Proceedings of the Estonian Academy of Sciences,
2018, **67**, 2, 169–174
https://doi.org/10.3176/proc.2018.2.09
Available online at www.eap.ee/proceedings

MATERIALS SCIENCE

Novel method for producing electrospun composite nanofibre yarns

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Received 19 October 2017, accepted 14 March 2018, available online 24 April 2018

Abstract. The current study focuses on the development of a novel electrospinning method with high productivity for nanofibre yarn production. By analysing different recent approaches for nanofibre yarn preparation systems it was found that getting rid of mechanical contacts between the fibres and the collector before the formation of the yarn leads to a significant growth in the production rate. A novel electrospinning unit was designed and tested. The formed fibres were aligned and twisted over the core yarn using a column with rotary air movement. The produced core-shell yarns were studied under scanning electron microscope to determine the morphology of the fibres and yarns. Also, the linear density of the yarn was calculated to determine the growth in mass after covering the core yarn with nanofibres.

Key words: nanofibre yarn, air vortex, electrospinning.

1. INTRODUCTION

Electrospinning, a simple process to produce continuous fibres less than 1000 nm in diameter, is widely used to manufacture nonwoven membranes from nanofibres. Less common is investigation into electrospun yarns. At laboratory scale, various approaches to nanofibre yarn preparation are available, but so far there is no official statement about electrospinning instrumentation that would be able to produce continuous nanofibre yarn at industrial scale.

The simplest electrospinning setup consists of a spinneret filled with a polymer solution (most commonly a syringe with a needle), an earth-grounded collector plate, and a high voltage power supply to charge the spinneret [1,2]. Once the electrostatic force between the electrodes overcomes the surface tension of the polymer solution, the fibres will be pulled out from the droplet of the solution on the needle tip and collected on the collector [1–3]. With this method it is possible to produce nonwoven structures. Aligned fibres can be collected by a high-speed rotating disk or a drum collector [4,5]. Also dual collectors, such as parallel plates, where the fibres bridge between them, are used [6]. By reducing the collection area Teo and Ramakrishna [7] succeeded in producing short nanofibre bundles using such a method.

Recently many articles and patents have been published describing novel methods for nanofibre yarn production. Bazbouz and Stylios [8] reported a mechanism involving two grounded perpendicularly placed collector disks that are rotated around their axis. The resulting system produces highly twisted nanofibre yarns with a diameter around 20 μm at the production rate of 8 m/min [8]. Another approach to nanofibre yarn production is usage of oppositely charged spinnerets resulting in the attraction of the ejecting fibres to each other. In that case no grounded collector is necessary due to the fact that the oppositely charged fibres in contact will discharge themselves. In the mentioned approach the yarn is collected on the take-up system at a speed of 5.79 m/h (approx. 0.1 m/min) [9]. A similar concept using conjugated needle nozzles was proposed by Ali

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et al. [10] with the difference that a non-grounded funnel collector was used. Fibres form a web in front of the collector. The web is stretched to the funnel and is taken up from the collector with a winder. Twists on the yarn are generated by the rotation of the funnel. The resulting system has a production rate of 5 m/min. Lately Shuakat and Lin [11] published an article about a needleless electrospinning technique based on a ring collector collecting the fibres and forming a web similarly to the funnel method. The difference is in the fibre feeding direction: if in the funnel method fibres are collected to the collector and the membrane is pulled off at the opposite angle, then in the ring method the axis remains the same. The production rate achieved with this method is 240 m/h (4 m/min).

In the present paper a new method for nanofibre yarn production is reported. In the case of the proposed method the fibres are electrospun directly to an air vortex medium that twists the fibres together into an oriented bundle over the core yarn. After the formation of the fibre bundle over the core it will be pulled to the twisting unit where it will be twisted to the yarn and collected on a bobbin. The production rate can be controlled and varied by the fibre quantity and twist level given to the yarns. In this paper the working conditions of the system are explained and the effects of the nodal points for achieving an increase in the production rate are demonstrated.

2. MATERIALS AND METHODS

Poly(acrylonitrile) (PAN) powder (Mw = 150 000) was supplied by Polysciences, Inc., USA. Dimethylformamide (DMF) (purity ≥ 99%) was obtained from Merck KGaA, Germany. The electrospinning solution was prepared by dissolving PAN in DMF at a concentration of 10 wt%. The dissolving and mixing process was carried out on a magnetic stirrer at a constant speed at 45 °C for 24 h. The PA 6 yarn with a linear density of 11 dtex from Fein-Elast, Estonia, was used as the core yarn. The linear density of the nanofibre yarns was determined using gravimetric analysis with an analytical balance Mettler AE163. A Zeiss ULTRA 55 scanning electron microscope (SEM) was used for studying the fibres and yarns. For airflow simulations Ansys Fluent CFD software was used.

3. EXPERIMENTAL SETUP

Previous setups for nanofibre yarn production contained rotating collectors such as an ungrounded funnel or a rotating grounded hemisphere. It turned out that if the electrospun fibres have already reached contact with the collector surface, it will be extremely difficult to draw them away from there without yarn discontinuation in time. Individual fibres are weak and they have a tendency to break under tension. This limits the production rate of such systems.

A novel electrospinning setup was designed to carry out the experiment. The main aspects in the design were to eliminate all contact points between the fibres and any object before fibre bundle formation. The setup is shown in Fig. 1.

The electrospinning unit consists of a custom-built electrospinning chamber, two high-voltage power supplies (Spellman SL120PN30/230), and two peristaltic pumps (New Era NE-9000). The parts of the electrospinning chamber are shown in Fig. 2. The electrospinning chamber includes two main sections: the spinning chamber and the collecting unit (detail A) with a twisting assembly (2) and fans (3). The fans constantly remove air from the twisting chamber thus lowering the inner pressure. Additional air will be drawn into the twisting chamber from the spinning chamber through the opening (4) at the top of the chamber. This in turn reduces the pressure in the spinning chamber and additional air is supplied through the side inlet slots (5). The air guiders (6) will improve the rotational movement of the air column and air vortex will be created. The vortex speed depends on the amount of the air removed from the chamber by the fan assembly, which is controlled by a 12 V motor speed controller (Fig. 1). To increase the rotational velocity of the air column, a compression fan (detail B) was added to the spinning chamber floor (also controlled via the motor speed controller). The fan takes additional ambient air into the system. Before the air enters the chamber, it will be compressed and accelerated in the vortex direction. Peristaltic pumps were used to pump water into the syringe holder (detail C). The syringe holder contains two syringes. One syringe contains water (7) and the other a polymer solution (8). Pumping the water to the lower syringe creates pressure, which is transmitted to the upper syringe by the syringe pressure rods (9) facing each other. The polymer solution in the upper syringe will flow through the pipe to the spinneret (10). This design eliminates the possibility of charging the peristaltic pumps while the polymer solution is charged with high voltage. A similar design with two adjacent syringes was used also in 2014 by Pokorny et al. in 2014 [12]. Two multi-needle spinnerets (10) facing each other inside the spinning chamber were used. The distance of the spinnerets from the centre was 15 cm and their height from the spinning chamber floor was 15 cm. Needles with a diameter of 0.9 mm were used; in each spinneret there were up to 16 of them. The spinnerets were connected to high voltage power supplies of opposite polarity. The core yarn (11) is pulled through the spinning chamber (1) and the twisting unit (2). The end of the core yarn is attached to the take-up roller (12).

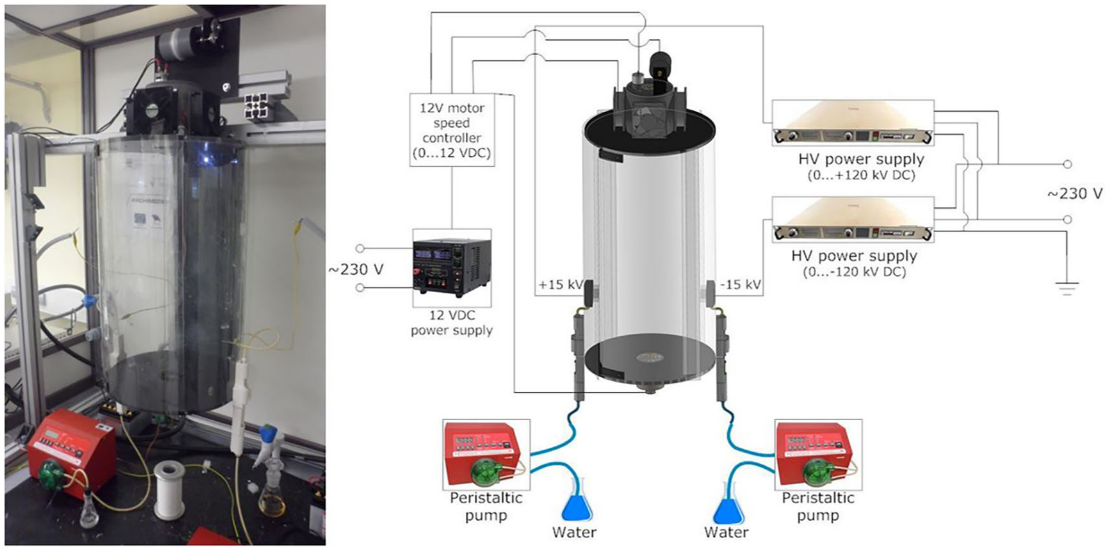

Fig. 1. Setup of the electrospinning unit and the electrical diagram.

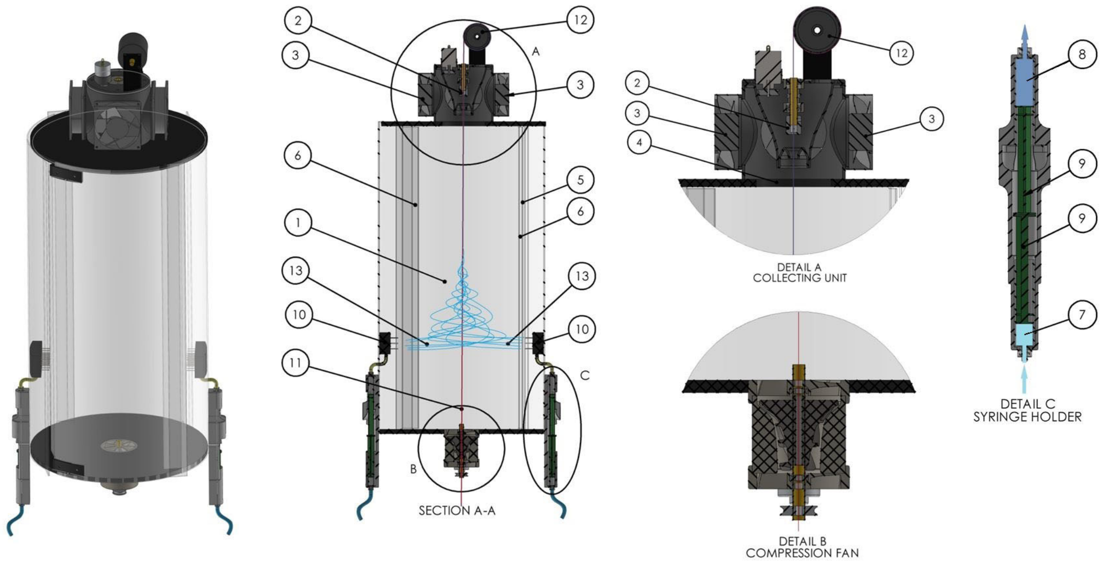

Fig. 2. Scheme of the electrospinning chamber: electrospinning chamber (1), twisting assembly (2), fan (3), opening at the top of the chamber (4), inlet slot (5), air guider (6), syringe with water (7), syringe with solution (8), syringe pressure rod (9), spinneret (10), core yarn (11), take-up roller (12), nanofibres (13).

Once the spinning process has started, the fibres (13) are ejected from the spinnerets due to the electrostatic force that is pulling them towards the centre of the spinning chamber. The air vortex forces the fibres to move helically, which lengthens the path of the fibres before the oppositely charged fibres are in contact. During the travel inside the vortex, the fibres are stretching and aligning. The fibres are wrapping the core yarn and travel through the twisting mechanism onto the take-up roller. The take-up speed on the roller and the rotational speed of the twisting unit are controlled by changing the voltage on 12 V DC motors (a motor speed controller is used).

When the yarn is collected it could be used with the core yarn or the electrospun fibres could be extracted by removing the core yarn by selected dissolutions. To produce nanofibre bundles with this system, the twisting mechanism has to be excluded and the take-up roller has

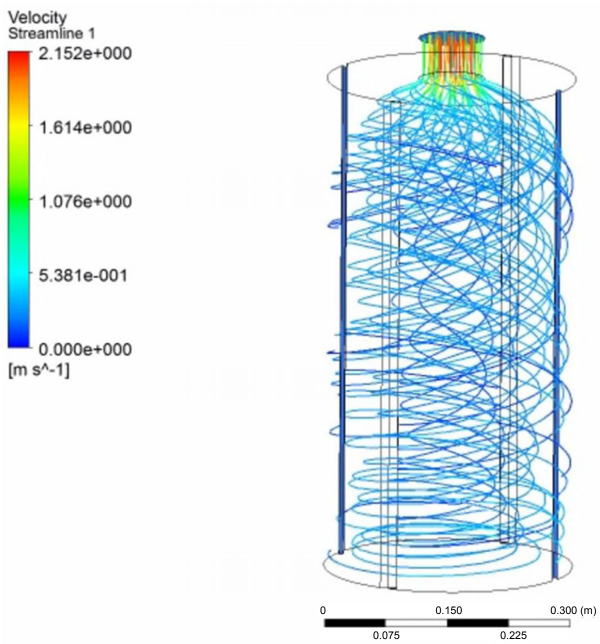

Fig. 3. Simulated air flow streamlines in the spinning chamber.

to be attached directly in front of the upper opening of the chamber where the air is extracted.

The shape of the electrospinning chamber was selected based on the Finite Element Method (FEM) analysis. Also other shapes could be considered; however, it was found by computer simulation that the cylindrical shape ensures the best performance of the air vortex (see Fig. 3). Since the total maximum flow rate created by the fans used in the current setup is 160 m^3/h, it was taken as one of the main parameters for the simulation calculations. The ambient air pressure was set at 101.325 kPa.

The simulation showed good homogeneous performance of the air vortex. At the inlet slots on the side of the spinning chamber the air velocity was close to 0 m/s, and it was accelerated helically to around 0.5 m/s while approaching the air vortex rotation axes. At the opening at the top of the chamber, where the air is extracted by the fans, the air flow velocity was close to 2.15 m/s.

4. DISCUSSION

The novel technique of nanofibre yarn spinning was tested with the 10% PAN solution. Yarns with nanofibre coating were produced by using different electrospinning parameters. The distance between spinnerets was 30 cm. The number of needles varied from 1 to 16 on each spinneret, the applied voltage was form ±15 to ±20 kV, and the pumping rate was from 1.6 to 4.0 mL/min.

The feed rate of the solution has to be sufficient to supply the spinnerets with a constant solution flow. If the needles start to dry up, they will behave like collectors for the opposite spinneret, which will result in fibre collection on stuck nozzles.

The distance between the spinnerets has to be large enough to let fibres elongate and dry. The distance in the air vortex system can be shorter than in the case of conventional electrospinning due to the curve path of the fibre motion. Also, the shorter the distance between the spinnerets, the lower voltage can be used.

During the experiments, different spinning parameters were used (see Table 1). Selection of the parameter values was based on the number of nozzles in the spinneret. In Table 1, the pumping rate and the number of the needles are presented for a single spinneret. The speed of the twisting spindle was set as constant at 9500 RPM (limited by the maximum power of the motor in the current setup). These parameters were optimal for carrying out continuous yarn production with minimal material loss.

The minimum yarn collection speed in the air vortex spinning system is set by the vertical speed of the air column in the spinning chamber. The air movement speed, including the vertical speed, can be regulated by the fan speed but there are some limitations. If the air movement speed is too low, the electrostatic force will be stronger than aerodynamic forces and the web from nanofibres will be formed in the air between the spinnerets without wrapping the core yarn. Usage of a compression fan helps to increase the rotational air movement in the spinning chamber. Another positive effect is that the accelerated compressed air will actually slow down the vertical speed of the air column movement. In the present case, the fast rotary movement of the air in the lower region of the electrospinning chamber brings about a venturi effect and the static pressure in the contact region between the compressed air and the slowly moving air column will decrease, which decelerates the vertical speed of the air column. A deceleration in the vertical speed of the air column is necessary in order to give the fibres more time to align and create more twists over the core yarn.

Produced nanofibre yarns were investigated under SEM and the thickness of the fibres and yarns was measured. An increase in the number of the needles on the spinneret resulted in an increase of the nanofibre amount and more uniform coating over the core yarn (Fig. 4b, c, and d). The average diameter of the fibres

Table 1. Yarn electrospinning parameters. The core yarn was pure 11.0 dtex PA6

No. of needles	Pumping rate, mL/min	Voltage, kV	Collection speed, m/min	Linear density, dtex
1	0.8	±14.5	35.5	17.5
6	1.8	±15.0	37.7	18.3
16	2.0	±20.0	39.3	20.5

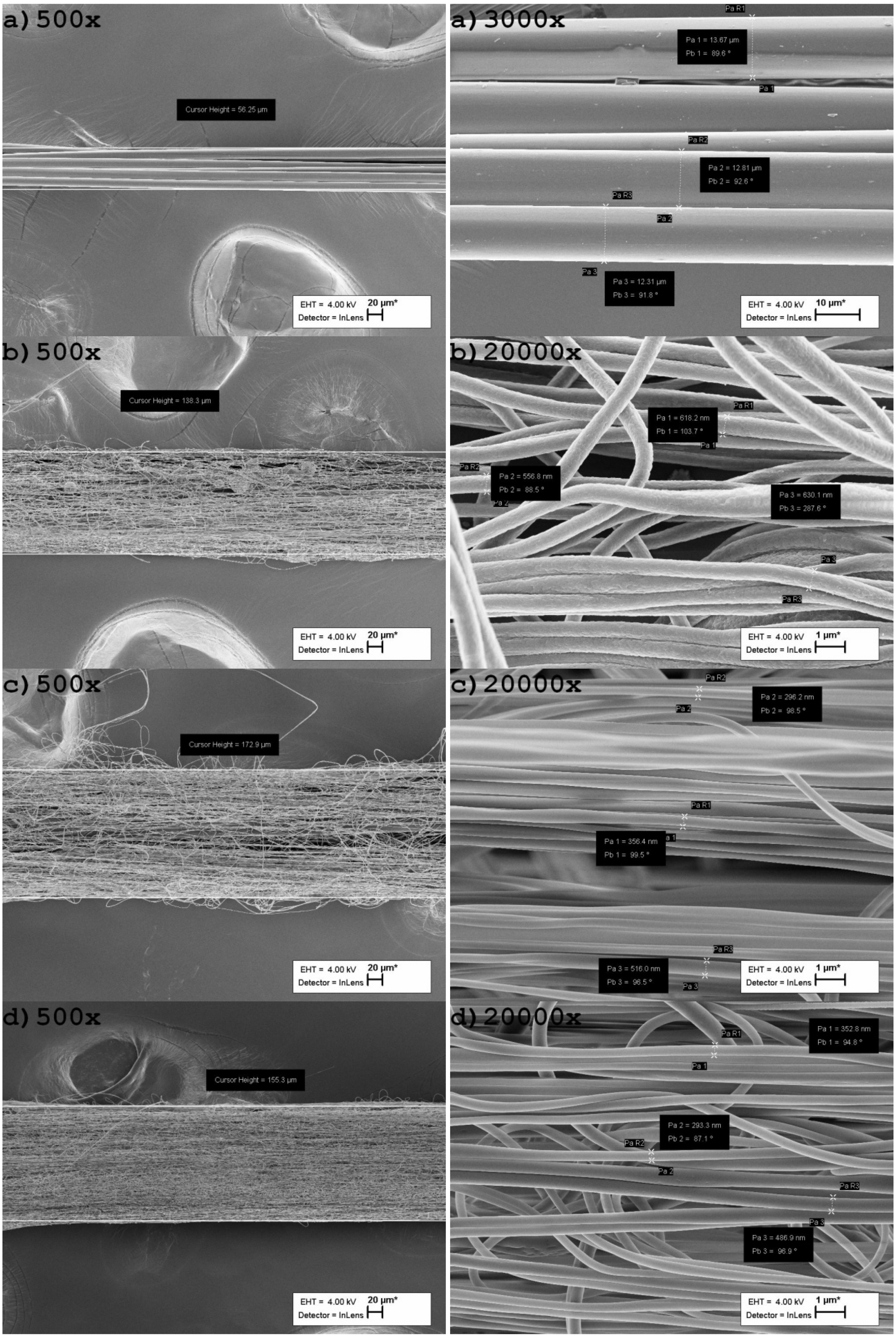

Fig. 4. SEM images of poly(acrylonitrile) (PAN) nanofibre coating with different spinneret configurations used: (a) PA6 core without PAN nanofibre coating; (b) coated using 1-needle spinnerets; (c) coated using 6-needle spinnerets; (d) coated using 16-needle spinnerets.

decreased from 601 to 377 nm with the increase of the voltage from 14.5 to 20 kV. At the same time, the average diameter of the yarn remained constant at around 155 μm. Note that the average diameter of the core yarn was around 56 μm.

The linear density of the yarns was determined using a gravimetric method. For this, 1.5 m of yarn was weighed on the analytical balance and then the decitex (dtex) value was calculated. Decitex is the mass of the yarn in grams per 10 000 m. To calculate its value the following formula was used: $dtex = \frac{mass\ of\ the\ measured\ yarn}{1.5} \times 10000$ m. The linear density of the PA6 yarn was already known and used as reference. The results for different number of needles are presented in Table 1.

It was found that by increasing the voltage and the number of needles in the spinnerets the fibre production rate increased. As the SEM images show, with the growth of the nanofibre production rate the fibres became wrapped over the core yarn more densely and it improved also the linear density. In the case of the same diameter of the yarns the mass of the fibres increased at the higher production rate.

5. CONCLUSIONS

A novel electrospinning method for nanofibre yarn production was developed. This method enables producing aligned composite nanofibre yarns with an average diameter of 155 μm at the production rate from 35.5 to 39.3 m/min. The increase in the production rate as compared to previous approaches was achieved by the removal of any kind of contacts between the mechanical objects and nanofibres before yarn formation. Nanofibres were electrospun directly in the air vortex medium which twists the fibres over the core yarn. Production of multilayered yarns demonstrated a uniform well-oriented fibre coating. Elevated number of the needles in spinnerets improved the fibre fabrication rate, which in turn increased the linear density of composite nanofibre yarns. The proposed method has a high potential in industrial-scale production.

ACKNOWLEDGEMENTS

The Prototron Foundation is acknowledged for partial financial support on this research. The publication costs of this article were covered by the Estonian Academy of Sciences.

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Uudne meetod elektrokedratud nanokiuliste komposiitniitide valmistamiseks

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On keskendutud uudse elektroketrusmeetodi väljatöötamisele, millega saavutatakse nanokiulise lõnga valmistamisel märgatav tootlikkuse kasv. Analüüsides erinevaid hiljutisi lähenemisviise nanokiuliste lõngade valmistamiseks, leiti, et mehaaniliste kontaktide kõrvaldamine kiudude ja kollektori vahelt enne lõnga moodustumist võimaldab tootmismahul märkimisväärselt kasvada. Töö käigus konstrueeriti uudne katseseade, mida katsetati erinevatel tingimustel. Seadmes moodustunud õhupöörises elektrokedratud kiud joondusid, kattes läbi elektroketruskambri veetud südamikniidi ühtlase kihina. Kiudude ja lõnga pinnakvaliteeti hinnati skaneeriva elektronmikroskoobiga. Ühtlasi määrati ka lõnga joontihedus, et kindlaks teha massi kasv lõngas peale südamikniidi katmist nanokiududega.