potential. The morphology of the fibres could be adjusted by changing various parameters of the fabrication process, such as PVA, MoS_2 concentrations and the applied voltage. All the samples prepared at different parameters are listed in **Table 1,** labelled with ticks. The concentration of PVA and MoS_2 represent their weight ratio to the total spinning solution.

Characterisation

Transmission electron microscopy (TEM, Tecnai-G2-F30) were performed by directly spinning fibres on a TEM grid. The morphology of the fibres was characterised using a scanning electron microscope (SEM, Hitachi S-4800) and atom force microscopy (AFM, Agilent 5400). X-ray diffraction (XRD, XRD-6000 Bruker) were conducted at an interval of 0.2 degree. Thermogravimetric (TG) analysis was performed at a heating rate of $10\ °C\ min^{-1}$ (1090B TG instrument, DuPont Inc). Dynamic mechanical thermal analysis (DMTA) was performed using a DMTA Q800 (TA, USA). Fibre samples were cut to 5×20 mm with a thickness of around 30 μm.

■ Results and discussion

Miromomophology of MoS_2 nanosheets and PVA/MoS_2 fibres

Typical TEM images of MoS_2 nanosheets by the Li^+ intercalation and exfoliation process are shown in **Figure 1.a**. It is obvious that 2D MoS_2 nanosheets of several hundred nanometers diameter were obtained. Actually, MoS_2 nanosheets with a wide range of diameters were prepared by this process. MoS_2 nanosheets obtained by this method are highly crystallised, which can be confirmed by the clear lattice feature of (100) in **Figure 1.b**. The typical morphology of the fibres produced (14% PVA, 0.025% MoS_2, 12 kV) was measured by SEM. It is sh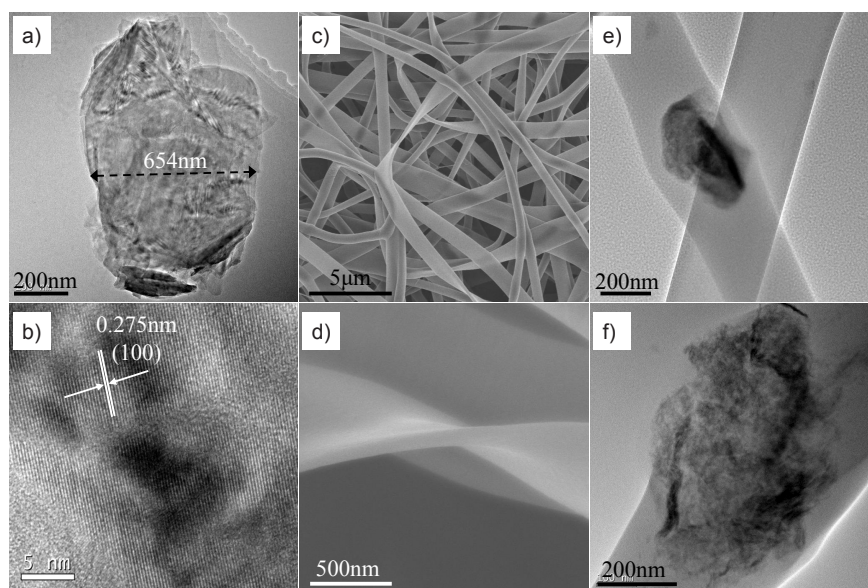own that the PVA/MoS_2 fibres have a smooth surface without obvious protuberance on the fibre (**Figure 1.c**). The average diameter of the nanofibre is around 600 nm. Interestingly, the fibre structure shows ribbon-like morphology, in contrast to the round shape of commonly reported nanofibres. This ribbon-like structure can be clearly observed from the side view of the fibre (**Figure 1.d**), in which the width is around 1.5 μm. while the thickness is less than 100 nm. In particular, the ribbon-like fibre in **Figure 1.d** is suspended but not adhered to the substrate, which indicates that this morphology is not directly caused by the substrate effect. The PVA/MoS_2 fibre produced was also characterised by TEM measurement (**Figure 1.e and 1.f**). The MoS_2 sheets are apparently embedded inside the PVA fibres. Importantly, with the existence of MoS_2 sheets, there is a larger diameter of the composite fibre. The results in **Figure 1** imply the important role of MoS_2 in the morphology of PVA/MoS_2 composite fibres.

Figure 1. a-b) TEM images of MoS_2, c-d) SEM and e-f) TEM images of PVA/MoS_2.

Table 1. Parameters of voltage, MoS_2 and PVA concentrations for the fibre preparation.

| Samples | MoS_2 (w/w) and PVA concentration | Voltage, kV | | | |
		6 kV	8 kV	10 kV	12 kV
PVA nanofiber	PVA: 14%	✓	✓	✓	✓
PVA/MoS_2 nanofiber	MoS_2: 0.0125% PVA: 14%				✓
	MoS_2: 0.025% PVA: 14%	✓	✓	✓	✓
	MoS_2: 0.05% PVA: 14%				✓
	MoS_2: 0.025% PVA: 8%				✓
	MoS_2: 0.025% PVA: 16%				✓

Influence of spinning voltage on the morphology of PVA and PVA/MoS_2 fibres

To check the voltage effect, we firstly produced PVA nanofibres at different voltages: 6, 8, 10 & 12 kV, using 14% PVA solution. For all the applied voltages, there are stable injections for the electrospinning, and smooth fibres are collected on the negative substrate, which means that the electrical repulse force induced by the high voltage can balance the surface intension. The fibre collecting yield at 12 kV is obviously higher than that at 6 kV, due to the high electrical charge on the Taylor cone surface. The nanofibre morphology gradually changed from ribbon-like to a round shape as the electrical potential increased from 6 to 12 kV (**Figure 2.a-2.d**). In previous reports, Satya Shivkumar *et. al* obtained a ribbon-like shape of PVA fibres at 30 kV/10 cm by increasing the concentration or molecular weight of the PVA, in which the high
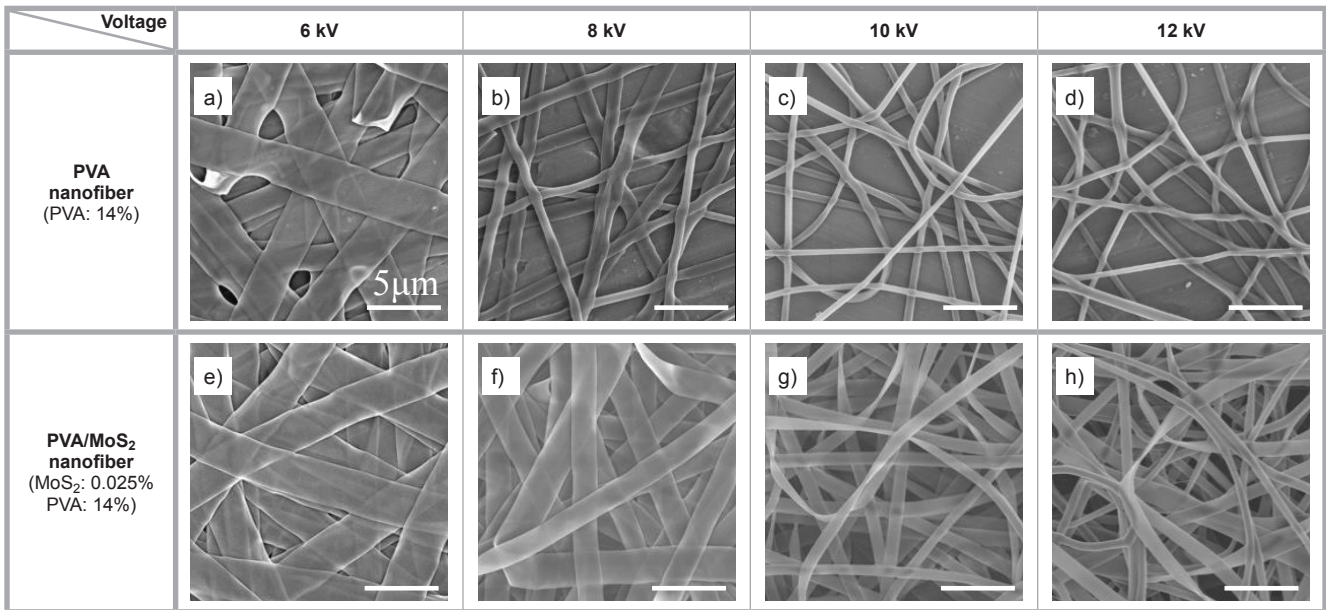
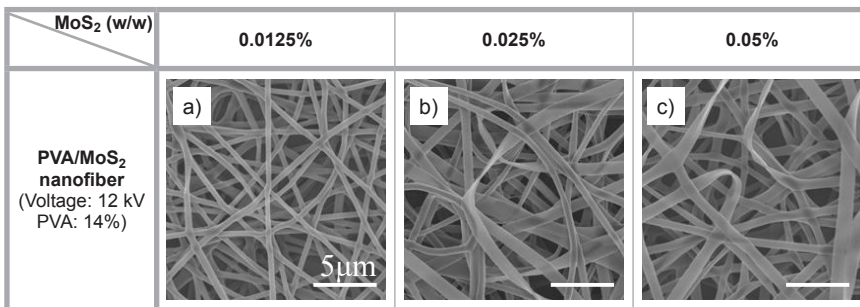**Figure 2.** SEM images of PVA and PVA/MoS$_2$ fibres at different voltages.

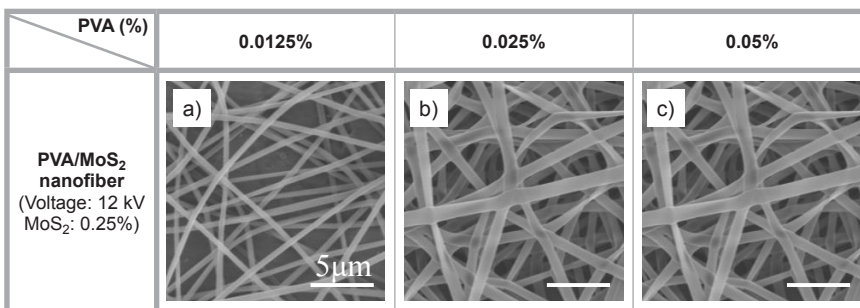**Figure 3.** SEM images of PVA/MoS$_2$ fibres at different MoS$_2$ concentrations.

Figure 4. SEM images of PVA/MoS$_2$ fibres at different PVA concentrations.

viscosity of the spinning solution is the main factor.

When MoS$_2$ nanosheets were added into the PVA solution, a remarkable difference was observed compared with plain PVA fibre (***Figure 2.e-2.h***). Firstly, at voltages from 6-10 kV, all the fibres presented a regular ribbon-like shape. Especially, at a high voltage of 10 kV, ribbon-like fibres are still very uniform in width. When the voltage was increased to 12 kV, the width of the fibres

was not very uniform, and there was the appearance of a small number of round fibres; however, most fibre remained of ribbon-like morphology. Moreover, the average width of the PVA/MoS$_2$ fibre is 2.9, 1.5, 0.9 & 0.75 μm, obtained at 6, 8, 10 & 12 kV, respectively, which is larger than that that of plain PVA fibre obtained at the same voltage. ***Figure 2*** suggests that PVA/MoS$_2$ composite fibres present a ribbon-like shape more than pure PVA, which probably resulted from the surface tension change of the polymer solution.

This morphology change also demonstrated the large interaction between the nanosheets and PVA.

Influence of MoS$_2$ concentration on the morphology of PVA/MoS$_2$ fibres

We also checked the effect of MoS$_2$ nanosheet concentration on PVA/MoS$_2$ morphology, in which the PVA concentration was kept at 14%. From the SEM results (***Figure 3***), it was found that a certain quantity of MoS$_2$ nanosheets is favorable for yielding a ribbon-like structure. For example, when the MoS$_2$ concentration is 0.0125% (***Figure 3.a***), the fibres obtained have a round shape similar to pure PVA fibres. While using a 0.025% (***Figure 3.b***) or higher MoS$_2$ concentration (***Figure 3.c***), the fibre shape presents ribbon-like morphology.

Influence of PVA concentration on the morphology of PVA/MoS$_2$ fibres

For electrospun PVA nanofibres, it is generally concluded that high polymer concentration may result in a ribbon-like structure. Herein, different PVA concentrations were applied to study the morphology of PVA/MoS$_2$ fibres, where the MoS$_2$ concentration was kept at 0.025%. For the 8% PVA solution (***Figure 4.a***), we only obtain a round shape of PVA/MoS$_2$ fibres, even when a low voltage of 8 kV was applied. When a higher concentration of 14 and 16% PVA were used (***Figure 4.b and 4.c***), the fibres showed a ribbon-like morphology. ***Figure 3*** and ***Figure 4*** indicate that the concentrations of both PVA and MoS$_2$ are important to

PVA/MoS$_2$ fibre morphology. In our experimental condition, ribbon-like fibres were obtained only when the PVA and MoS$_2$ concentrations were higher than 14% and 0.025%, respectively.

AFM analysis

The fibre morphology was further characterised by the tapping mould AFM technique. Nanofibres from the spinning solution (14% PVA, 0.025% MoS$_2$) were collected on a clean and hydrophilic silica wafer and dried under a lamp bulb. Before AFM measurement, the silica with fibres was submerged in a crosslink solution (glutaraldehyde in ethanol) for 24 h, and then dried in air. This treatment fixed the fibres tightly to the silica substrate without destroying the original morphology or size of the fibres. It is indicated that the fibres obtained at different voltages possess a remarkable ribbon-like structure (***Figure 5***). The width is much larger than the thickness of the fibres. Interestingly, the thickness at the two edges is larger than that in the central domain. This phenomenon was also observed with other polymers before. As the voltage increases, the fibres obtained become much narrower and thicker, which is consistent with the results of SEM measurement.

XRD and TG analysis

X-ray diffraction (XRD) was used for determining whether MoS$_2$ nanosheets were indeed present as separated sheets in the nanofibres. In the XRD patterns (***Figure 6.a***), pure PVA exhibits a broad diffraction peak around 19.51°, resulting from its (101) crystal planes, and the strongest peak (2θ = 14.4°) was observed
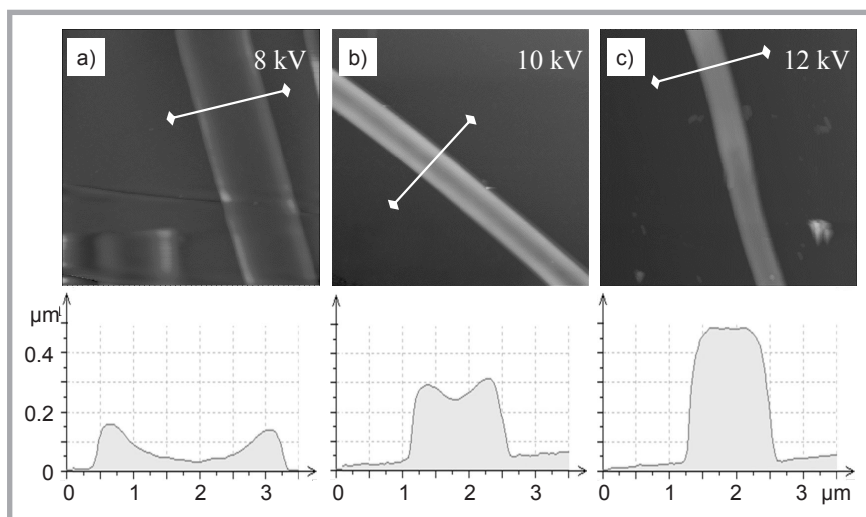

***Figure 5.** AFM images and profiles of PVA/MoS$_2$ fibre prepared at different voltages.*

corresponding to the (002) plane of bulk MoS$_2$ (JCPDF No: 24-0513). After the MoS$_2$ nanosheets were electrospun into PVA nanofibres, the XRD pattern of the PVA/MoS$_2$ nanocomposite only showed the diffraction peak of PVA, with no such observed for MoS$_2$ powder. It can be seen that no bulk MoS$_2$ exists in the nanofibres and that the MoS$_2$ nanosheets are separated from each other with no aggregation. Furthermore, TG analysis was used for rapid evaluation of the thermal stability for various polymers (***Figure 6.b***). Both pristine PVA and PVA/MoS$_2$ nanofibres decompose in a two-step process. When the first major weight loss occurs at ~330 °C, the decomposition pattern and mass loss (~58%) are nearly the same for the two samples, which can be attributed to be the degradation of PVA chains. The second mass loss stage occurs from 360 to 470 °C, and is due to

dehydration in the PVA chains. However, the mass loss of the PVA/MoS$_2$ nanocomposite is slower compared to that of pure PVA at the second stage. The mass loss of the composites is only around 82%, which is obviously lower compared with the mass reduction of 91.5% for pure PVA fibres at 470 °C. The TG analysis results suggest that the existence of MoS$_2$ nanosheets inside PVA/MoS$_2$ would retard the thermal degradation of PVA chains, which might be attributed to the limit of the motions of polymer chains through the interactions between MoS$_2$ nanosheets and PVA.

DMTA analysis

To better understand the PVA/MoS$_2$ interaction, we also performed dynamic mechanical thermal analysis (DMTA) measurements for pure PVA and PVA/MoS$_2$
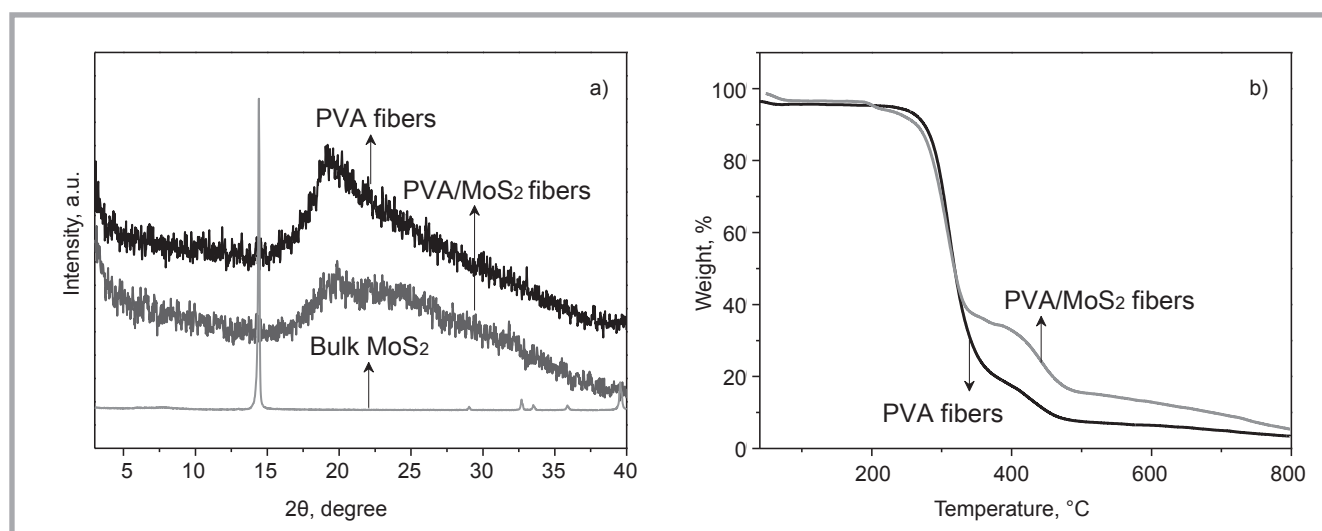

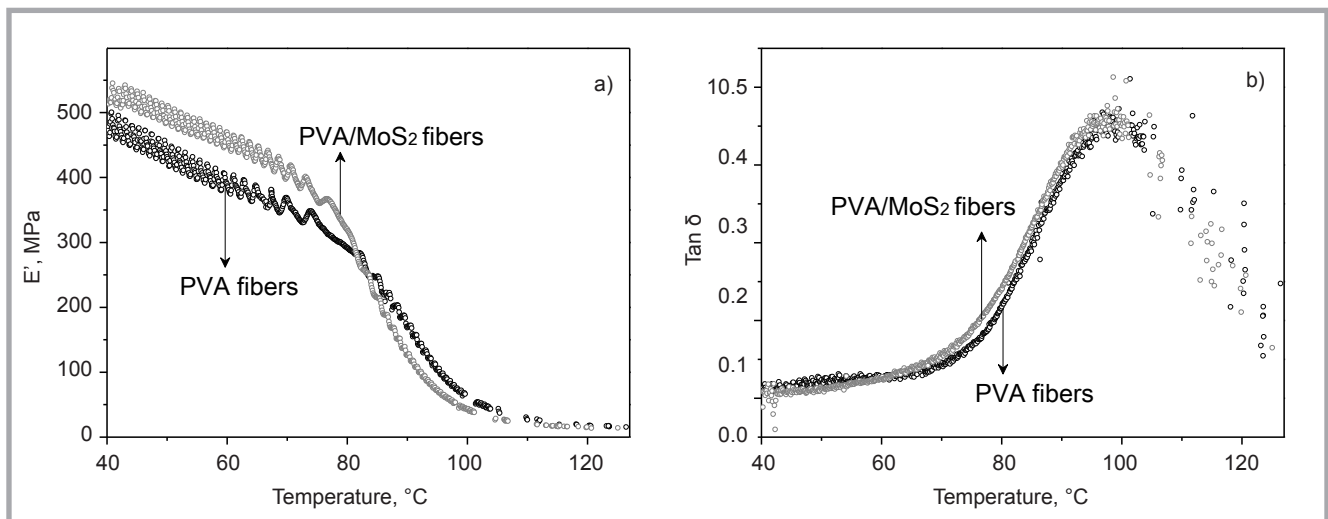***Figure 6.** a) XRD patterns, b) TG plots of fibre samples.*

Figure 7. a) Storage modulus (E') and b) tan δ of PVA nanofibre and PVA/MoS₂ composite nanofibres as a function of the temperature in the tensile mode (frequency = 1 Hz).

composite nanofibres. In the DMTA test, the storage modulus (E') is a measurement of the stiffness, which is proportional to the energy stored elastically. The loss modulus (E"), on the other hand, measures the energy transferred to heat. The temperature at the maximum of tan δ is usually taken as the glass transition temperature (Tg). The storage modulus of a polymer decreases rapidly, whereas the loss modulus and tan δ reach a maximum when the polymer is heated up through the Tg region. The samples for DMTA measurement were prepared at the following condition: 14% PVA, 0.025% MoS₂ and 12 kV. As can be seen, the PVA/MoS₂ fibres show an 7.5% increase in the storage modulus E' compared with that of pure PVA fibres at 40 °C (*Figure 7.a*). A similar increase in the elastic modulus of PVA polymer films made from 2D materials was reported. It can be observed that the Tg value of PVA is around 98.7 °C at the maximum of tan δ (*Figure 7.b*), which is consistent with the value in the literature. In contrast, no obvious change in Tg was observed for the PVA/MoS₂ nanofibres, which was perhaps due to the low weight percentage of the embedded MoS₂ nanosheet. The DMTA results show that the storage modulus of PVA fibres could be improved with MoS₂ nanosheets.

■ **Conclusions**

We succeeded in synthesising MoS₂ nanosheets and producing PVA/MoS₂ composite nanofibres by the electrospinning proess. In comparison with pure PVA polymer fibres, the PVA/MoS₂ fibres

obtained tend to form a ribbon-like morphology. The PVA and MoS₂ concentration, as well as the spinning voltage applied are important for the formation of ribbon-shaped PVA/MoS₂ composite fibres. The higher the concentration of PVA and filled MoS₂ and the lower the spinning voltage, the easier the formation of ribbon-shaped PVA/MoS₂ composite fibres. Moreover, the incorporation of MoS₂ nanosheets can improve the thermal stability and enhance the storage modulus of the composite nanofibres. This work comprises an extensive approach to producing composite nanofibres containing other 2D inorganic materials, such as BN, NbSe₂, Co₉Se₈ and MoSe₂.

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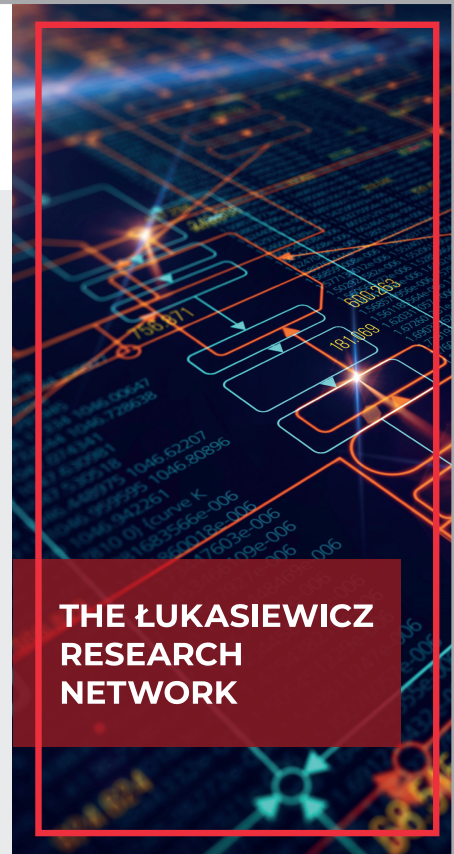
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- Institute of Biopolymers and Chemical Fibres
- The Institute of Biotechnology and Antibiotics
- The Institute of Ceramics and Building Materials
- Industrial Chemistry Research Institute
- Institute of Heavy Organic Synthesis BLACHOWNIA
- The Electrotechnical Institute
- Pharmaceutical Research Institute
- Institute for Engineering of Polymer Materials and Dyes
- Institute of Logistics and Warehousing
- Institute of Aviation
- The Institute of Precision Mechanics
- Institute of Mechanised Construction and Rock Mining
- Institute of Non-Ferrous Metals
- Stanisław Staszic Institute for Ferrous Metallurgy
- KOMEL Institute
- New Chemical Syntheses Institute
- Metal Forming Institute
- Foundry Research Institute
- Maksymilian Pluta Institute of Applied Optics

- Institute of Organization and Management in Industry ORGMASZ
- Rail Vehicles Institute „TABOR"
- Institute of Industrial Organic Chemistry
- Institute of Leather Industry
- The Polish Centre for Welding Technology
- The Institute of Innovative Technologies EMAG
- Institute of Medical Technology and Equipment
- Wood Technology Institute
- The Institute for Sustainable Technologies – National Research Institute
- Institute of Electron Technology
- Institute of Electronic Materials Technology
- The Tele and Radio Research Institute
- Textile Research Institute
- The Institute of Advanced Manufacturing Technology
- Industrial Research Institute for Automation and Measurements PIAP
- Industrial Institute of Agricultural Engineering
- Automotive Industry Institute
- PORT Polish Center for Technology Development

A cure against excessive fragmentation

Until recently, there were 111 research institutes in Poland, operating under the supervision of 16 ministers. This led inevitably to fragmentation of human, financial and intellectual resources. The institutes were not able to achieve what is know as the 'economies of scale'. The Łukasiewicz Research Network offers uniform and simple rules of cooperation with businesses, commercialisation of knowledge, development and use of academic infrastructure. More cooperation instead of destructive competition and fragmented resources.

ADDITIONAL INSTRUMENTS TO SUPPORT INNOVATION

National Centre for Research and Development: comprehensive and effective R&D support

- Development of support programmes for applied research and R&D activities
- State and EU funds for commercialisation of research and transfer of the results into the economy
- Support for young researchers

Good legislation for innovation – tax reliefs and support to reinforce cooperation between the research and business sectors

- The first Innovation Law, regarded as the best legislative act of 2017 in the area of taxation (according to the (De)Regulation Report published by the Employers of Poland organisation)
- The second Innovation Law, in force since 1 January 2018, which increases the R&D tax relief up to 100%, makes it easier to finance start-ups, enables the HE institutions and Polish Academy of Science bodies to establish companies in order to manage research infrastructure

Industrial doctorate – Cooperation between research and business sectors

- Doctoral theses are of practical nature and the results may be implemented nearly immediately
- Companies are able to deduct 100% of staff expenses related to the employment of Ph.D. students
- Ph.D. students are able to develop their professional and academic careers at the same time