Crystalline nanocellulose from Liberec significantly increases the efficiency of advanced filtration materials

Czech-Canadian teams are collaborating in research on nanofibrous materials suitable for the production of recyclable filters not only for water but also for other liquids capable of capturing microplastics.

Cooperation in research focused on using nanofibrous materials for technical applications is the main focus of collaboration between interdisciplinary scientific teams of the Technical University of Liberec (TUL) and the Canadian University of Waterloo, one of the world's top universities. It is essential for Liberec scientists that the Canadian university achieve excellent scientific results by using natural-based additives, which can be used to functionalise nanofibre carriers prepared at TUL. In addition to filters, these additives are also added to nanofibre filter membranes and carriers (scaffolds) developed and patented by Liberec academics as part of their research.

Foreign cooperation with universities in Canada was initiated by the Faculty of Mechanical Engineering of TUL (FME TUL) and according to the Dean of the Faculty, Associate Professor Jaromír Moravec, this partnership is one of its long-term strategic goals. It is based on the long-standing relationship between TUL and Conestoga Polytechnic, whose dean and great supporter was Professor Tony Martínek, a graduate of the Faculty of Mechanical Engineering who emigrated to Canada in 1967. Shortly after November 1989, he offered his alma mater advisory assistance and also facilitated exchanges of academics and students. In addition to teaching and exchange internships, the faculty also planned to develop cooperation in science and research.

"In May 2022, the team of Associate Professor Jan Valtera from the Department of Textile and Single Purpose Machines FME TUL initiated participation in the N4SNano network (International Network for Sustainable Nanotechnology). At the same time, we established cooperation with the University of Waterloo, also with the support of the Consulate General of the Czech Republic in Toronto, under the leadership of Ms Eva Libs Bartonová, and we have subsequently deepened our mutual cooperation. In 2022, Professor Sushanta Mitra visited us, and in his lecture, he presented the research areas at the Canadian university. Then, during his two-month internship, Canadian PhD student James Cheon was also involved in AC electrospinning research. His supervisor, Dr. Michael Tam, then conducted several seminars at our faculty on specialized topics in the field of nanomaterials. The University of Waterloo is one of the most prestigious universities in Canada and one of the top universities in the world with a wide range of specializations in the field of engineering and technology," said Associate Dean of the Faculty of Engineering at TUL, Associate Professor Jaromír Moravec, adding that this cooperation has moved to a higher level in the last year.

TUL surprised by the high level of nanotechnology research

During their visit to Liberec at the end of 2022, representatives of the University of Waterloo (UW) confirmed that the main focus of cooperation between this top overseas institution and the Technical University of Liberec (TUL) is nanotechnology and nanomaterials. The Liberec researchers had already discussed the possibilities of joint research projects with Professor

Sushanta Mitra, whose research focuses on innovations in sustainable nanomaterials for advanced engineering applications. And this idea took a real form last year.

"During our visit to Canada in June 2023, we presented the results of our materials research and assisted with filter testing and modification of test equipment on site. We also held discussions on future collaboration and talked about specific topics for joint research. A student team was also born at the University of Waterloo and the Technical University of Liberec, which focuses its research mainly on the preparation of nanofibrous materials with crystalline nanocellulose suitably modified for effective filtration or antibacterial applications," said Associate Dean of the FME TUL, Associate Professor Jan Valtera.

Nanocellulose can increase the efficiency of filter materials

According to Associate Professor Valtera, by cooperating with Canada, the TUL is taking its own research on nanofibre materials to an international level and the joint research shows significant synergistic effects. While the Canadian university is involved in the preparation and modification of nanocellulose using its unique technology, in Liberec they are focusing on the production of wound filters and layers that are made up of nanofibres containing modified crystalline nanocellulose particles.

"Nanofibre membranes are commonly used in filters today. However, we are investigating the effect of nanocellulose on improving their efficiency, using a highly productive alternating current wetting technology to prepare the nanofibre supports," said Associate Professor Valtera.

Canadian scientists were also interested in the technology of Liberec colleagues for the production of coiled filter candles. Their base is a perforated spool on which nanofibre yarn is wound in several layers. Finally, the spool can be fitted with a protective cage. As for the base spool, it can be a commercial version with standard dimensions, but if necessary, the Liberec researchers use 3D printing to produce customized parts, and a Czech-Canadian team is currently investigating the effectiveness of nanocellulose-containing nano yarns wound onto perforated cavities.

TUL owns the patented technology

The nanofibre yarns are produced on a functional patent-protected prototype machine designed by the scientific team of Professor Jaroslav Beran from the Department of Textile and Single Purpose Machines of the Faculty of Mechanical Engineering with the team of Professor David Lukáš from the Department of Chemistry of the Faculty of Science, Humanities and Education of TUL. It is the use of a linear composite material containing nanofibres produced by wetting polymer solutions with alternating electric current. The line for the softening of polymer solutions by AC electrospinning technology has already won a gold medal at the International Engineering Fair in Brno in 2017 in the category of Innovative Processing Technology, and in 2019 it attracted great acclaim in Barcelona at the ITMA textile machinery fair. The Liberec scientists used a needleless electrode instead of a needle or capillary for the new softening method.

"The spinning process produces a so-called nanofibre plume resembling water from a shower or smoke from a fire, carried by an 'electric wind'," said Professor David Lukáš, who explained the new process at the time. The core yarn is unwound from a spool and passed through a spinning

chamber where the nanofibre plume is wrapped around the core. Then residual solvents are evaporated in the drying and fixing zone, the nanofibre is heat fixed and the adhesion of the nanofibre layer to the core yarn is increased by a twisting device. The new technology is protected by several patents in the Czech Republic and abroad.

Towards 100% coreless nanofibre yarn

However, the development in this area at the Technical University of Liberec has moved forward to 100% nanofibre yarns that do not contain a core. This is the result of more than eight years of intensive research work, to which the support of the Nanoprogres cluster organisation has contributed significantly. The method of their production is that the "born" nanofibres from the polymer solution are dispersed in the air, and sections of nanofibres with opposite electrical polarity form the aforementioned nanofibre plume, which can be mechanically manipulated.

"But the nanofibre plume is not the final victory on the road to thread formation. Nanofibers have a high adhesion to any surface, so giving a twist to a nanofibre siding, which can even take the shape of a sleeve rising in the surrounding air, is impossible. Therefore, we have been looking for methods to manipulate the nanofibre material and have succeeded in transferring the mechanical torque to the nanofibre material," explains Professor David Lukáš, recalling that the first line for the production of 100% nanofibre yarn was also produced at the University with the support of the aforementioned cluster organisation Nanoprogres.

It was first presented last June at the ITMA International Textile Machinery Fair in Milan, where it attracted a lot of attention. "The line is unique in that it is significantly more productive than DC spinning because it allows for yarn/thread production at 15 m/min. It is therefore suitable for larger batches and the lower cost of the products also plays a role. It is the only device described to date that produces 100% nanofibre yarn at a semi-production level using alternating electric current. We rightly believe that our method is very robust for producing such yarns from a wide range of different polymer materials," says Professor David Lukáš, describing the unique technology.
According to Associate Professor Valtera, the advantage of 100% coreless nanofibre yarns is that they are not composite materials, and the entire volume of the yarn can be used for a specific function.

Earnings in Canada

Liberec scientists, together with Canadian teams, are now capitalizing on their experience with nanofibrous materials and the results of their research work in research focused on the effects of crystalline nanocellulose. This is added to the nanofibre material produced at the University of Liberec by dissolving it in a wetting solution.

"We are sending samples of nanofibre structures with added crystalline nanocellulose to Canada for subsequent efficacy tests. It's a win-win situation and improves the quality of our joint research. The Canadian university has the laboratories to prepare nanocellulose in the quantities needed and the capability to prepare different types of nanocellulose modifications, and it also has the necessary equipment to analyse the material, which our students are also working on. From our side, this is material and partly technological research," says Associate Professor Valtera, adding that in Liberec, they are able to produce sufficiently strong 100% nanofibre

threads, and together with Canadian colleagues they are verifying the application of these threads for filtration.

"And because we don't have any subsidies yet, we work with affordable polymer materials such as polyvinyl butyral (PVB). Our idea is to validate different nanofibre structures with added crystalline nanocellulose for filtration purposes. Such filters could also trap microplastics that are released into wastewater, for example, when washing textiles. At the same time, we are also working to ensure that nanofibres and microplastics are not released from the filters. Both of these are a big challenge and an important task in terms of environmental protection," says Associate Professor Valtera, adding that another shipment of filters left Liberec for Canada before Christmas.

Unique winding device for candle filters

A unique device developed at the Faculty of Mechanical Engineering is connected to the nanofibre production machine for the precise winding of nanofibre bobbins. "Both composite and pure nanofibre yarn can be wound. The winder is directly integrated into the machine, eliminating any rewinding process. The resulting bobbins can then, in addition to their classical textile use, be advantageously used for the filtration of liquids or gases in the form of so-called filter candles," says research team member Ing. Petr Žabka, Ph.D.

He points out that it is necessary to avoid abrasion of the yarn, and therefore the standard yarn guides are not used, but the yarn is guided in special pulleys. Another significant change from conventional winders is the motorised pressure of the bobbin on the support roller. This is important to achieve the required winding hardness. All parameters can be continuously changed during winding so that the structure and winding can be controlled.
"Thanks to the nanofibre structure, we can filter even very small particles. The large surface area of the nanofibres plays a major role in this. Experiments with various additives, in this case, cellulose nanocrystals, confirm that we can achieve more than 99% retention even for particles smaller than a micrometre," says Dr. Žabka.
He adds that in addition to the cooperation with the University of Waterloo, Czech companies within the Nanoprogress cluster, with which the Faculty of Engineering has long cooperated, have already shown interest in the winding equipment and filtration products.

Not only filters

Canada is a strong timber power and there is a large base for processing biomass, especially cellulose, which is actually waste from wood processing. There is a lot of it and its efficient use is at the forefront of Canada's concerns, where there is also a lot of pressure to protect the environment. Related to this is the interest in exploiting the properties of nanocellulose. And with the University of Waterloo having developed a unique technology for the preparation and modification of nanocellulose, research into its use is taking on new dimensions.

"The use of nanocellulose is an interesting research project and we have the potential to solve it together. The important thing is that the nanocellulose produced is naturally based, recyclable and does not pollute the environment. Our goal is to produce a filter material from which the trapped substances can be released and reused in a controlled manner after saturation with waste substances," says Associate Professor Valtera.

According to Professor David Lukáš, who has long been involved in tissue engineering and, in particular, in the treatment of skin diseases and injuries, functionalised nanocellulose has the potential to be used in this field as well. Scientists from the Technical University of Liberec have already had success in developing suitable materials and structures for scaffolds. These are materials that will serve as scaffolds for growing tissue cultures of human cartilage cells (chondrocytes).

The scaffold, or substrate, is seeded with the patient's own cell culture and grown outside the body to produce the patient's own full tissue. In general terms, this can be any tissue: bone, nerve, cartilage or liver cells. Studies at the 2nd Medical School have previously shown that cells are best grown on nanofibrous layers, which scientists use the alternating current electrospinning method to prepare.
"The role of functionalised nanocellulose is that it should support the healing process in skin treatment. More specifically, it promotes cell proliferation and migration and also prevents the scaffold from being colonised by germs (such as bacteria). And we will focus our research on the potential of crystalline nanocellulose to achieve at least some of these goals. This is not a simple matter, as the functionalizing agent usually affects the wettability of the mixed polymer solution. We, therefore, have to rely on a series of trial-and-error experiments to fine-tune such a spinning solution," outlines Professor Lukáš.