BIODEGRADABLE AC ELECTROSPUN NANOFIBROUS YARNS: TECHNOLOGY, MODIFICATIONS, POSTPROCESS TREATMENT AND PROPERTIES FOR SURGICAL SUTURES

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Abstract

The use of nanofiber threads as surgical sutures has been proposed as a way to improve the quality of repairs. However, due to the insufficient robustness of current manufacturing methods, it has not been possible to produce and scale continuous nanofiber threads on an industrial scale, and therefore they are not currently available for clinical use. To solve this problem, we have developed an effective method that enables the production of two classes of electrostatically spun yarns (100% nanofiber yarns and composite yarns with a nanofiber shell) with the potential for use as surgical suture materials. The presentation of production technologies through process and material parameters for the production of nanofiber yarns using alternating current electrostatic spinning, including possible modifications and subsequent treatments such as braiding or surface treatment of nanofibers in yarns, demonstrates the versatility of these materials, in particular through detailed images from a scanning electron microscope. In particular, attention is focused on the basic manufacturing processes and evaluation procedures for nanofiber threads with applications as surgical sutures. The aim of this paper is to show the possibilities and examples of creating biodegradable nanofiber threads as surgical sutures, particularly from a technological perspective.

Keywords: AC electrospinning, nanofibers, surgical sutures.

1. INTRODUCTION

Nanofiber materials are generally well known, as is their potential for use in medicine. It is only necessary to find applications where their effect will be sufficiently high. One such application is surgical sutures. Soft tissue injuries represent a substantial social and economic burden. Medical fibers are commonly used to repair these injuries during surgery. Patient's outcomes are, however, not perfect. Around 40% of surgical repairs are failing within the first few months after surgery due to poor tissue regeneration [1]. Surgical site infections (SSI) are the second most common healthcare-associated infections that cause prolongation of hospital stay and intensive care admission. In the European economic area, approximately 500,000 surgery patients are affected by the SSI yearly (reported by European centre for disease prevention & control - 2012). In order to overcome the SSI due to the implantation of sutures, it is necessary to fabricate antimicrobial fibers/yarns [2]. Development of new materials for surgical sutures is currently turning to functionalized nanofiber-based materials [3, 4] or only nanofibrous threads [5-7]. Sutures must meet a number of physical, chemical and biological requirements [8]. The required mechanical properties are strength and easy feasibility of a firm knot making [9], [10]. The critical properties of suture materials are biocompatibility, biodegradability or appropriate degradation time, and functionalization with various biologically active substances [10]. The use of nanofibers/yarns as medical fibers, including suture materials, has been proposed to improve repair quality [11]. However, due to the insufficient robustness of current production methods, continuous nanofibers cannot be produced and scaled up on an industrial scale and are currently not available for clinical use. To address this issue, we have developed an effective method that enables the production of two classes of electrospun

yarns (100% nanofiber yarns and composite yarns with nanofiber coatings) with the potential for use as surgical sutures of sufficient length with different structures from different materials [12] and with different polymer-additives composition for example for antibacterial behaviour [13]. Furthermore, nanofiber threads or composite threads with nanofibers can be processed using other technologies. For surgical sutures, the braiding method [14] is relevant, as it improves the overall mechanical properties, reduces the risk of nanofiber abrasion, and also allows for further high versatility or modifiability of the final thread structures. Examples of nanofiber thread structures are shown in **Figure 1**.

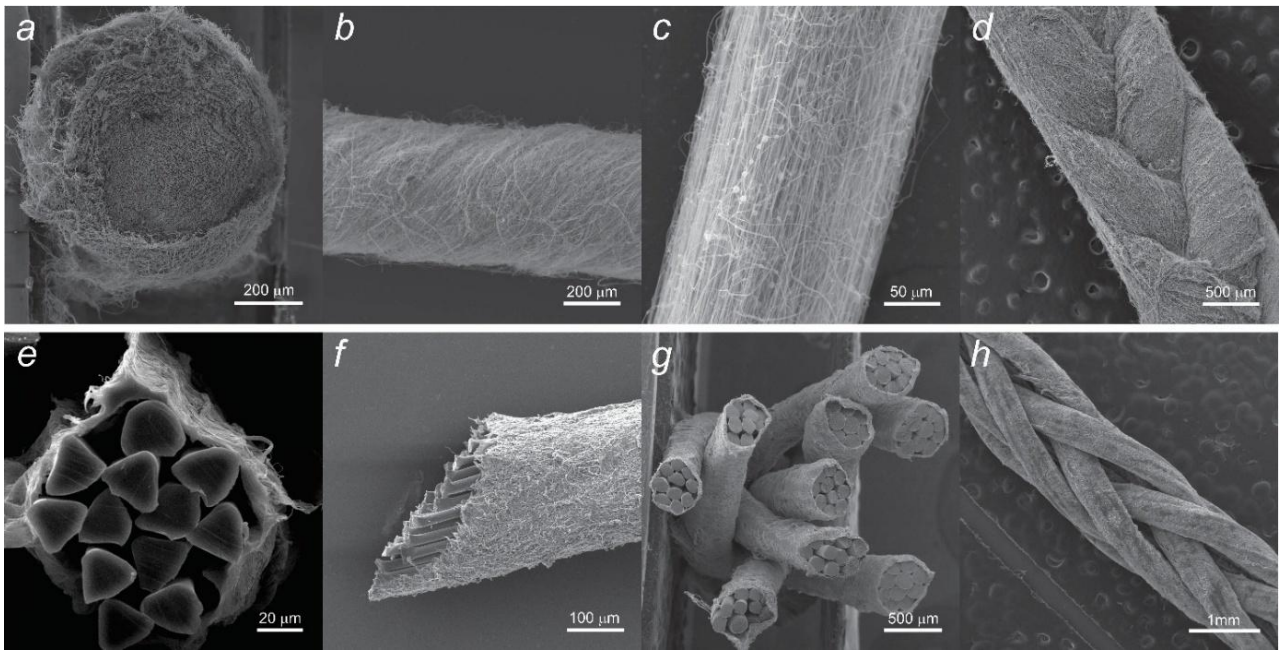

Figure 1 Examples of nanofiber thread structures at scanning electron microscope images: a) 100% nanofibrous thread – cross-section; b) 100% twisted nanofibrous thread; c) 100% nanofibrous thread without twist; d) braided threads from 100% nanofibrous threads; e) composite thread consisting of a microfiber core and a nanofibrous shell – cross-section; f) composite thread with nanofibrous shell; g) braided thread from composite threads – cross-section; h) braided thread from composite threads.

2. MATERIALS AND METHODS

This section will present specific examples of selected biodegradable threads with nanofibers that have been developed and optimized by a team of co-authors for subsequent in vitro and in vivo testing as surgical sutures. All basic materials used here can be used to create biodegradable materials for medical applications.

A polycaprolactone (PCL) spinning solution was used to create 100% nanofiber biodegradable material. PCL with two average molecular weights of 45,000 g/mol (PCL45; Purasorb PC08; Corbion) and 80,000 g/mol (PCL80; Merck) in a weight ratio of 1:2 in a solvent system of acetic acid, formic acid, and acetone in a weight ratio of 1:1:1 with a final polymer concentration in the solution of 10 wt%. This solution was left on a magnetic stirrer for 4 hours at a temperature of 45 °C and then stored in a refrigerator at 8 °C until spinning. Spinning, i.e., the creation of 100% nanofiber thread, was carried out based on the principle described in patent WO 2024/046515 A2 by alternating current (AC) electrospinning. Nanofibers created by AC electrospinning are collected on an electrically inactive rotating drum. The nanofiber thread must then be twisted. This is achieved by removing the nanofibers from the surface of the rotating drum and passing the nanofiber material through a rotating eye. This is followed by drying the nanofiber thread to remove residual solvents and winding it onto a suitable spool. In this manufacturing process, the thread is given a false twist.

Furthermore, a series of biodegradable composite threads with nanofibers was produced. The central microfiber threads produced by melt spinning technology were: i) polycaprolactone in monofilament form – 1 microfiber with diameter 100 μm; ii) polycaprolactone in multifilament form – 10 microfibers with diameter 100 μm; iii) a copolymer of caprolactone and glycolic acid (in a ratio of 95:5) – one microfiber with diameter 100 μm. All these threads were supplied by the collaborating institution ITA RWTH Aachen, Germany. These microfiber threads were coated with a nanofibrous coating using the AC electrospinning process.

This process was carried out on equipment also patented by our team WO 2016/192697 A2. It involves capturing the upward flow of nanofibers using a ballooning central thread. The final composite thread is then formed by the core microfibrous thread and the nanofibrous shell. This is followed by drying to remove the solvent and continuous winding onto a suitable spool. The linear weight of the nanofiber coating of the thread is proportional to the draw speed of the core thread. The following polymer solutions were used to create nanofibers in the composite thread packaging: i) PCL45 and PCL80 in a weight ratio of 1:2 in a solvent system of acetic acid, formic acid, and acetone in a weight ratio of 1:1:1 with a final polymer concentration in the solution of 10% by weight (nanoPCL); ii) a copolymer of caprolactone and lactic acid in a ratio of 85:15 (PLCL; Purasorb PLC 7015, Corbion) in a solvent system of acetic acid, formic acid, and acetone in a weight ratio of 1:1:1 with a final polymer concentration in the solution of 10% by weight (nanoPLCL); iii) PCL45 and PCL80 in a weight ratio of 1:2 in a solvent system of acetic acid, formic acid, and acetone in a weight ratio of 1:1:1 with a final polymer concentration in the solution of 10% by weight; 0.002; 0.02; 1 and 5 wt% of the model antibacterial agent chlorhexidine dihydrochloride Mw 578.37 (CHX; TCI Tokyo Chemical Industry) was added to the polymer solution; (nanoPCL+CHX). First, relatively high values of CHX addition (1 and 5wt%) to the PCL nanofiber coating were tested, particularly with regard to determining the effect of CHX addition on the electrospinning process in terms of productivity (change in linear weight of the resulting threads) and with regard to changes in the diameters of electrospun PCL fibers with CHX addition. During the course of this project, preliminary biodegradation tests were carried out on these surgical threads with nanofibers, and it was found that the central multifilament and monofilament made of PCL degrades too slowly for the selected applications (closing wounds in the trunk and limb regions and in subcutaneous tissue). Subsequently, experiments with smaller amounts of CHX (0.02 and 0.002wt%) were carried out with a central monofilament made of PCGL. These concentrations were set according to a literature review [15, 16] for further possible study of the creation of surgical sutures with an antibacterial effect, where it is necessary to monitor the compromise between the antibacterial effect (the effect on prokaryotic cells) and cytotoxicity (the effect on eukaryotic cells). The nanofiber coating was applied to the core thread at the highest possible stable speed of core thread withdrawal. This meant that it was possible to wind at least 100 m of composite thread in one piece without any problems with core thread breakage, continuous winding or drying, or interrupted nanofiber flow. Optimization was carried out in such a way as to achieve continuous application of nanofibers with the least possible variability in the process and sensitivity to disruption of yarn homogeneity. An overview of maximum core thread withdrawal speeds for stable continuous production of composite filaments with nanofibers is given in **Table 1**. The addition of a model antibacterial substance was tested in a wide range of weight percentages of dry fiber. This was chosen to determine the effect of CHX addition on the spinning process (stability and productivity) and further on the final diameters of nanofibers produced from these solutions.

Both composite nanofiber threads and 100% nanofiber threads were further subjected to a braiding test, always braiding eight threads plus a centre thread. This arrangement of nine threads results in the most circular diameter, which is very important for surgical threads. An EY-RA-8-4(110 II) braiding machine supplied by BTTO (manufacturer: Hsiang Chuan Machinery, Taiwan) was used. Imaging of threads was done by scanning electron microscopy (SEM) Vega S3B Tescan. Samplese were coated by 7nm of gold before observing at SEM. Image J Fiji was used for image analysis.

3. RESULTS AND DISCUSSION

The results in **Table 1** represent the values of thread diameters and fiber diameters measured using images from a scanning electron microscope and the linear weight (fineness) values of these threads. The “tex” unit represents the weight of one kilometre of thread. The thread diameters are listed here because surgical sutures are classified according to thread diameter. The USP (United States Pharmacopeia, USP 43–NF 38, 2020) standard scale or the EP (European Pharmacopoeia) scale is used. The structures of the manufactured nanofiber threads can be seen in selected SEM images in **Figure 2**.

Table 1 Results of introducing biodegradable nanofibrous thread and composite nanofibrous threads produced by AC electrospinning. The production speed represents for individual nanofibrous threads withdrawal speed of produced threads during AC electrospinning process and it is maximal withdrawal speed for stable and continuous production of the thread and for braided threads it is the speed of braiding.

thread type	production speed [m/min]	final linear density - fineness [tex]	thread diameter [µm]	nanofiber diameter [nm]
100% nanofibrous thread				
PCL	3	98±5	604±84	1138±504
core threads				
PCL multifil	x	272±3	345±68	x
PCL monofil	x	27±1	100	x
PCGL monofil	x	9±1	100	x
Composite threads				
PCL multifil / nanoPCL	50	281±3	510±20	581±440
PCL monofil / nanoPCL	30	35±1	173±18	546±250
PCL monofil / nanoPCL+CHX 1	30	34±1	164±7	557±176
PCL monofil / nanoPCL+CHX 5	30	34±1	164±10	551±178
PCGL monofil / nanoPCL	50	14±1	152±7	478±314
PCGL monofil / nanoPLCL	45	15±1	153±4	423±154
PCGL monofil / nanoPCL+CHX 0.002	50	15±1	183±7	552±156
PCGL monofil / nanoPCL+CHX 0.02	50	15±1	162±8	564±149
braided threads from 9 individual threads				
braided PCL 100% nanofibrous yarn	3	970±8	3306±47	x
braided PCL monofil/ nanoPCL	3	124±1	640±37	x
braided PCGL monofil/nanoPCL	3	150±2	538±18	x

Table 1 shows that optimizing process parameters made it possible to influence the fineness of the resulting threads. This makes it possible to achieve repeatability in production and to compare the doses of added active substances. Furthermore, it can be concluded that the addition of chlorhexidine does not significantly affect the diameters of the nanofibers. It can also be concluded that for PLCL nanofibers in composite thread packaging, the nanofibers are more tightly bonded, the nanofiber packaging is less fluffy, and thus the thread is smaller in diameter while maintaining the same fineness.

4. CONCLUSION

In the project within which these nanofiber surgical sutures are being developed, we have decided to target the application of sutures for suturing the skin of the limbs or torso and subcutaneous tissue. For these applications, it is most appropriate to use USP 4/0 (EP2) sutures with a diameter of 0.150-0.199 mm or USP 3/0 (EP 1.5) sutures with a diameter of 0.200-0.249 mm. It is clear that 100% nanofiber sutures are currently too large in terms of diameter, and it should be noted, although this was not presented here, that their mechanical properties do not meet the required values either. The choice for further testing of manufactured biodegradable threads with nanofibers will therefore focus on composite threads, both single and braided. One

way to reduce the diameter to the required value is to combine composite threads and basic microfiber threads during braiding.

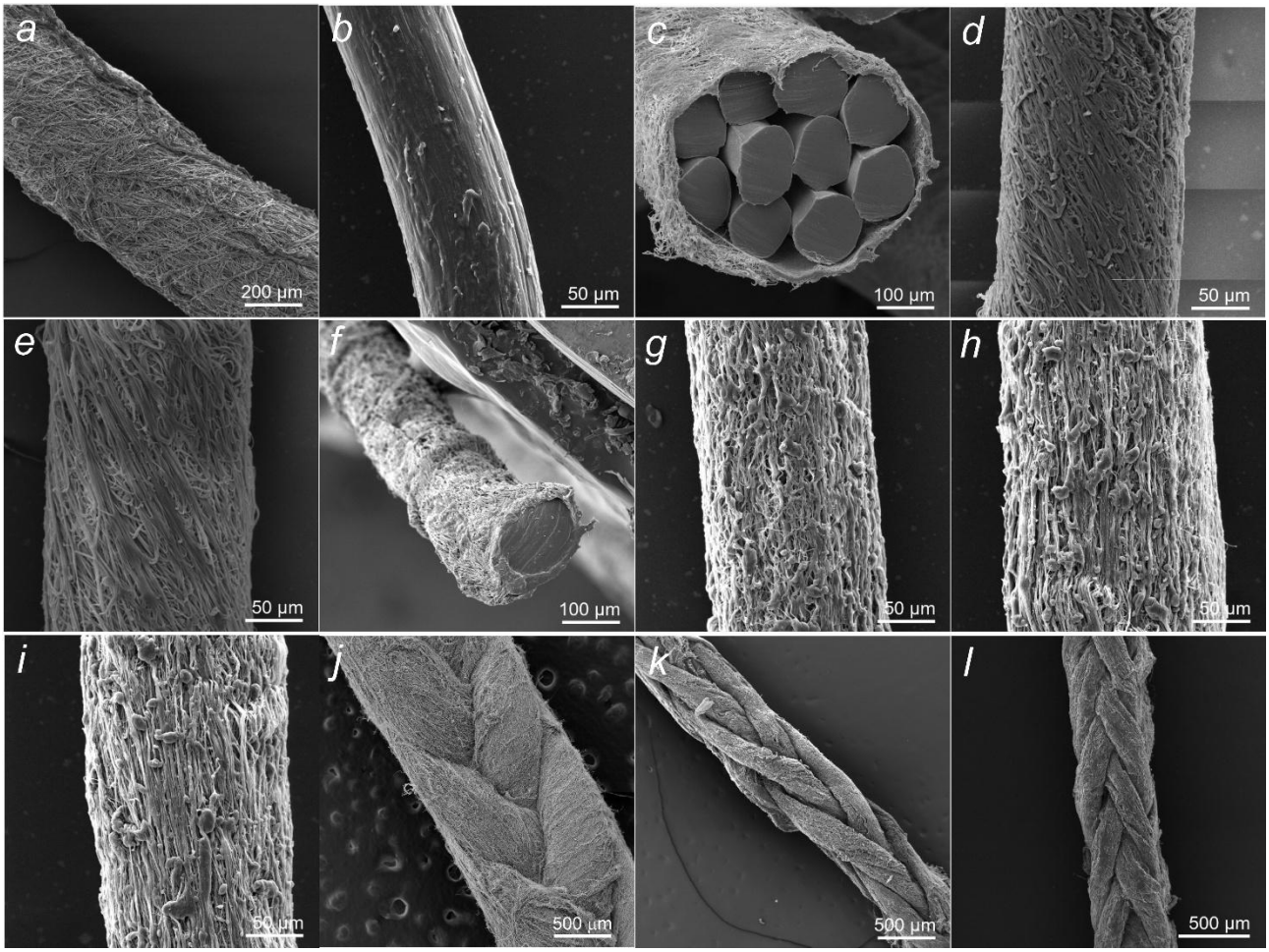

Figure 2 SEM images of biocompatible biodegradable threads optimized as surgical sutures here: a – PCL 100% nanofibrous yarn; b – PCGL monofil; c -PCLmultifil/nanoPCL (cross-section); d - PCL monofil / nanoPCL+CHX 1; e - PCL monofil / nanoPCL+CHX 5; f - PCGL monofil / nanoPCL (cross-section); g - PCGL monofil / nanoPLCL; h - PCGL monofil / nanoPCL+CHX 0.002; i - PCGL monofil / nanoPCL+CHX 0.02; j - braided PCL 100% nanofibrous yarn; k - braided PCL monofil/ nanoPCL; l - braided PCGL monofil/nanoPCL.

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