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Research Letter

The interplay between conductivity and viscosity in electrospinning charged biopolymers

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Abstract

Electrospinning is a valuable polymer processing technique, producing ultrafine fibers with applications in filtration and biomedicine. However, the scope of its impact is narrow due to the limited range of materials compatible with the process. This study reveals conductivity as a pivotal variable governing formation of smooth fibers in charged biopolymer systems. The conventional reliance on rheology to identify the entanglement concentration and tie it to spinnability faced limitations in this two-polymer system comprising polyethylene oxide and sodium alginate. These conclusions can be applied to design electrospinning solutions that expand the range of processable materials, with focus on environmental sustainability.

Introduction

Electrospinning stands as an important polymer processing technique, yielding ultrafine non-woven fibers that are of particular interest in filtration and biomedical applications.[1] However, the impact of these ultrafine fibers is currently limited by the range of materials compatible with electrospinning. While high molecular weight and flexible polymers like polyethylene oxide (PEO), polyvinyl alcohol (PVA), and polyvinyl pyrrolidone (PVP) readily undergo electrospinning, there is a growing interest in expanding the types of materials that can be electrospun. Complex mixtures involving polymer blends, biopolymers, polyelectrolytes, and other charged polymers have generated interest due to their unique contributions to non-woven structures.[2,3] Each of these mixtures must be designed as a formulation to be both functional as a final product and processable through electrospinning.

Despite the popularity of electrospinning in materials research, the production of ultrafine fibers for new, advanced applications is hindered by inherent challenges in balancing desired product properties (i.e., fibers composed of biosourced and biodegradable polymers) and required material property variables that enable processing (i.e., entanglements between polymer chains). Specifically, prior work has shown that smooth fibers can be formed when the polymer concentration is > 2–2.5 × the critical entanglement concentration (C_e),[4,5] but this poses limitations on spinning polymers such as polyelectrolytes, which do not readily entangle. Since many biopolymers are polyelectrolytes, this restricts the development of new sustainable fiber materials.

We use the term biopolymers here to describe polymers that are derived from renewable resources and occur naturally, and some of the most commonly electrospun ones include gelatin,[6] chitosan,[7,8] hyaluronic acid,[9] sodium alginate (SA),[10] dextran,[11] and more. Many of these polymers contain functional groups that can be charged and, thus, are polyelectrolytes, and they also often have stiff backbones due to their polysaccharide nature. This makes them prone to an extended conformation in solution and leads to a low number of entanglements. Researchers, such as Lu et al., have been electrospinning natural and biopolymers for many years, but in most cases they are electrospun with a carrier polymer that improves the spinnability.[12–15] They have also used ions or small molecules to improve spinnability, such as when Ca^{2+} cations were used with SA, providing ionic linkages to stabilize the jet rather than entanglements.[16] However, in these cases, the design of the complex polymer solutions is not systematic, and a stronger understanding of which solution properties control the morphology of the electrospun fibers is necessary.

To harness the electrospinning potential of such materials, a fundamental study of solution conductivity, entanglements, and other variables that may inhibit or promote smooth fiber formation is necessary for developing formulation strategies to control the morphology resulting from electrospinning. In the electrospinning process, a voltage is applied to a conductive polymer solution. Initially, a droplet of the solution is suspended at a needle tip. If the electrostatic forces surpass surface tension forces, the droplet deforms into a Taylor cone, emitting a thin jet. Electrospinning occurs in two stages after the Taylor cone is formed: the straight jet region and the whipping region. In the straight jet, the diameter of the fluid stream decreases with only axisymmetric forces. In the whipping region, Rayleigh instabilities and electric field-induced non-axisymmetric instabilities lead to lateral motion of the jet and/or jet breakup.[17] The whipping instability leads to significant

thinning and drying of fibers, resulting in a decrease in the fiber diameter as it travels to the grounded plate.[18] The Rayleigh instability leads to jet breakup into droplets, resulting in a beads or beads-on-string morphology, which is typically undesirable when electrospinning. To achieve a smooth fiber morphology, stabilizing forces must dominate over the Rayleigh instabilities. For this to occur, a polymer solution has been shown to require a sufficient presence of entanglements, previously demonstrated as > 2.5 entanglements per polymer chain,[4] although other approaches have been used to stabilize the jet, as reviewed in Ewaldz et al.[19]

Throughout studies of electrospinning, many researchers have identified solution variables that must be optimized to achieve the correct balance of forces. These include the polymer concentration, polymer molecular weight, solution conductivity, solvent dielectric constant, surface tension of the solution, and more. These parameters impact the formation of the Taylor cone and the relative strength of each instability and thus the ability to form fibers. For example, as the polymer concentration and molecular weight increase, the number of entanglements increases and the jet is stabilized against breakup via Rayleigh instabilities.[20] Or as another example, solutions with a high surface tension require a high elasticity in the jet to spin, usually provided by higher concentration or polymer molecular weight, but if low surface tension solutions are used, the drive for breakup via Rayleigh instabilities is lower and lower polymer concentrations or molecular weights may be used.[21] With this understanding of the connection between the electrospinning process and the solution properties of simple polymer solutions, we can build to understand how to design spinning solutions for complex materials such as biopolymers and polyelectrolytes.

Electrospinning with biosourced and biodegradable materials is desirable, particularly as we consider the lifecycle of our plastic products and aim to reduce harm of single-use materials. In general, the environmental considerations during product development are becoming increasingly relevant. A promising material that has both environmental and biocompatible advantages is sodium alginate, a polyelectrolyte derived from brown algae.[22] This biopolymer poses challenges for electrospinning due to its rigid and charged structure, hindering entanglements even at high molecular weights and concentrations. Polymer solutions with sodium alginate have a high viscosity even at low concentrations, making processing via electrospinning difficult with sodium alginate alone.[12,13,23] Because of this, sodium alginate has not been able to be electrospun without incorporation of additives. Various strategies have been developed to enable successful electrospinning of sodium alginate (SA) including the use of a carrier polymer, co-solvent system, and surfactant.[12,15,23,24] In our work, we employ PEO as a carrier polymer and Triton X-100 as a surfactant, facilitating the electrospinning of SA in a water-based solution at a 70:30 ratio of SA to PEO.

The use of a carrier polymer is an electrospinning strategy that includes a high molecular weight polymer with many entanglements to prevent breakup of the electrospinning jet when the functional material (polymer or particles) does not provide enough elasticity to the jet. This strategy has been used successfully for many systems[12,13,25] including for SA, where ratios ranging from 1:1 to 3:1 SA:PEO[12] have been successfully electrospun. The other additive we chose is Triton X-100, a surfactant. This lowers the surface tension of water, leading to less drive for breakup of the electrospinning jet through the Rayleigh instabilities. Prior work by Ewaldz et al.[21] showed that the use of Triton X-100 lowers the required elasticity needed to stabilize against jet breakup, which will decrease the amount of the carrier PEO we will need to add. This aids in maximizing the composition of sodium alginate in the fibers, allowing us to reach the 75:25 SA:PEO ratio.

Although the use of a PEO carrier and Triton X-100 surfactant aid in electrospinning through control of the fluid rheology and surface tension, there is another key parameter that comes into play with SA. In this formulation, the presence of a charged polyelectrolyte (SA in this case) brings inherent conductivity through its counterions, resulting in orders of magnitude higher conductivities compared to formulations containing only PEO.[15] Thus, the competition between viscosity, surface tension, and conductivity complicates the design of SA solutions for electrospinnability.[26]

While there is prior work that optimizes the spinnability of SA, as discussed above, achieving successful electrospinning of this polyelectrolyte remains a challenging process requiring extensive trial and error. Literature methods offer valuable starting points for formulation; however, due to the complex interplay of parameters related to electrospinning, more insight into the specific effects of each variable and, importantly, which solution properties are most important, can decrease the number of iterations needed to achieve a new formulation containing sodium alginate. Thus, here we specifically examine the interplay between polymer concentration, which is tied to the solution viscosity parameter, and the conductivity in controlling the window for successful electrospinning of smooth ultrafine fibers. For SA, we show that there is a very narrow electrospinning window to produce smooth fibers and that conductivity is a consistent variable to predict spinnability across all smooth fiber-forming formulations, falling within a precise range regardless of the other formulation properties.

Materials and methods

Materials

All polymers and solvents were used as received. Poly(ethylene oxide) (PEO) (M_w = 1000 kg/mol) was purchased from Alfa Aesar. SA was used with a viscosity of 10.5 Pa-s for a 4 wt% solution in water. Deionized water (18.2 MΩ cm) was obtained from a Milli-Q system. Reagent grade Triton X-100 was purchased from VWR. Solutions were prepared in varying concentrations (w/w) and mixed on a MaxQ 416 HP orbital shaker until homogenous (~48 h).

Solution characterization

Solution conductivity of the electrospinning formulations was measured using a VWR Symphony B30PCI conductivity probe. Samples were tested at room temperature. Zero-shear viscosity measurements were performed using a TA Instruments DHR-3 rheometer with a parallel plate geometry. A shear rate sweep was performed between 1 and 1000 s^{-1} at 25℃. The full measured rheology data for the fiber-forming solutions are shown in the supplementary information Fig. S3. Measured solution parameters for the formulations that resulted in smooth fibers are listed in the supplementary information, Table S1.

Electrospinning

Solutions were loaded into a 5-mL syringe and placed in a syringe pump (KDS-100) with a flow rate from 0.1 to 0.5 mL/hr. The syringe needle was affixed to a top plate, and the collector plate was in a parallel plate configuration with plate distance of 10 inches. An applied voltage of 28 kV was used to drive electrospinning for all samples.

Non-woven characterization

Scanning electron microscopy (SEM) micrographs of the non-woven samples were captured using a Zeiss Ultra60 FE-SEM at a 5–10 kV operating voltage. Samples were sputter coated with gold using a Hummer 6 sputterer prior to imaging.

Results and discussion

When formulating an electrospinning solution, it is typical to conduct tests across a range of concentrations and material combinations to identify successful solutions that produce smooth fibers. This becomes even more involved when each solution must be processed at an optimized set of experimental variables. Due to the expected impact of viscosity, surface tension, and conductivity on electrospinnability of SA, we examined formulations with total polymer concentrations between 1 and 6 wt% and different ratios of SA to PEO as well as those with and without surfactant. No formulations without surfactant formed smooth fibers, so here we only provide the data for those electrospun with Triton X-100, which lowered the surface tension of the solutions to approximately 21 mN/m.

To best analyze the electrospinnability of the formulations, we prepared processing regime maps (Fig. 1), which use symbols to designate which solutions formed fibers (pink triangles), formed a beads-on-string morphology (dark blue circle), led to particles or beads (light blue squares) or did not form a Taylor cone and only dripped from the nozzle (red triangles). Typical images of the fiber, beads-on-string and beads morphology are shown in Fig. 2, and SEM images for all formulations that resulted in smooth fibers are shown in Fig. 3. We prepared these maps based on the total polymer concentration (x-axis) vs. the conductivity, the ratio by mass of SA:PEO or the SA concentration (y-axis) to analyze the spinnability window as a function of different key parameters. Both the total polymer concentration and the SA:PEO ratio are related to the solution viscosity, while the SA concentration is tied to both the conductivity and viscosity.

As can be seen from Fig. 1, only a small number of formulations (4) demonstrated spinnability into smooth fibers, as indicated by the pink triangles. Interestingly, this limited subset of successful formulations showed no significant correlation with overall polymer concentration or the PEO to SA ratio, indicating that the viscosity was not the primary driver of smooth fiber formation. Looking in more detail at Fig. 1(b), it is clear that the fiber-forming formulations do occur at moderate polymer concentrations and SA:PEO ratios, while beads form at low total polymer concentrations (light blue squares), beads-on-string at moderate (blue circles) and dripping occurs at high polymer concentrations (red triangles). This is consistent with known trends for how spinnability changes with viscosity, but due to the charged nature of the SA solutions, there are not distinct cutoff concentrations and non-fiber morphologies occur at all total polymer concentrations.

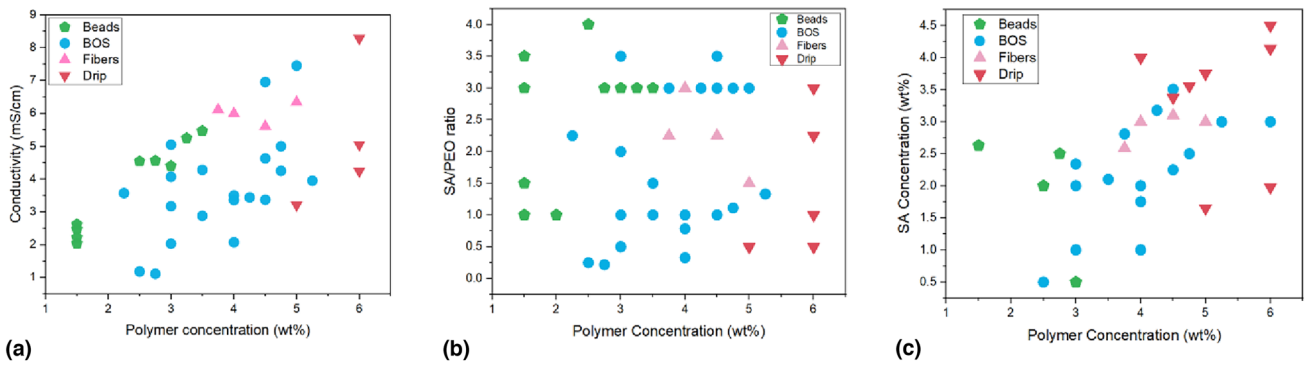

Figure 1. Processing regime maps showing the relationship between total polymer concentration and (a) conductivity, (b) SA:PEO ratio, and (c) SA concentration. The narrow window of smooth fiber formation (pink triangles) is highlighted in all maps, but the clearest trend is seen in Fig. 2(a). Other morphologies are displayed as beads (green pentagons), beads-on-string (BOS) (blue circles), and drip (red upside down triangles).

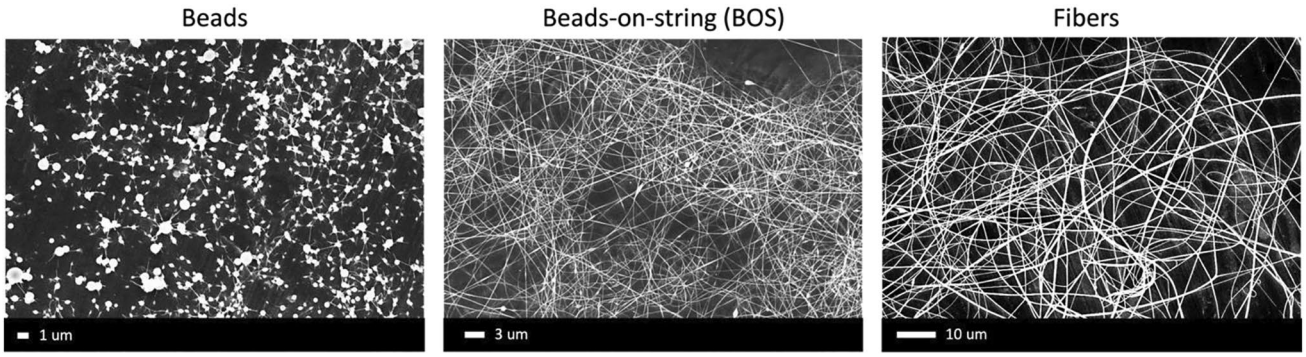

Figure 2. Representative SEM images for each morphology regime.

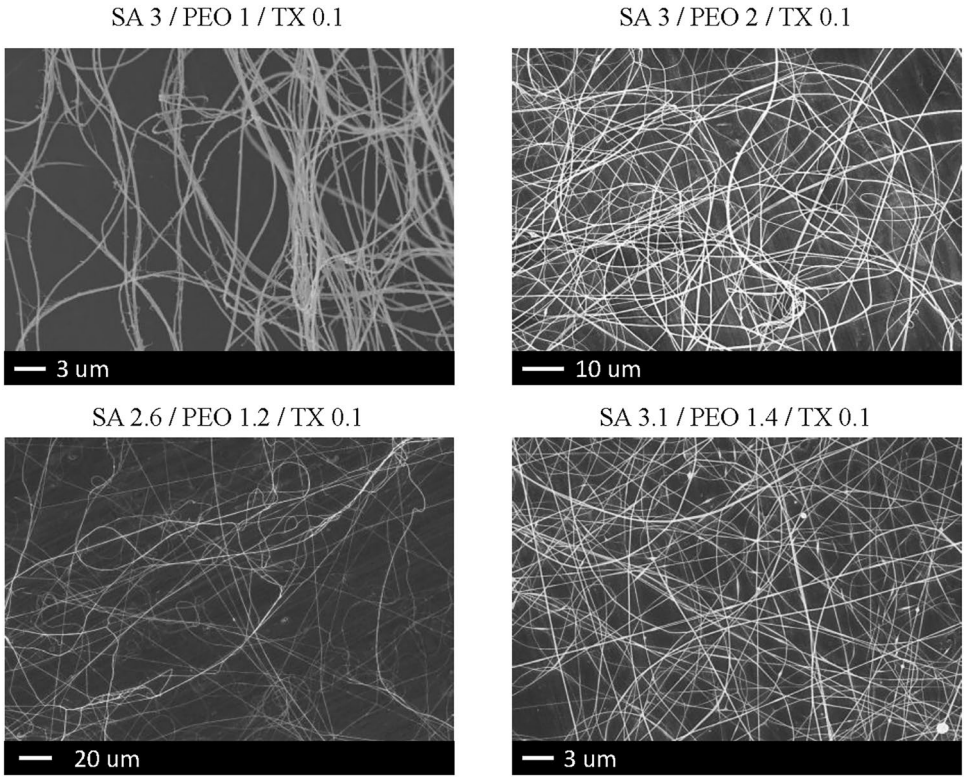

Figure 3. Representative SEM images of the four formulations that resulted in smooth fibers. SA sodium alginate, PEO polyethylene oxide, TX triton x-100.

The most distinct trend in achieving smooth fiber formation is associated with the solution's conductivity. The four formulations that led to smooth fibers occurred in a very narrow conductivity range [Fig. 1(a)]. Conductivity is well known to be an important factor in the outcome of electrospinning. Formulations lacking conductivity (those with a value of zero) are incapable of undergoing the electrospinning process, while a higher solution conductivity reduces the necessary applied voltage for the generation of ultrafine fibers.[27] Previous investigations have explored the addition of salt to increase the surface charge and thereby increase the conductivity and dielectric constant of the polymer solution.[28] This addition can increase the conductivity of typical systems from 0 to 25,000 μS/cm (while the conductivities from the addition of SA range from 20 to 80 μS/cm).[27] Adjusting these solution variables using additives is a common practice to achieve smaller fiber diameters and a smooth fiber morphology. What has been less studied is how the typical design guidelines for using polymer concentration to adjust spinnability[29,30] hold when using a charged polymer to increase the conductivity, motivating us to examine this interplay between electrospinning parameters in more detail.

Statistical comparative analysis of conductivity, polymer concentration, and SA concentration on morphology is displayed in the form of a box-and-whisker plot in Fig. 4. The use

of box-and-whisker plots allows for ease of identification of medians in the dataset, represented by the line in the middle of the box. The lower quartile (Q1) and the upper quartile (Q3), which are the values at which 25% of the data points are below or above, respectively, delineate the bounds of the box. The bars extending from the box are "whiskers" that extend to the minimum and maximum of the data, indicating the full range of the data. In Fig. 4(a) we plot the conductivity measurements for solutions that form each morphological category: beads, beads-on-string (BOS), and fibers. The smooth fiber morphology data points all show conductivities between 5.60 and 6.34 mS/m, which are higher than Q1 values for beads and BOS, though not higher than the full range for BOS. This shows that the conductivity is a critical formulation variable that must be targeted to achieve fibers when electrospinning with a charged system such as SA.

The link between overall polymer concentration and morphology [Fig. 4(b)] is less clear, particularly between BOS and fiber morphologies, which have similar median and upper quartile values. This is likely because both the amount of SA and the amount of PEO contribute to the overall polymer concentration, resulting in a wide range of solution characteristics. Figure 4(c) shows that there is better correlation of the morphology to SA concentration. The smooth fibers have a median that is significantly higher than that for the beads or BOS and there is no overlap in Q1 or Q3 values with BOS or beads morphologies, although again there is some overlap between the full range of datasets. The trends seen in SA amount closely align with those seen for conductivity, which is unsurprising since the addition of more SA increases the conductivity. This provides additional evidence that conductivity of the polymer solutions containing SA is the most impactful parameter to controlling formation of smooth fibers, overwhelming the effect of the viscosity.

While the solution conductivity of samples with smooth fiber morphology is within a narrow range, there are some formulations that have conductivities within the suitable range but were not able to form smooth fibers. These are the ones that fall outside the bounds of 3.75–5 wt% total polymer concentration. The overall polymer concentration and the SA concentration are not simply tied to viscosity or entanglement requirements, as in typical systems with only neutral polymers. In this two-polymer system where one polymer (PEO) provides necessary entanglements for electrospinning, and the other polymer (SA) contributes heavily to viscosity and conductivity, there is no direct correlation to zero-shear viscosity, which explains the lack of correlation to electrospun morphology. Because in this system, zero-shear viscosity cannot be relied upon to find necessary entanglements for smooth fibers, another method of characterization must be relied upon.

Thus, this work shows that a design strategy for a spinnable polymer solution will need to specifically optimize to a narrow conductivity range, which may take many experiments to identify, but that a wider range of total polymer concentration is acceptable, requiring only a few experiments to identify the range. An initial screening study for total polymer concentration followed by a detailed optimization for conductivity could, thus, be an efficient approach to a spinnable polymer solution.

Previous work on designing electrospinnable formulations often relies on rheological characterization, so we examine the utility of such an approach here. Typically, this involves identifying the critical entanglement concentration or molecular weight through shear rheology[21] and designing the formulation to have > 2–2.5 × the critical entanglement concentration for the polymer. This process is laborious, as it requires measurement of the relative viscosity at many polymer concentrations and identification of the point where the slope of relative viscosity versus concentration changes, as shown in Fig. 5(a) for PEO in water with triton X-100.

Figure 5(b) shows that, with mixtures of PEO and SA, the clear transition of the slope at a critical entanglement concentration is not visible. To guide the eye and highlight this, we show a line through the data for polymer solutions that form beads (semi-dilute unentangled regime) and a line through the data for polymer solutions that form BOS (semi-dilute entangled regime). We select this cutoff because it is typically more obvious than the one between BOS and fibers. The viscosity does generally increase linearly with overall polymer concentration [Fig. 5(b)], but although the lines have different slopes,

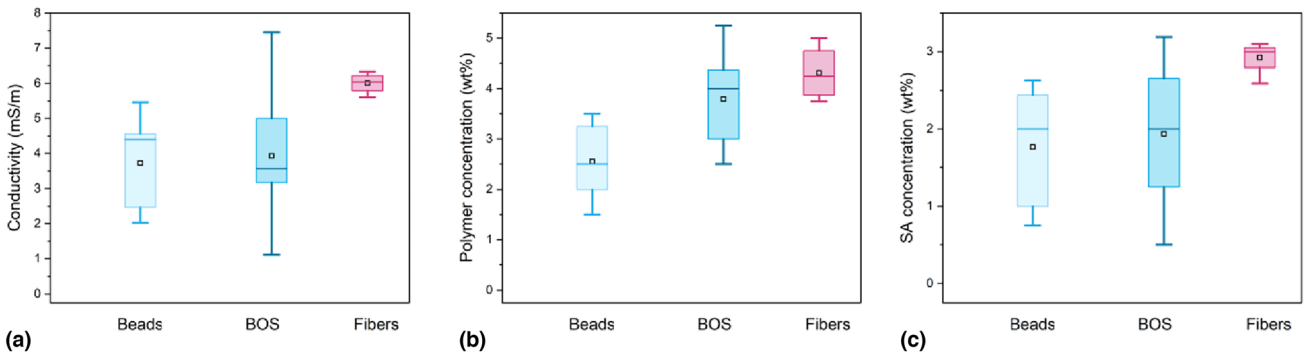

Figure 4. Descriptive statistics of each morphological outcome as it relates to (a) conductivity, (b) polymer concentration, and (c) sodium alginate concentration.

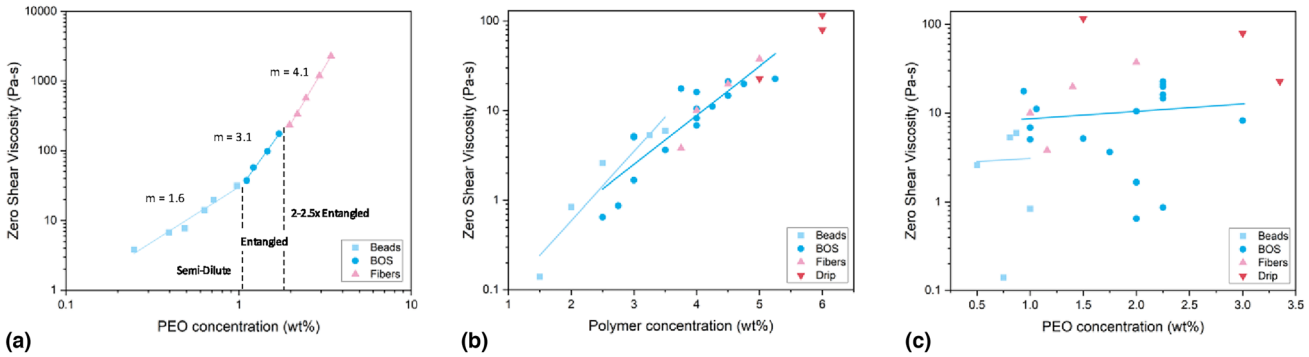

Figure 5. Zero-shear viscosity vs (a) PEO concentration without SA present (adapted from data in Ref. 21), (b) total polymer concentration for the SA/PEO formulations, and (c) PEO concentration in the SA/PEO formulations. Morphologies are displayed as follows: fibers (pink triangles), beads (light blue squares), beads-on-string (BOS) (dark blue circles), and drip (red triangles).

they are not statistically significantly different (0.7716 ± 0.1420 vs. 0.5486 ± 0.0793, with a p value of 0.05 a t test showed that these means were not significantly different). Thus, the rheological data do not show the transition at the critical entanglement concentration that is known to correspond to the beads to BOS morphology transition.[4]

Since PEO is added as a carrier polymer to increase the number of entanglements, we also examined the viscosity as a function of the PEO content in the spinning solution. Note that in this case, the SA amount varies throughout the data, and thus, we will only expect to see a clear trend if the PEO alone determines the solution rheology with no contributions from the SA. As can be seen in Fig. 5(c), this is not the case and there are no trends in how the viscosity varies with the amount of PEO. To compare to Fig. 5(a and b), we still show the linear fit of the solutions that formed a beads morphology (light blue line) and BOS (dark blue line), and again it is clear there is no transition that would indicate a critical entanglement concentration. This indicates that, although PEO is used as a carrier polymer to add entanglements, it does not fully overwhelm the contributions of SA to the rheology of the polymer solution. Overall, this analysis of the solution rheology indicates that for mixtures of a carrier polymer and SA, using the common critical entanglement design strategy is not possible, and the new design strategy proposed here can instead aid in developing new formulations containing this important biopolymer.

Conclusions

In this study, we aimed to improve the ability to design polymer solutions for electrospinning charged biopolymers, focusing on the challenges posed by SA specifically. The key finding of our work lies in the identification of conductivity as a critical variable governing the formation of smooth fibers in charged biopolymer systems. Unlike traditional electrospinning systems where use of the critical entanglement concentration can predict spinnability, our study demonstrates that conductivity serves as a more reliable indicator for predicting smooth fiber formation. The conductivity values within a narrow range (5.60–6.34 mS/m) were found to be associated exclusively with successful smooth fiber morphologies. Further studies with added salt rather than focusing on inherent solution conductivity, as done here, could illuminate the breadth of this region and further interplay between polymer structure and conductivity in predicting spinnability. In addition, with the parameter space examined here, future studies could be planned to deconvolute the effects of conductivity, polyelectrolyte molecular weight, carrier molecular weight, and surface tension, adding to the ability to rationally design biopolymer electrospun fibers.

Contrary to expectations based on conventional systems, our study reveals that the location of the critical entanglement concentration, as determined by shear rheology, could not be identified through measurement of viscosity versus polymer concentration (total or either individual) for mixtures of SA and PEO and thus cannot be used to correlate polymer concentration with morphological transitions in mixtures of a carrier polymer with charged biopolymers. Thus, a design strategy based on screening polymer solutions for the wide range of concentrations that are spinnable paired with a detailed optimization for the solution conductivity provides an effective path to spinning solution design.

The significance of this research extends beyond design strategies for SA ultrafine fibers. The incorporation of conductivity as an optimizing parameter in electrospinning formulations is expected to reduce the trial and error associated with the development of ultrafine fibers using a broader range of starting materials. By identifying conductivity as the key predictor of smooth fiber formation for SA, our findings add value for a more informed formulation process in electrospinning with charged biopolymer systems. This knowledge can be applied to expand the impact of electrospinning, especially with respect to bioderived and biocompatible materials, with applications in biomedical technology and environmental sustainability.

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Author contributions

Carroll-Bassham: conceptualization, data curation, formal analysis, investigation, methodology, writing—original draft, writing-reviewing and editing; Brettmann: conceptualization, funding acquisition, project administration data curation, formal analysis, investigation, writing—original draft, writing: reviewing and editing.

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Data availability

Data will be made available upon reasonable request.

Declarations

Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

Supplementary Information

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