Needleless electrospun polyacrylonitrile/konjac glucomannan nanofiber mats

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Abstract
In this study we report for the first time about the preparation of polyacrylonitrile (PAN)/konjac glucomannan (KGM) nanofiber mats, needleless electrospinning from the low-toxic solvent dimethyl sulfoxide (DMSO) and the formation of carbon nanocomposites. Konjac glucomannan is a biopolymer and renewable, environmentally friendly raw material and a well-known polysaccharide, which is non-toxic and biocompatible material and is extracted from the Amorphophallus konjac plant. The addition of poloxamer in electrospinning PAN/KGM solution resulted in the reduction of membrane areas and decrease of beads in nanofibers. The concentration of 1.5% or 0.5% of konjac glucomannan in PAN/KGM nanofiber mats was not detected to affect the morphology of the nanofiber mats. The PAN/KGM nanofiber mats received oxidative stabilization and subsequent carbonization. It could be observed that after the oxidative stabilization process the average diameter of PAN/KGM nanofibers increased and after carbonization decreased compared to stabilized nanofibers. Alternative renewable raw materials such as KGM electrospun with synthetic polymers offer the possibility to reduce the environmental impact and are the alternative to new technical materials and lowers the cost of carbon materials. The combination of PAN with konjac glucomannan and the properties of both polymers open up a wide range of applications for the PAN/KGM nanofiber mats and carbon nanocomposites produced in this study, for example, for pharmaceutical and biomedical applications, as absorbents for the removal of pollutants in wastewater and as filter media for air purification, as well as for optical and chemical sensors.

Keywords
Wire-based electrospinning, PAN nanofibers, morphology, konjac glucomannan, dimethyl sulfoxide, PAN/KGM nanocomposites

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Introduction
The electrospinning technique is preferable to produce ultra-thin nanoscale fibers or fiber composites in the nanometer range¹,² as well nanofibers or nanofiber composites can be produced from a variety of materials.³ The needle-free electrospinning technology enables the production of nanofiber mats on an industrial scale. Instead of a needle-based electrospinning, a wire is used for the production of nanofibers. The schematic illustration of needle-free electrospinning machine is shown in Figure 1. Wire-based
electrospinning works as follows: a polymeric solution is filled into a carriage which moves along the lower electrode wire and moistens it with electrospinning solution in a high-voltage field. The upper electrode wire is located on top and is shielded with a non-woven material. The high voltage field causes many Taylor cones to form along the wire so that nanofibers fly upwards and adhere on the non-woven, typically polypropylene.4

By modifying electrospinning parameters, various nanofiber morphologies can be produced as well as nano membranes, that is, non-fibrous mats, and mixtures of both morphologies. Changing the ratio of membrane to fiber areas is possible by varying the distance between the high-voltage electrode and the substrate.5 Diverse natural and non-natural polymers enable forming nanocomposites with inorganic or organic substances,6,7 ceramics8 or even producing metal-carbon composites9 that can be used in a wide range of applications such as for neuro-inspired engineering or drug delivery.13,14 Due to their large surface-to-volume ratio, nanofiber mats and membranes are also used for various other applications such as filter materials,15,16 optical and chemical sensors or catalysts,17 pharmaceutical and biomedical applications,18,19 food packaging20 or removal of pollutants in wastewater.21

Some natural polymers such as KGM cannot be wire-based electrospun solely and require a carrier polymer such as PAN or polyethylene glycol (PEG).

Konjac glucomannan (KGM) is a kind of natural neutral polysaccharide extracted from the Amorphophallus konjac plant22 and konjac oligosaccharides provide a high medicinal value.23 KGM has favorable properties such as very high-water absorption, high viscosity and high expansion rate and is widely used in the food industry.24,25 After processing from raw plant to flour, konjac contains 60% to 70% of glucomannan, a starch-like substance consisting of branched carbohydrate chains26 and KGM possesses the highest known water-binding capacity of all natural products.27 According to biomedical research, modified KGM is promising in anti-obesity therapies, has antidiabetic, anti-inflammatory and antimicrobial properties, and is favorable in wound treatment therapies.28,29

The poloxamers such as KGM also exhibit biocompatibility. It possesses temperature-dependent self-organization and thermoreversible behavior, which makes poloxamer-based biomaterials promising candidates for biomedical applications. Poloxamer belong to the class of synthetic triblock copolymers consisting of hydrophobic poly(propylene oxide) and two hydrophilic chains of poly(ethylene oxide).30 Adding poloxamer to other polymers during electrospinning can influence the morphology of the nanofiber.31

Due to its water stability and the possibility to spin it from the low-toxic solvent dimethyl sulfoxide (DMSO), polyacrylonitrile (PAN) is of great interest for many applications as a functional nanofiber mat and is a typical base material for producing carbon nanofibers using thermochemical conversion.32 On the other hand, conductive carbon nanofibers are used in many applications such as batteries, solar cells and filters.33 They can be obtained from PAN nanofibers which are oxidatively stabilized and then carbonized under inert gas atmosphere. By mixing PAN with other polymers or inorganic materials,34 various carbon nanofiber morphologies and nanocomposites can be produced and physical properties such as conductivity, absorption or surface structure and porosity can be changed to further increase the specific surface area.3

Furthermore, mixing PAN with different polymers and particles results in various composites with defined properties. Some polymers which cannot be produced with the wire-based electrospinning technology and DMSO as a solvent can be mixed to PAN or PEG and electrospun. Therefore, PAN is often used as a carrier material for the production of nanofibers with polymers that cannot normally be spun alone, such as KGM. KGM is a biopolymer and renewable, eco-friendly raw material, therefore not harmful to the environment35 and a cheaper source of carbon fibers compared to expensive petroleum, which is costly and has a limited rather than endless capacity. Due to the good biocompatibility of KGM, PAN/KGM nanofiber mats can be used in medical applications, for example, in wound dressings, or due to their good water absorption capacity in filter applications, or even for the production of carbon nanofibers.36 Experimental results of Hongbo et al.37 showed that the heat treatment destroyed the structure of KGM particles and reduced their crystalline degree and obviously the swelling capacity, but increased the acid and alkali resistance and the surface morphology changed to rough surface and these properties open up new applications of PAN/KGM nanofiber mats where these properties are in demand.

Carbon is mainly produced from petroleum-based PAN, as the carbon yield is relatively high compared to other carbon
sources. This fact is reflected in high costs for carbon fibers, and mineral oil reserves are also limited. Therefore, alternative renewable raw materials such as Pleurotus ostreatus mycelium or KGM electrospun with synthetic polymers offer the possibility to reduce environmental impact and are the alternative to new engineering materials.

In this study the influence of poloxamer on the morphology of the PAN/KGM nanofiber mats and the changes in diameter of the different nanofibers after thermal treatment will be investigated. Furthermore, carbon nanocomposites will be produced from PAN/KGM nanofiber mats.

### Materials and methods

The following solution for the production of nanofibers contained 16 wt.% PAN dissolved in DMSO. DMSO is an organosulfur compound and a colorless liquid and was selected as the solvent because of its low-toxic properties. The spinning solution for the nanofiber mat contained between 0.5% or 1.5% konjac rubber powder. In two types of nanofiber mats, 5% poloxamer was added. This polymer can have an influence on nanofiber morphology and is often used in typical biomedical applications such as drug delivery, skin tissue engineering or wound healing. A total of four different types of nanofiber mats were produced containing different amounts of konjac glucomannan and two of them containing poloxamer. For all electrospinning solutions, the polymer solution was stirred for 2 h on a magnetic stirrer at room temperature. Table 2 gives an overview of the nanofiber mats produced.

Nanofiber mats were produced with a needle-free electrospinning machine “Nanospider Lab” (Elmarco Ltd., Liberec, Czech Republic).

The following spinning parameters were used for electrospinning: high voltage 80 kV, nozzle diameter 0.9 mm, carriage speed 100 mm/s, lower electrode/substrate distance 240 mm, ground electrode/substrate distance 50 mm, temperature in the chamber 20.2°C, relative humidity in the chamber 33%. A relative humidity between 31% and 33% has proven to be suitable for the needleless electrospinning process of PAN. At higher humidity, PAN nanofiber mats tend to have an irregular morphology with some membrane parts. Electrospinning was performed for 30 min for all electrospun nanofiber mats. These spinning parameters were chosen because they proved to be optimal in earlier electrospinning experiments with PAN. Wire-based electrospinning typically uses high voltages in the range of 70 to 80 kV in contrast to needle-based electrospinning with characteristic voltages in the range of 20 kV.

The nanofibers after electrospinning were separated from the non-woven for the thermal treatment (Figure 2). The oxidative stabilization is a substantial process before carbonization and occurred in a muffle furnace B150 (Nabertherm, Lilienthal, Germany). According to previous investigations, a typical stabilization temperature of 280°C at a heating rate of 1 K/min, followed by an isothermal treatment at this maximum temperature for 1 h, is optimal for the nanofibers. A furnace (Carbolite Gero, Neuhausen, Germany) was used to carbonize nanofibers at the temperature of 500°C, approached by a heating rate of 10 K/min, in a nitrogen stream of 150 mL/min (STP), followed by an isothermal treatment for 1 h at the maximum temperature.

Scanning electron microscopy (SEM) was performed using a Zeiss 1450 VPSE (Oberkochen, Germany) with a resolution of 5 nm at a nominal magnification of 5000 × and all samples were sputtered with a 20 nm gold layer prior to SEM analysis. VHX-600K (Keyence, Neu-Isenburg, Germany) was used for optical images of the nanofiber mats with a confocal laser scanning microscope (CLSM). The software ImageJ 1.51j8 (from National Institutes of Health, Bethesda, MD, USA) was used for the fiber diameter investigations. The Fourier Transform Infrared (FTIR)

| Table 1. Overview of used materials. |
|-------------------------------------|
| **Materials** | **Manufacturer** |
| PAN | X-PAN, Dralon GmbH, Lingen, Germany |
| DMSO | min. 99.9%, obtained from S3 Chemicals, Bad Oeynhausen, Germany |
| Konjac glucomannan | Konjac rubber powder (Natural Gluten Free Thickener, premium quality, refined powder, from Special Ingredients Ltd., Chesterfield |
| Poloxamer | “Lutrol F 68,” 7680–9510 Da, 2 × 40% hydrophilic parts, BASF, Ludwigshafen am Rhein, Germany |
| Electrospinning substrate, polypropylene (PP) | Elmarco Ltd., Liberec, Czech Republic |

| Table 2. Overview of the produced nanofiber mats. | |
|-----------------------------------------------|---------------|
| Type of nanofiber mats | Composition of parts in electrospinning solution |
|-------------------------|-------------------------------------------------|
| 1 | 1.5% konjac glucomannan (KGM) |
| 2 | 0.5% konjac glucomannan (KGM) |
| 3 | 1.5% konjac glucomannan (KGM), 5% poloxamer |
| 4 | 0.5% konjac glucomannan (KGM), 5% poloxamer |

Nanofiber mats contain the 16 wt% PAN dissolved in DMSO.
spectrometer Jasco FT/IR-4100, (Jasco Deutschland GmbH, Germany) working with attenuated total reflection (ATR) with resolution 2 cm⁻¹, data interval 0.5 cm⁻¹, range 4000 to 700 cm⁻¹, averaging over 32 scans was used for detailed investigations of material chemistry and nanofiber morphology.

**Results and discussions**

Optical investigations were performed using CLSM. Different types of PAN/KGM nanofiber mats are shown in Figure 3. The CLSM images focus on the morphology of the nanofiber mats and show the changes of the nanofiber mat surface.

Figure 3(a) and (b) depict that the PAN/KGM nanofibers are mostly relatively smooth, straight and uniform in shape, with some beads and membranes visible. These beads are typically formed when electrospinning is performed from solutions based on DMSO or other slow evaporating solvents with relatively low solids content. DMF and DMAc solvents have much higher vapor pressures than DMSO which is generally supportive for electrospinning. Experiments have shown that increasing the solution temperature and thus the vapor pressure severely equalizes fiber diameters and reduces bead formation.⁴⁷ According to the results of the study by Tsivintzelis et al.,⁴⁸ the solvent systems acetone-DMA and acetone-DMF led to the production of uniform membranes with fibers of approximately the same size. On the contrary, the

![Figure 2. PAN nanofiber mats can be easily separated from PP non-woven.](image)

![Figure 3. CLSM images of PAN/KGM nanofiber mats. (a) Type 1 with 1.5% konjac. (b) Type 2 with 0.5% konjac. (c) Type 3 with 1.5% konjac and 5% poloxamer. (d) Type 4 with 0.5% konjac and 5% poloxamer.](image)
acetone-DMSO system led to the formation of structures with many interconnected fibers and larger fiber diameters, mainly due to the lower volatility of DMSO compared to the other solvents.

It can be seen that Figure 3(c) and (d) are different from the previous images 3(a) and 3(b) and only show a few beads and small membrane areas. This is probably due to the influence of the added poloxamer in the electrospinning solution according to the properties of this polymer. Due to the catechol-Fe interaction, the dopamine-modified Pluronic F68 showed high self-healing and elongation properties and has a temperature-dependent self-assembly and thermos-gelling behavior which may have influence the morphology of the nanofibers was influenced. In addition, poloxamers are commonly used for the production of thermo-reversible gels or improving the drug solubility.

Figure 4 shows the PAN/KGM nanofiber mats type 4 after oxidative stabilization (Figure 4(a)) and carbonization (Figure 4(b)). After the stabilization process, the usual brown color of the stabilized PAN becomes visible. CLSM images depict that the nanofiber mats show barely visible nanofibers in comparison with CLSM images on electrospun nanofibers, where the individual fibers are relatively well visible (cf Figure 3(d)). Apparently, the concentration of 1.5% or 0.5% of konjac glucomannan does not seem to have an effect on the morphology of the nanofiber mats, not even after thermal treatment. Therefore only the PAN/KGM nanofiber mats with 0.5% konjac glucomannan (KGM) and 5% poloxamer are investigated further.

Typically PAN is chosen as precursor for the production of carbon fiber due to high yield of carbon material. For the production of carbon nanofibers, thermal treatment is decisive for the later morphology of nanofibers. In the first step, this process involves oxidative stabilization, which is typically carried out between 260°C and 280°C to generate a structure suitable for high temperature treatment. In the second step, the carbonization is performed usually at 800°C. In this study, carbonization was carried out at 500°C to determine whether this temperature is sufficient for a biopolymer such as konjac glucomannan.

Oxidative stabilization is a heat treatment process to produce carbon fibers. During the stabilization process, PAN chains begin to crosslink, allowing the newly composed polymer structure to resist high temperature processing. Oxidative stabilization is crucial and responsible for the resulting shrinkage behavior of the nanofiber mats. During stabilization and carbonization processes, chemical and entropic shrinkage occurs.

In addition, volatilization of elemental carbon is minimized in the subsequent carbonization step, thus maximizing the final carbon yield. Oxidative stabilization reactions consist mainly of dehydrogenation and cyclization processes. During cyclization, nitrile groups (C≡N) are cross-linked and form chain molecules in the form of \(-C≡N=C≡N=-\). Several factors such as the pyrolysis temperature, heating rate, fiber tension, total stabilization time and dwell time, air flow rate and pre-stabilization treatment influence this stabilization process.

Figure 5 depicts the SEM images of PAN/KGM nanofiber mats before and after thermal treatment. The SEM investigations underline that the fibers are conglutinated during the stabilization process and form stable connection points which can also increase the mechanical properties since slippage between the fibers is prohibited. The SEM investigations emphasize that the individual fibers become thicker during the stabilization process. In addition, typical morphological changes from relatively linear shapes toward meandering fibers with adhesions along the intersections can be observed, resulting in more visible beads per area, and commonly occurring in PAN nanofiber mats as an effect of temperature treatment. When comparing the stabilized with the carbonized nanofiber mats, it should be noted that the dimensional changes are not so crucial. This underlines the assumption that the stabilization process was optimal and that the dimensional stability of the nanofibers is given after stabilization.
During the stabilization process, the nanofiber mats were not fixed and this resulted in stronger meandering of the fibers, which is supported by high temperatures and high heating rates. The diameter distributions of the nanofibers after electrospinning, oxidative stabilization and carbonization are shown in Figure 5. PAN/KGM nanofiber mats show a larger mean value as well as a wider distribution of nanofiber diameters after stabilization than after electrospinning. After carbonization, the average diameter of PAN/KGM nanofibers was again smaller than after the stabilization process. This effect was also observed in previous studies that the average diameter of PAN/magnetite nanofibers increased after stabilization but carbonization at 500°C resulted in smaller diameters in some cases and at 800°C in larger diameters again. In this study, carbonization at 500°C did not lead to an increase in diameter of the nanofibers, and this effect will be investigated further in subsequent studies.

The application of a mechanical tensile load during heat treatment can improve the mechanical properties of the nanofibers.
fibers. The stabilization of fixed nanofiber mats leads in most cases to straight, even fibers. This dimension stability effect can also be achieved by adding other polymers or particles to the PAN spinning solution. It must be mentioned that in previous studies with PAN the addition of other polymers or particles in some cases resulted in even and uniform fibers and in dimensional stability during stabilization and carbonization processes. In this case, the nanofibers do not shrink as much as typical PAN nanofiber mats, but form relatively straight, uniform carbon nanofibers. PAN/KGM nanofiber mats change their areal weight due to chemical stabilization and carbonization processes. The mass yield after stabilization is 72%, while after carbonization at 500°C, an overall carbon yield of 42% is reached. This result is qualitatively and quantitatively comparable with the study on the stabilization of temperature-dependent carbon yield of pure PAN samples or gelatin added to PAN.1,46

Besides these morphological investigations, Figure 6 shows the FTIR investigations of the PAN/KGM nanofiber mats. A stretching vibration of the nitrile functional group C≡N at 2240 cm⁻¹, a carbonyl (C=O) stretching peak at 1732 cm⁻¹ and ester (C–O and C–O–C) vibrations of the comonomers, bending and stretching vibrations of CH₂ at 2938 cm⁻¹, 1452 cm⁻¹ and 1380 cm⁻¹ are visible for the original nanofiber mat. The stretching peaks at 1730 cm⁻¹ can be attributed to the carbonyl of the aceto groups in KGM.61

Stabilization changes most of these peaks. Now, especially C≡N stretching vibrations at 1582 cm⁻¹ and C=C stretching vibrations at 1660 cm⁻¹ are visible, as well as C–H bending and C–H₂ wagging at 1360 cm⁻¹ are visible, while the peak at 1730 cm⁻¹ attributed to KGM has nearly vanished, indicating that this material is also chemically modified. Finally, the carbonized samples do not show the typical behavior of carbonized PAN nanofiber mats with only a few relatively flat peaks due to very few functional residual groups, which corresponds to the high absorption of carbon, but some peaks are still present, which leads to the conclusion that at 500°C no complete carbonization has occurred and a typical temperature of about 800°C is required for an efficient carbonization.9,59

Conclusion

In this study we report for the first time on the production of PAN/KGM nanofiber mats, needless electrospun using low toxic solvent DMSO, which are promising in various fields such as biomedical area, tissue engineering, cell growth, wound dressings, but also for filter applications due to the excellent water absorption and swelling properties and the bio-capability of KGM.

By adding poloxamer to the electrospinning solution the morphology of the nanofiber mat could be improved so that almost no beads and almost no membrane surfaces are visible. Unexpectedly, the different concentrations of konjac glucomannan do not affect the morphology of the nanofiber mats even after thermal treatment, which normally leads to an increase in the diameter of the nanofiber when other polymers are added to PAN nanofiber mats. These findings will be investigated in more detail in the near future.

In addition, PAN/KGM nanofiber mats were oxidatively stabilized and carbonized. Alternative renewable raw materials such as electrospun KGM with synthetic polymers have the potential to reduce environmental pollution and represent an alternative for new technical materials since they can be used in areas where conductive carbon nanocomposites are needed, such as battery storage, sensors or microelectronic parts.

Declaration of conflicting interests

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