Cation-Functionalized Chitin Fibres – Development of a Continuous Spinning Process for Ion-Functionalized Biopolymer Fibres Based on Chitin

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Introduction, Problem Statement and Objectives

The textile industry is facing an increasing number of challenges, including climate change, resource scarcity and growing consumer awareness of sustainability, which demand new solutions across the entire value chain. Currently, the market is dominated by synthetic fibres derived from fossil resources, which significantly contribute to environmental and climate issues [1–2]. However, natural fibres are not inherently sustainable either. Their cultivation often requires substantial water usage, as well as the use of fertilisers and pesticides, which has an adverse effect on their environmental footprint [3].

In this context, chitin — the second most abundant naturally occurring polymer after cellulose — has gained significant attention as a promising, highly functional bio-based raw material [4]. It is produced in large quantities as a by-product of the food industry, particularly during the processing of crustaceans and shellfish. This makes chitin widely available, cost-effective, and sustainable. Chitin and its derivatives, such as chitosan, exhibit numerous desirable properties: they are biodegradable, bioactive and biocompatible, and they possess high mechanical strength due to their crystalline structure. Consequently, chitin is ideally suited to high-quality, functional textile applications, particularly in the field of single-use medical products, where demand is continuously growing alongside significant waste generation. However, the main challenge lies in the technological use of this raw material: due to its semi-crystalline molecular structure, chitin is barely soluble. While this structure gives the material its beneficial properties, it considerably complicates its processing into textile structures. Conventional dissolution methods rely on aggressive solvents that pose health and environmental risks, such as trichloroacetic acid or LiCl/DMA. These solvents are responsible for polymer degradation, weakening of the material, and the need for complex purification steps [5–7]. Such processes are unsuitable for medical applications and difficult to scale up for industrial use.

A more sustainable alternative is the use of ionic liquids (IL). These modern solvents have the potential to dissolve chitin without compromising its structural integrity. However, technological barriers remain high and current processes are predominantly discontinuous, with production volumes being limited [8–10]. Therefore, a commercially viable and fully sustainable process that enables the continuous production of chitin fibres on an industrial scale is still lacking.

The objective of IGF project 22568, 'Cation-functionalised chitin fibres', was to develop an IL-solvent-based, continuous wet-spinning process for producing 100 % pure chitin multifilament yarns that is gentle on the material and scalable in terms of process technology. The project also aimed to develop functional textiles with integrated bioactive cations (e.g. calcium or strontium ions, which support bone regeneration) to enable new applications in industry, particularly in the rapidly growing smart and medical textile sectors.

Results

In the IGF project 'Cation-functionalised chitin fibres', a continuous spinning process suitable for SMEs was successfully developed. This process enables the production of pure chitin multifilament yarns on an industrial scale. Through targeted functionalization with bioactive ions, fibre properties could be specifically tailored and controlled enzymatic degradability was achieved. The following section provides a detailed overview of the project's key outcomes and technological developments.

Process development for the continuous manufacturing of chitin multifilament yarns

Throughout the project, various ionic liquids were systematically evaluated for their suitability as solvents for filament spinning. The most promising results were obtained using 1-ethyl-3-methylimidazolium propionate (EMIMOPr, PROIONIC GmbH, Raaba-Grambach, Austria). This IL efficiently dissolved various grades and sources of chitin at moderate temperatures (60–90 °C) without degrading the polymer. Furthermore, EMIMOPr could be fully removed from the fibres during subsequent processing. As illustrated in Figure 1, the FTIR spectra of the raw chitin powder (grey) and the multifilament yarns (red) obtained from it show that the chemical structure of chitin remains unchanged after spinning and no solvent residues are present.

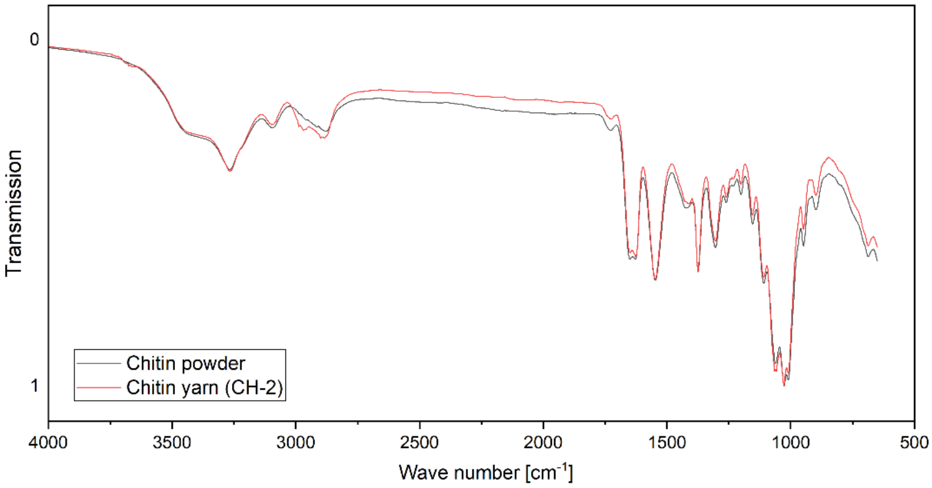

Figure 1: Comparison of FTIR spectra (4000–700 cm^{-1}) of chitin powder and multifilament yarns after the spinning process

EMIMOPr was used to prepare stable spinning dopes with chitin concentrations ranging from 3 wt% to 5 wt%. To ensure a stable spinning process, especially during the upscale, the rheological properties were thoroughly analysed and adjusted. The laboratory-scale spinning process was successfully adapted for a modular wet-spinning pilot plant (FOURNÉ MASCHINENBAU GmbH, Alfter-Impekoven, Germany), which features individually controlled zones for extrusion, coagulation, washing and drying. Particular attention was given to the configuration of the spinneret to achieve a continuous and stable spinning process and uniform filament morphology (Table 1).

Table 1: Results of yarn count and diameter measurements of chitin multifilament yarns (CH) spun from EMIMOPr

Yarn sample	Spinneret geometry	Yarn count [tex]	Filament diameter [µm]
CH-1	70µm/148f	112,58 ± 3,91	37,38 ± 1,19
CH-2	90µm/300f	124,53 ± 1,06	29,02 ± 4,69
CH-3	90µm/300f	127,03 ± 0,90	21,08 ± 2,82
CH-4	160µm/24f	94,55 ± 3,42	45,61 ± 5,49

Figure 2 shows scanning electron microscopy (SEM) and light microscopy images of the produced chitin filaments. A uniform, closed filament surface with distinct fibrillation, predominantly round cross-sections, and consistent diameters without defects were observed in all examined yarns.

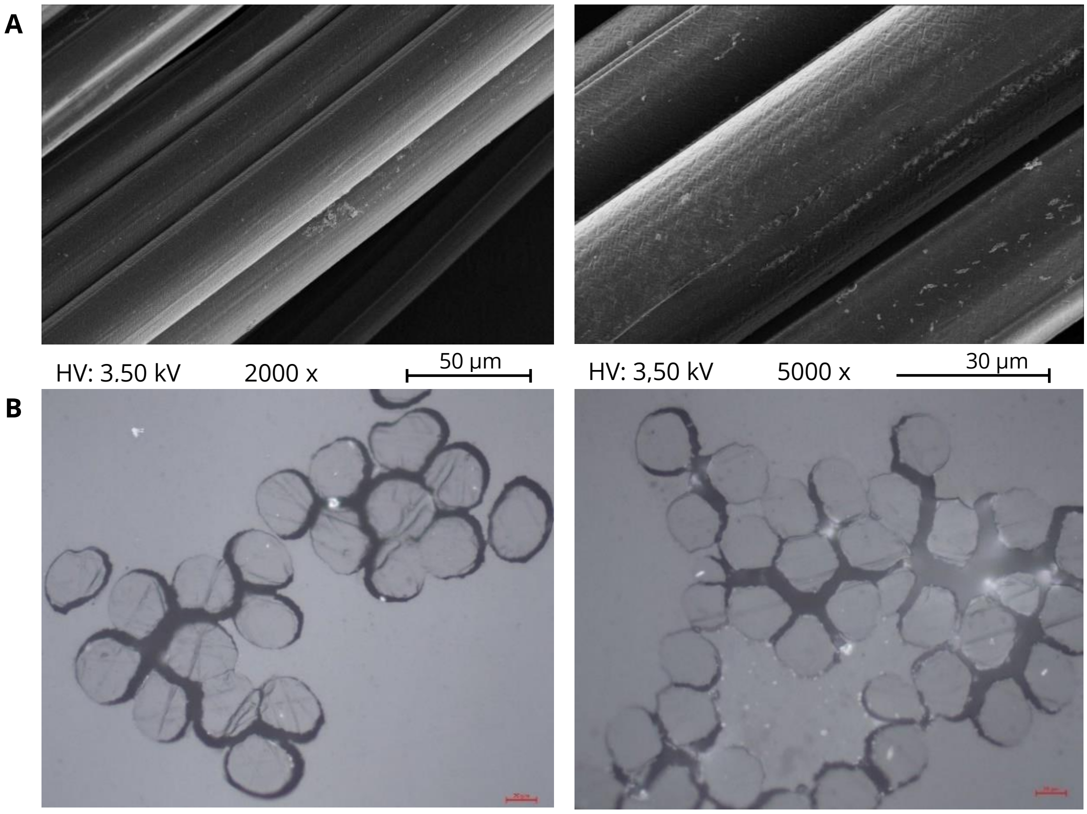

Figure 2: SEM images of multifilament yarn CH-1 (A); light microscopy images of filament cross-sections CH-1 (70 µm/148 filaments, left) and CH-3 (90 µm/300 filaments, right), magnification: 50x (B)

Compared to the results of previous projects and established spinning processes, particularly conventional chitosan spinning using acetic acid [11] and ionic liquid-based spinning using 1-ethyl-3-methylimidazolium acetate (EMIMOAc) for chitosan with a degree of deacetylation above 70 % [12], the chitin filaments manufactured in this project exhibited significantly higher tensile strengths of ≥ 20 N (Figure 3, right). These mechanical properties exceed all prior study results, highlighting the strong potential of the newly developed spinning process. However, the observed variability in mechanical properties depending on spinneret geometry, as well as equipment-related limitations that currently hinder the spinning of higher-viscosity solutions, provide a solid foundation for future projects aimed at further optimising and developing the process.

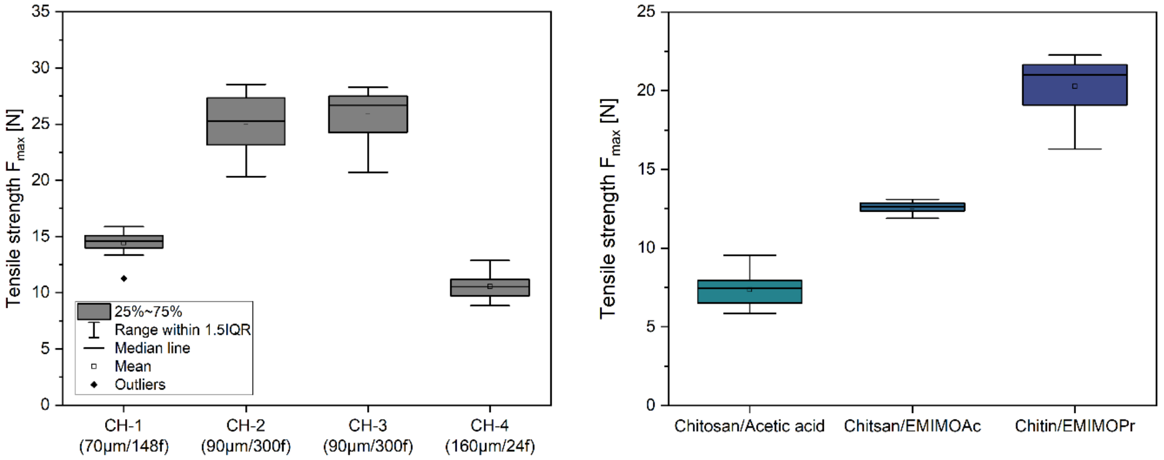

Figure 3: Tensile strength (F_{max}) of chitin multifilament yarns spun from EMIMOPr (left); comparison of tensile strength of chitosan fibres from acetic acid-based wet spinning [11] and chitosan fibres spun from EMIMOAc [12] (right)

Functionalization of chitin fibres with bioactive ions

Another objective was the integration of bioactive calcium, strontium and magnesium ions into the chitin fibers during the spinning process. These ions provide additional functional properties that are particularly relevant for medical textile applications, such as bone-regenerating implants or wound dressings. Three different methodological approaches were designed and investigated: (1) adding the ions directly to the spinning dope, (2) functionalization during the coagulation process and (3) post-spinning functionalization. Figure 4 provides a schematic overview of the investigated approaches, using calcium ion functionalisation as an example.

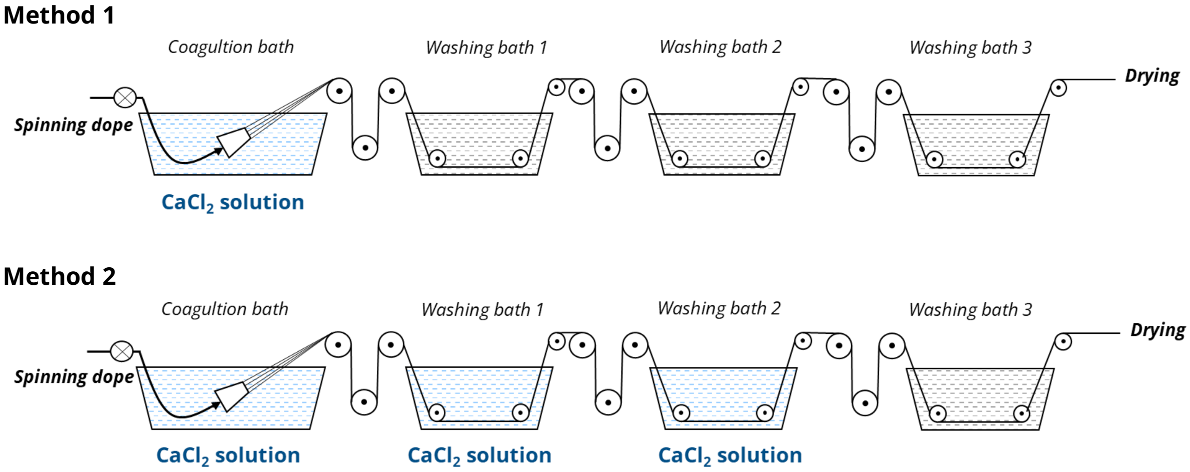

Figure 4: Filament functionalization with calcium ions during the spinning process

Promising results were achieved through in-line functionalization during coagulation. By adding calcium, magnesium, or strontium salts to the coagulation bath (deionized water), the ions were effectively embedded into the filaments before complete solidification. A homogeneous distribution of ions was achieved without the mechanical integrity of the fibres being negatively affected. Analyses conducted in collaboration with industrial and academic partners (Anton Paar GmbH, Institut für Abfall- und Kreislaufwirtschaft der TUD), including EDX mapping (Figure 5), optical emission spectroscopy (ICP-OES) (Figure 6), zeta potential measurements and FTIR spectroscopy, confirmed the integration of ions into the chitin filaments. Calcium ions in particular exhibited a strong affinity to chitin, remaining within the fibres even after extensive washing and drying.

To evaluate the release behaviour of the ions under physiologically relevant conditions, systematic elution studies were performed. The results showed that the majority of the ions were released within seven days, with only a small fraction remaining embedded in the fibres in the long term. This release profile is advantageous for applications in bioactive textiles or drug delivery systems as it supports the early onset of effects such as anti-inflammatory, wound-healing or mineralization-promoting activity, thereby enhancing the materials' overall functionality.

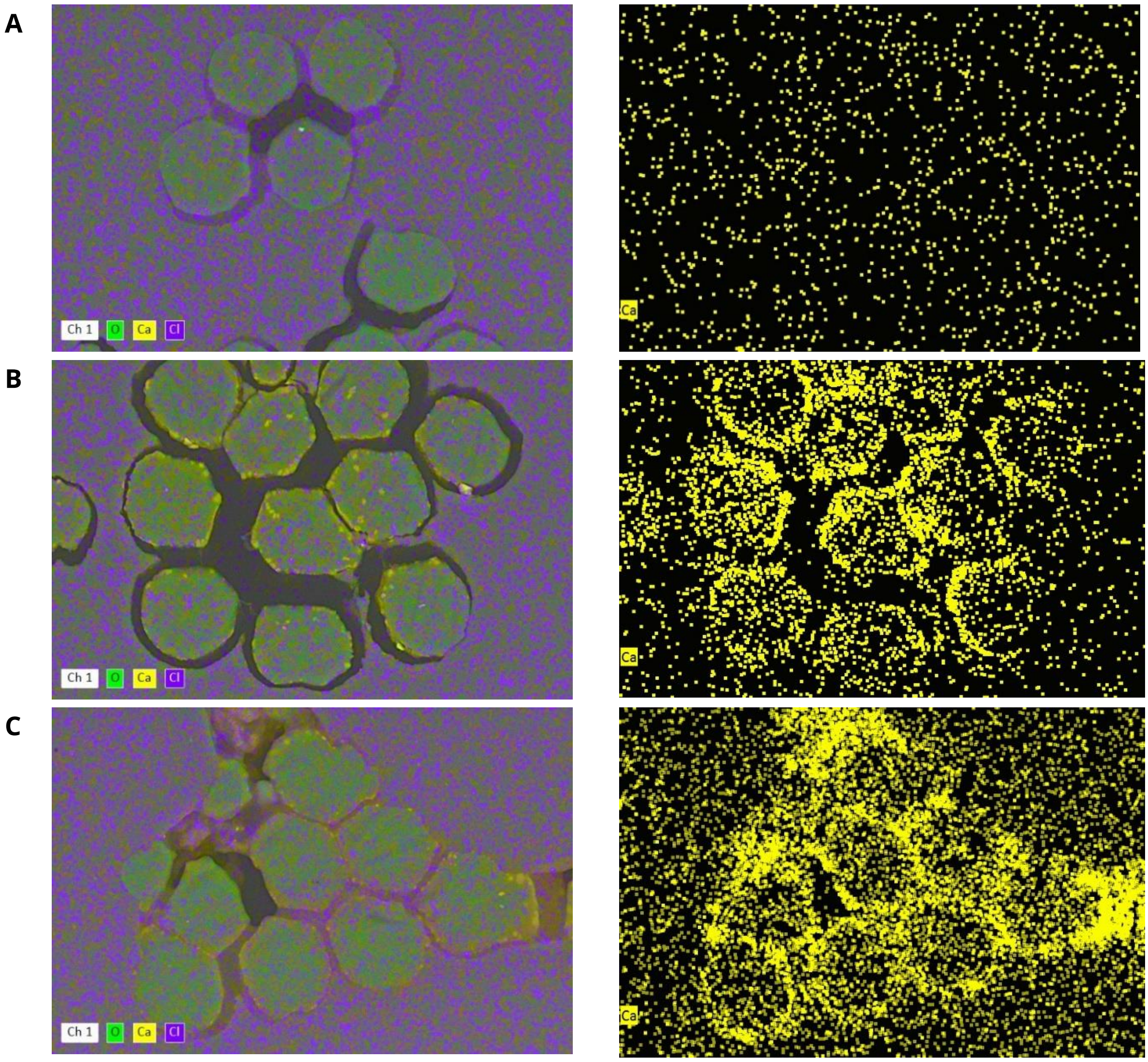

Figure 5: EDX mapping (20 kV, spot size 4.0) of chitin multifilament yarns: (A) without functionalization; (B) calcium-functionalized via Method 1, 1 min exposure in $CaCl_2$ solution; (C) post- spinning functionalization with 24 h exposure in $CaCl_2$ solution

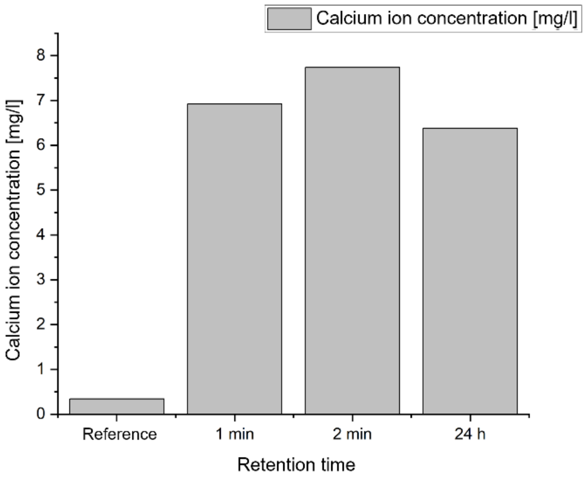

Figure 6: Results of ICP-OES analysis of calcium content in chitin multifilament yarns functionalized during the spinning process

Textile processing of chitin multifilament yarns

Despite the inherent brittleness of chitin fibres, a typical property of crystalline biopolymers, it was possible to produce textile structures by making targeted adjustments to the processing. By combining chitin yarns with supporting yarns (e.g. cotton or viscose), twisted yarns could be generated and processed into woven and knitted fabrics. Initial samples, such as mesh and fabric, confirmed the fibres' basic suitability for technical and medical textile applications (Figure 7). While the yarns still exhibit high brittleness, these results demonstrate strong potential for future applications. Further improvements in flexibility could be achieved by applying fibre sizing agents or blending with other biodegradable polymers, such as viscose, cellulose, or cotton. This would expand the range of applications in medical and technical textiles. Overall, the project presents a promising approach to using bio-based materials in demanding textile applications.

Figure 7: Chitin multifilament yarns spun from EMIMOPr (left) Twisting trials with chitin multifilament yarn on a laboratory double-yarn twisting machine (DIRECTWIST, AĞTEKS Ltd.) (middle); knitted sample with viscose support yarn (black) (top right); plain-woven fabric made from pure chitin yarn (bottom right)

Conclusion

As part of the IGF project 'Cation-functionalised chitin fibres', a continuous, SME-adapted spinning process was successfully developed for industrial-scale manufacturing of ion-functionalised chitin multifilament yarns from low-cost raw materials using IL, making one of the world's most abundant biopolymers economically accessible for fibre-based applications. Bioactive ions, like calcium, strontium, and magnesium ions, were incorporated to enable the functionalisation of fibres during the spinning process, resulting in enhanced enzymatic stability and biodegradability. These characteristics are essential for a range of advanced applications in both medical and technical textiles. The project's outcomes provide a solid scientific and technological basis for developing next-generation products in the fields of medical textiles, regenerative medicine and tissue engineering.

Acknowledgements

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