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Transforming Alginate into Beaded Polymeric Fibers Using Core-Sheath Pressure-Spinning

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Abstract - Alginate (ALG), derived from brown algae, is a promising renewable resource for sustainable materials due to its abundant availability and biodegradability. However, conventional methods of processing ALG into nano- or microfibers face significant challenges due to its intrinsic physicochemical properties. This study investigates the conversion of ALG into beaded polymer fibers using core-sheath pressure spinning (CSPS), focusing on fiber morphology, yield, and structural characterization. To fabricate fibers, ALG is combined with the spinnable polycaprolactone (PCL) polymer. The study analyzes the influence of rotational speed on fiber formation and demonstrates that higher speeds result in finer fibers, with diameters decreasing from 1.98 µm at 2,500 RPM to 1.02 µm at 25,000 RPM. The presence of bead structures in the fibers is attributed to the high surface tension of water, one of the solvents used. The application of a rotational speed of 10,000 RPM is determined to be optimal, since it is able to produce a fiber product with a moderate yield of up to 741 mg while lower speeds result in incomplete fiber production and higher speeds gave reduced yield. Confocal microscopy confirms the core-sheath architecture of the fibers, with ALG as the core and PCL as the sheath. Fourier Transform Infrared Spectroscopy validates the chemical composition of the fibers, confirming the presence of both polymers. The results suggest that CSPS is a scalable and environmentally friendly approach to producing ALG-based polymeric fibers with potential applications in drug delivery, where the beaded structure may enhance drug encapsulation and sustained release.

Keywords**:** alginate; pressure-spinning; polymeric fibers; core-sheath fibers; beaded fibers.

1. Introduction

Alginate (ALG) has attracted considerable attention due to its versatile properties and numerous applications in various fields. Due to the abundant availability of raw ALG in nature and its advantage of being easily recyclable, ALG-based materials are helping to drive the transition from a fossil-fuel-dependent economy to a more environmentally friendly and sustainable one. Moreover, ALG's inherent biocompatibility makes it an exciting biomedical material [1]. ALG has been utilized in various forms, including nanoparticles, microparticles, microspheres, and nanofibers, and has found extensive application in the food, textile, and biomedical industries, as well as in bioremediation [2].

Electrospinning is a widely utilized technique for the production of ALG fibers. However, the electrospinnability of ALG is hindered by several factors, including its polyelectrolytic nature and strong hydrogen bonding network. The high electrical conductivity of ALG results in significant charge accumulation, which complicates the processing of ALG solutions. Additionally, reduced chain entanglement, gelation at low concentrations, and high surface tension contribute to the challenges associated with electrospinning ALG [2].

An alternative to electrospinning is pressurized gyration (PG), a straightforward and efficient approach that does not require the application of an electric field and thus can obviate some of the challenges associated with electrospinning [3]. Core-sheath pressure spinning (CSPS) represents an advancement on PG and has emerged as an alternative to electrospinning for the production of core-sheath polymeric fibers. CSPS is a simple manufacturing process that utilizes a combination of gas pressure and centrifugal force to generate fibers [4]. To the best of our knowledge, only one study has attempted to

produce ALG fibers using PG. However, ALG could not be spun alone by PG. To address this challenge, a blend of 5 wt% medium-viscosity ALG with 15 wt% polyethylene oxide (PEO) was developed to prepare blended ALG/PEO pressure-spun nanofibers [5].

In this study, we hypothesize that CSPS can enable the fabrication of ALG-based pressure-spun fibers, thereby overcoming its conventional spinnability limitations. The strategy employed in this study involved the use of ALG as the core, with a spinnable material, polycaprolactone (PCL), serving as the sheath. This approach was implemented through the CSPS method to produce beaded core-sheath fibers. The approach presented in this study is advantageous due to its practical implementation and suitability for large-scale manufacturing.

2. Materials and Methods

2.1. Materials and solution preparation

ALG (CAS: 9005-38-3) was acquired from Scientific Laboratory Supplies, UK. PCL pellets (average Mn 80,000) (CAS: 24980-41-4) and chloroform (CAS: 67-66-3) were purchased from Sigma Aldrich, UK. All materials were used as received. The polymeric solutions were prepared by dissolving 4% (w/v) ALG in distilled water and 15% (w/v) PCL pellets in chloroform. Both solutions were then stirred using a magnetic stirrer (MS7-H550-Pro, DLAB, China) at a rotation speed of 260 RPM at an ambient temperature of 16–18 °C for 48 h until a homogeneous solution was obtained.

2.2. Fiber production

All pressure-spun fibers were produced by CSPS utilizing an in-house built instrument and technique that was previously described [4]. In summary, the ALG and PCL spinning dopes (4 mL of each) were loaded into the core and sheath reservoirs, respectively. The spinning process was carried out for 30 s with a gas pressure of 0.2 MPa under ambient conditions (18-19 °C and 49–54% relative humidity). In this investigation, three different rotational speeds of the spinning vessel were applied to produce fibers, namely 2,500, 10,000, and 25,000 RPM, and then the resultant fiber samples were labelled as S1, S2, and S3, respectively.

2.3. Fiber characterization

The morphology of the pressure-spun fibers was observed using scanning electron microscopy (SEM; GeminiSEM 360, Carl Zeiss Microscopy GmbH, Germany). The surface of the samples was first gold sputter-coated with a coating thickness of 3 nm using an EM ACE600 coater (Leica Microsystems CMS, Germany). 100 strands of fiber were randomly selected from each sample depicted in SEM micrographs to calculate the mean diameter and size distribution of the pressure-spun fibers. Fourier transform infrared spectroscopy (FTIR) analyses were conducted on a Nicolet iS50 spectrometer (Thermo Scientific, USA). The collection range was 4000–400 cm^{-1}, with 10 scans per sample at a resolution of 4 cm^{-1}. This analysis aimed to confirm the presence of both materials in the sample. Confocal microscopy (LSM 710, Carl Zeiss, Germany) was used to observe the presence of ALG and PCL as well as to confirm the core-sheath structure in the pressure-spun fibers. For sample preparation, 3 mg of rhodamine-B was added to the core solution, while 3 mg of acriflavine hydrochloride was added to the sheath solution to allow fluorescence visualization. The excitation wavelength was set to 567 and 488 nm to detect the presence of ALG and PCL, respectively.

3. Results and Discussion

3.1. Fiber morphology and size distribution

The morphology of pressure-spun fibers was influenced by variations in rotational speed. Fig. 1.a shows that sample S1 exhibited irregular morphologies, resembling semi-finished fibers with a diameter of 1.98 µm. This irregularity was attributed to the incomplete formation of fibers at 2,500 RPM due to insufficient shear force caused by the low rotational speed of the motor, resulting in most of the polymeric solution being ejected from the nozzles in the form of wet droplets [6]. However, at an elevated motor speed of 10,000 RPM (S2), the formation of continuous fibers with a diameter of 1.61 µm could be achieved. Additionally, the presence of beads with a diameter of 23.8 µm within the fibers was observed. The presence of these beads is attributed to the use of water as a solvent for ALG. Distilled water exhibits a notably high surface tension of approximately 72 mN/m, which could be a contributing factor to the observed beaded morphology. A higher surface tension has been shown to impede fiber formation and potentially give rise to a bead-on-string morphology [7].

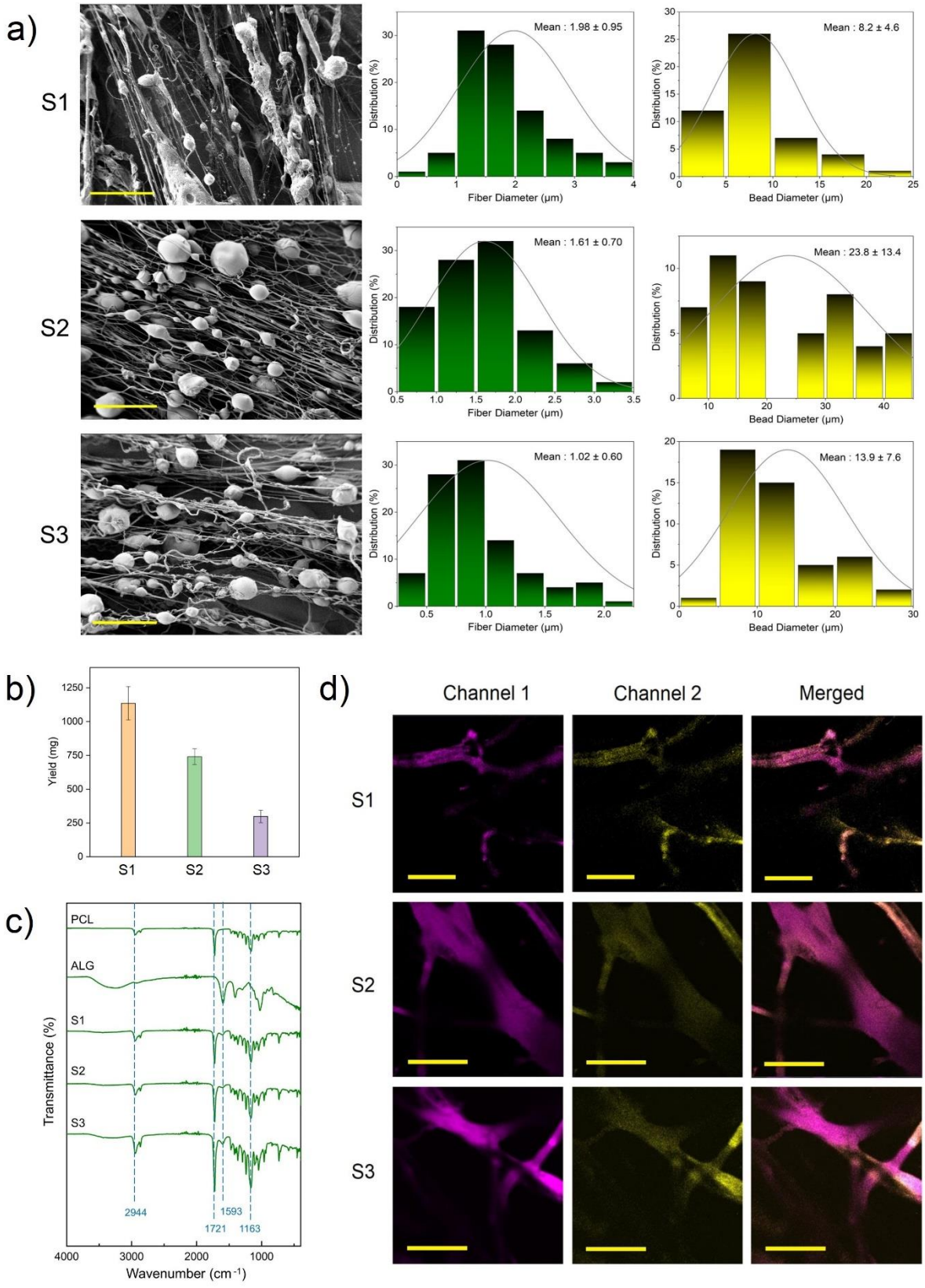

Fig.1: a) SEM micrographs together with size distributions of fibers and beads (scale bars = 100 μm); b) Yield of pressure-spun fiber; c) FTIR spectra of raw materials and fiber samples; and d) Confocal microscopy images of fiber samples (scale bars = 5 μm).

A rotational speed of 25,000 RPM (S3) produced beaded fibers with fiber and bead diameters of 1.02 µm and 13.9 µm, respectively. However, the elevated motor speed also led to the fibers becoming irregular and curly, a consequence of instability at an excessive gyration speed. Theoretically, increasing the gyration speed should increase the centrifugal force on the spinning vessel. A reduction in fiber diameter was thus observed with increasing gyration speed, as an elevated solution evaporation rate is concomitant with increased gyration speed [6, 8]. The beaded core-sheath fibers produced in this study hold promise for applications in advanced drug delivery systems. For instance, they could be used to embed drugs in the beaded regions, thereby reducing initial burst release and facilitating sustained release over an extended period [9].

3.2. Fiber yield

Given that one of the primary advantages of CSPS is its high production rate and scalability, it is important to measure the yield of the fibers produced. Fig. 1.b presents a graphical representation of the yield of all fibers produced at varying rotational speeds. It was observed that the application of a rotational speed of 2,500 RPM (S1) resulted in the highest yield (1,136 ± 123 mg). This yield is equivalent to 142% of the solute being converted to fiber product and exceeds the maximum polymer concentration in the solution (800 mg of total solute). This phenomenon is hypothesized to occur due to diminished airflow at lower speeds, resulting in some fibers being deposited as droplets or being incompletely dried, with residual solvent. As a result, the collected product consisted of wet fibers, which significantly increased the weight of the fiber and thus the apparent yield. Conversely, higher spinning speeds are expected to increase airflow, leading to more effective drying but also loss of material due to deposition in the gyration chamber and lower fiber yields [10]. In accordance with this theoretical assumption, as expected, samples S2 and S3 exhibited lower yields of 741 ± 59 and 299 ± 46 mg, respectively.

3.3. Fourier transform infrared spectroscopy (FTIR)

FTIR analysis was carried out to validate the presence of PCL and ALG. The FTIR spectra of the raw materials and pressure-spun fibers are plotted in Fig. 1.c. A prominent characteristic peak of PCL appeared at 1721 cm^{-1} and represents the C=O stretching vibration. The characteristic peak at 2944 cm^{-1} represented the CH_2 stretching from the polymer backbone [11]. A prominent peak around 1593 cm^{-1} in the ALG spectrum was attributed to the stretching vibration of the carboxyl group (C=O) in the uronic acid units (mannuronic acid and guluronic acid) [9]. The characteristic peaks of both materials were present in the FTIR spectrum of all fiber samples, indicating that the resulting pressure-spun fibers were composed of PCL and ALG.

3.4. Confocal microscopy

In this observation, two detection channels were utilized. The first channel detected the presence of PCL, while the second channel identified ALG. The images obtained through confocal microscopy are shown in Fig. 1.d, which shows the presence of ALG, represented by a yellow color, in the core of the fibers. The purple color emitted by Rhodamine B is localised in the PCL sheath layer. These colors were uniformly distributed, as confirmed by the merged color when the core and sheath colors were combined. This result validated the presence of both materials and core-sheath structures in the produced pressure-spun fibers. Therefore, it can be concluded that CSPS was capable of producing polymeric fibers from ALG material that previously could not be spun using single pressurized gyration.

4. Conclusion

This study demonstrated the successful fabrication of ALG-based polymer fibers using CSPS. The results showed that 10,000 RPM was the best rotation speed to use for producing beaded core-sheath fibers, giving a product with a diameter of 1.61 ± 0.70 µm and a yield of up to 0.74 g. Confocal microscopy confirmed the core-sheath architecture of the fibers, with ALG forming the core and PCL acting as the sheath. FTIR analysis validated the chemical composition of the fibers and confirmed the presence of both polymers in the fiber samples. The findings of this study demonstrate the capability of CSPS to act as a possible method to spin natural polymer materials that have traditionally been regarded as non-pressure-spinnable. Furthermore, this study showcases the potential of CSPS for the fabrication of ALG-based polymeric fibers, which have considerable potential for diverse biomedical applications. Future research will focus on optimizing spinning parameters to further improve fiber properties and exploring the application of these ALG-based beaded core-sheath fibers in advanced drug delivery systems.

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