

Ionic Liquid-regenerated Cellulose Fiber: A Promising Next Generation Regenerated Cellulose Fiber

Xin-Yu Wang^a, Kun-Kun Zhu^{a,b*}, Min Zhang^a, Yong-Xin Wei^b, Jing-Jing Yuan^b, Jin-Ming Zhang^{c*}, Jin-Feng Wang^{a*}, and Jun Zhang^c

^a National Local Joint Laboratory for Advanced Textile Processing and Clean Production, Wuhan Textile University, Wuhan 430200, China

^b State Key Laboratory of New Textile Materials and Advanced Processing, Wuhan Textile University, Wuhan 430200, China

^c CAS Key Laboratory of Engineering Plastics, Institute of Chemistry, Chinese Academy of Sciences (CAS), Beijing 100190, China

Electronic Supplementary Information

Abstract As the global textile industry has accelerated its transition to a circular economy, iterative innovation in regenerated cellulose fibers has become a key industry focus. With viscose fiber having been industrialized for over a century and lyocell fiber gaining market recognition because of its environmentally friendly process, which is the next regenerated cellulose fiber. Herein, ionic liquids with low vapor pressure, non-flammability, relatively simple recovery, and high dissolution efficiency were used to fabricate regenerated cellulose fibers. The viscose and lyocell properties of the fibers were systematically compared, including microscopic morphology, dyeing behavior, fibrillation resistance, mechanical properties, yarn-forming capacity, and fabric performance. The ionic liquid (IL) fiber exhibited a smooth surface and circular cross-section, with the highest tensile strength, moderate dyeing and fibrillation properties, and similar spinning and weaving performance. This work can provide a reference for the commercial application of regenerated cellulose fibers fabricated from ionic liquid.

Keywords Cellulose; Ionic liquid; Fiber; Viscose; Lyocell

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INTRODUCTION

For thousands of years, fiber has been an integral part of human life, fulfilling basic requirements such as clothing, storage, building materials, and everyday items.^[1–3] Natural fibers, such as cotton fibers, have consistently dominated the textile sector owing to their exceptional moisture absorption, skin-friendly softness, and outstanding mechanical properties, while the high land and water consumption of cotton crops has long constrained production.^[4] Synthetic fibers such as polyester fibers have huge outputs and low cost, and their non-degradable nature seriously threatens environmental safety.^[5] Thus, regenerated cellulose fibers are receiving increasing attention because they overcome the limitations of synthetic fibers and retain the beneficial properties of natural cotton fibers.^[6–8] Commercially successful regenerated cellulose fibers, including viscose, cuprammonium rayon, and lyocell fibers, have been developed using distinct solvent systems. However, cuprammonium fiber, which is hindered by its high production cost and minimal mar-

ket presence, has attracted limited research and industrial interest.

Viscose and lyocell are currently the only industrially scaled technologies for producing regenerated cellulose fibers. However, both fiber types face critical challenges associated with their solvents.^[9] The viscosity process relies on toxic carbon disulfide, which releases hazardous byproducts that endanger workers and ecosystems.^[10] Lyocell employs *N*-methyl morpholine-*N*-oxide (NMMO) to dissolve cellulose in an aqueous solution for fiber generation, which offers environmental advantages. Notably, NMMO can achieve a recovery rate exceeding 99% during production, further highlighting its eco-friendly advantages. However, lyocell fibers suffer from solvent instability, cellulose degradation, and energy-intensive solvent recovery owing to byproduct formation.^[10] Driven by rising demand for sustainable textiles, the development of green spinning technologies is imperative. This critically depends on eco-friendly solvents capable of efficiently dissolving cellulose while minimizing environmental and operational drawbacks.

Over the past two decades, ionic liquids have emerged as promising green solvents for cellulose dissolution and have been explored for the production of regenerated cellulose fibers. Rogers *et al.*^[11] found that cellulose could be directly dissolved in hydrophilic ILs such as 1-butyl 3-methylimidazoli-

* Corresponding authors, E-mail: kkzhu@wtu.edu.cn (K.K.Z.)

E-mail: zhjm@iccas.ac.cn (J.M.Z.)

E-mail: jinfeng.wang@wtu.edu.cn (J.F.W.)

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um chloride (BmimCl), without requiring prior activation or pretreatment. Zhang *et al.*^[12] reported a new type of highly efficient ionic liquid, 1-allyl-3-methylimidazolium chloride (AmimCl), that can be used directly for the dissolution and regeneration of cellulose. Complex cellulose resources, such as corn husk,^[13] wheat straw^[14] and textile waste, can be directly dissolved in AmimCl without pretreatment, which demonstrates their high dissolving capacity, safety, and reliability.^[15]

This work compares regenerated cellulose fibers from ionic liquids with viscose fibers and lyocell fibers in terms of the spinning process, micromorphology, structural characterization, dyeability, spinning and weaving properties, and fabric characteristics. The regenerated cellulose fiber fabricated from ionic liquid exhibited tensile strengths of (814.2±63.0) and (694.9±64.4) MPa for the dry and wet states, respectively, and outperformed viscose and lyocell. The dyeability and fibrillation resistance of ionic liquid fibers are intermediate between those of viscous and lyocell fibers. Moreover, ionic liquid fibers can be spun into yarns and woven into fabrics, like viscose and lyocell fibers.

Materials

Viscose filament (120D/30F) and Lyocell filament (120D/60F) were Purchased from Shaoxing Bo Nook Chemical Fiber Co. and Hengchuang Fabric Analysis Studio, Keqiao District, Shaoxing, respectively. An ionic liquid-regenerated cellulose filament (122D/30F) was provided by the Wuqing Pilot Plant Base of the Institute of Chemistry, Chinese Academy of Sciences.

Dyeing Property

As shown in Fig. S1 (in the electronic supplementary information, ESI), a solution with a dye concentration of 1wt% was prepared at room temperature, to which 1 g of filament sample was added (bath ratio of 1:20). Following a 30-min stirring process in a water bath thermostatic shaker at 60 °C, Na₂SO₄ was added, and the mixture was stirred for an additional 30 min at the same temperature. Subsequently, Na₂CO₃ was added to the mixture at the same temperature, and the stirring process continued for 1 h. Finally, the dyed fibers were saponified at 90 °C for 20 min in a soap solution with a bath ratio of 1:30. Three commercial-grade reactive dyes (red 195, yellow 176, and blue 194) were used without purification. The dye exhaustion ratio (*E*) was calculated as follows:

$$X = \frac{A_b}{A_a \times n} \times 100\% \quad (1)$$

$$E = 100\% - X \quad (2)$$

where *X* is the dye content in the residual dye solution and *A_a* and *A_b* are the absorbance values of the standard dye solution and residual dye solution, respectively, *n* is the concentration ratio (g/L) of the standard dye solution to the residual dye solution. The dye-fixing ratio (*F*) was calculated using the following equation:

$$Y = \frac{A_d}{A_c \times n} \times 100\% \quad (3)$$

$$F = E - Y \quad (4)$$

where *Y* is the unfixed dye content in the residual soaping solution and *A_c* and *A_d* are the absorbance values of the standard soaping solution and the residual soaping solution, respectively.

n is the ratio of the concentration (g/L) of the standard soaping solution to that of the residual soaping solution. The color strength (*K/S*) was determined using a Datacolor SF600 computer color-matching system (Datacolor International, USA). A standard whiteboard was used as the control during the test.

Fibrillation Test

The regenerated cellulose fibers were then subjected to a profibrillation test utilizing the ultrasonic shock method previously documented. The tows were meticulously combed to ensure that the fibers were aligned in the same direction. A precise quantity (2 mg) of fiber was then weighed, knotted in the middle, and meticulously cut flush at both ends to a precise length of 20 mm. A 50 mL beaker was used to contain 20 mL of deionized water, into which the prepared fiber samples were immersed. The specimen bottles were then placed in an ice-water bath and processed for 30 min at 500 W using a Xinzhi Cyto-clast pulverizer. The fiber samples were removed, and the filter paper was drained to remove surface moisture. The samples were then kept wet for microscopic observation. The unknotted portion of the 2–5 mm sample was then exercised and positioned directly onto a clean slide. Glycerol was then added dropwise to disperse the fiber samples, after which the slide was gently covered, and the fiber state was observed under a microscope.

Fabric Production

In the present study, three types of regenerated cellulose filaments were subjected to a twisting process at a rate of 30 twist/10cm using a merging and twisting machine. The resultant twisted strands were then woven on a small machine loom, and the plain fabric, which had a width of 10 cm, was woven using the smooth threading method of the eight pages healed.

Characterization

The regenerated cellulose filaments were prepared as cross-section samples using a slicer, and the cross-sectional morphology of the three types of fibers was observed using a polarized light microscope (CX40P) with a 50× objective lens. The surface morphology of the fibers after gold spraying was observed using a scanning electron microscope (SEM; PhenomPure, China Co., Ltd., Fuhner Scientific Instruments (Shanghai) Co., Ltd.) at an accelerating voltage of 5 kV.

The polymerization and crystallinity indices of the three fibers were calculated by gel permeation chromatography test. Fourier transform infrared spectroscopy (FTIR, Nicolet6700, Shimadzu Co., Ltd., Japan) was performed using the reflectance method in the wavelength range 4000–500 cm⁻¹. The crystallinity index of the three fibers was investigated using an X-ray diffractometer (XRD, PANalytical, Holland) under the test conditions of the reflectance method and a scanning range of 2θ=5°–90°. The tensile strengths of the three types of fibers were measured using a universal tensile tester (YG004, Laizhou Electronic Instrument Co., Ltd. China). The samples were stretched at room temperature at a rate of 10 mm/min, with a firmware separation distance of 10 mm. For each prototype, at least 30 independent specimens were measured to ensure that the strength and elongation values were credible under a constant pretension. Fabric coolness was evaluated using a fabric coolness tester (YG(B)616NX, Wenzhou Da Rong Spinning Instrument Factory) to assess the

coolness of the fabrics in contact with instantaneous coolness.

RESULTS AND DISCUSSION

Comparison of Regenerated Cellulose Fiber Production Process

Viscose (VC) fibers are produced from cellulose through chemical processing to yield cellulose sulfonate, which is subsequently dissolved in dilute hydrochloric acid to form a viscous solution. This solution was subjected to wet spinning in a coagulation bath (Fig. 1a). While the viscose production process demonstrates favorable moisture absorption and dyeability, the carbon disulfide (CS_2) employed in the xanthation step presents significant environmental and safety concerns owing to its toxicity, flammability, and potential explosivity. Moreover, the decomposition byproducts of this process have substantial environmental impact. In contrast, lyocell (LY) fibers are manufactured through a more sustainable approach using *N*-methylmorpholine-*N*-oxide (NMMO) as a recyclable, non-toxic solvent for cellulose dissolution, followed by dry-jet wet spinning (Fig. 1b). This process yields fibers with superior mechanical properties, including high tensile strength and excellent drape characteristics. However, the LY fibers demonstrate a tendency toward microfibrillation when exposed to wet alkaline conditions, resulting in surface fibrillation. The Lyocell production process comprises five principal stages: dissolution, filtration, regeneration, washing and finishing.^[16]

Ionic liquid-mediated regeneration of cellulose fibers (IL fibers) failed to achieve optimal structural properties. This process employs ionic liquids as solvents for natural cellulose

dissolution, followed by wet-spinning regeneration in a coagulation bath (Fig. 1c). Compared to conventional NMMO-based lyocell production, the ionic liquid process demonstrates comparable environmental benefits while offering superior economic advantages, particularly in solvent recovery efficiency. The significantly lower steam pressure requirements for ionic liquid recovery translate to reduced energy consumption compared with traditional solvent recovery systems.^[17] This study systematically evaluated the dyeing characteristics of three fiber variants to assess the suitability of IL-regenerated cellulose fibers for textile applications.

Comparison of Regenerated Cellulose Fiber Properties

The IL fibers exhibited good flexibility and a silklike luster in their appearance, similar to commercialized VC and LY fibers (Fig. 2a). Both the IL and LY fibers were regenerated directly from the cellulose gels, which were formed by physical cross-linking owing to the strong self-association tendency of the cellulose chains in the system.^[18] IL fibers have circular cross sections, which are markedly different from the lobulated shape of VC fibers, similar to LY fibers (Fig. 2b). During the formation process of VC fibers, the cross-sections of VC fibers were approximately round for fibers straight from the spinneret, but then shrank because of acid-alkali neutralization and the reverse of the xanthation reaction, leading to the lobulate structure.^[19] The surface of the VC fibers exhibited longitudinal grooves, the formation of which likely resulted from multiple factors. These include the fluid state of the material during nozzle extrusion, followed by fiber shrinkage during the drying and drafting processes. In contrast, the LY and IL fibers exhibited smoother surfaces (Fig. 2c). These differ-

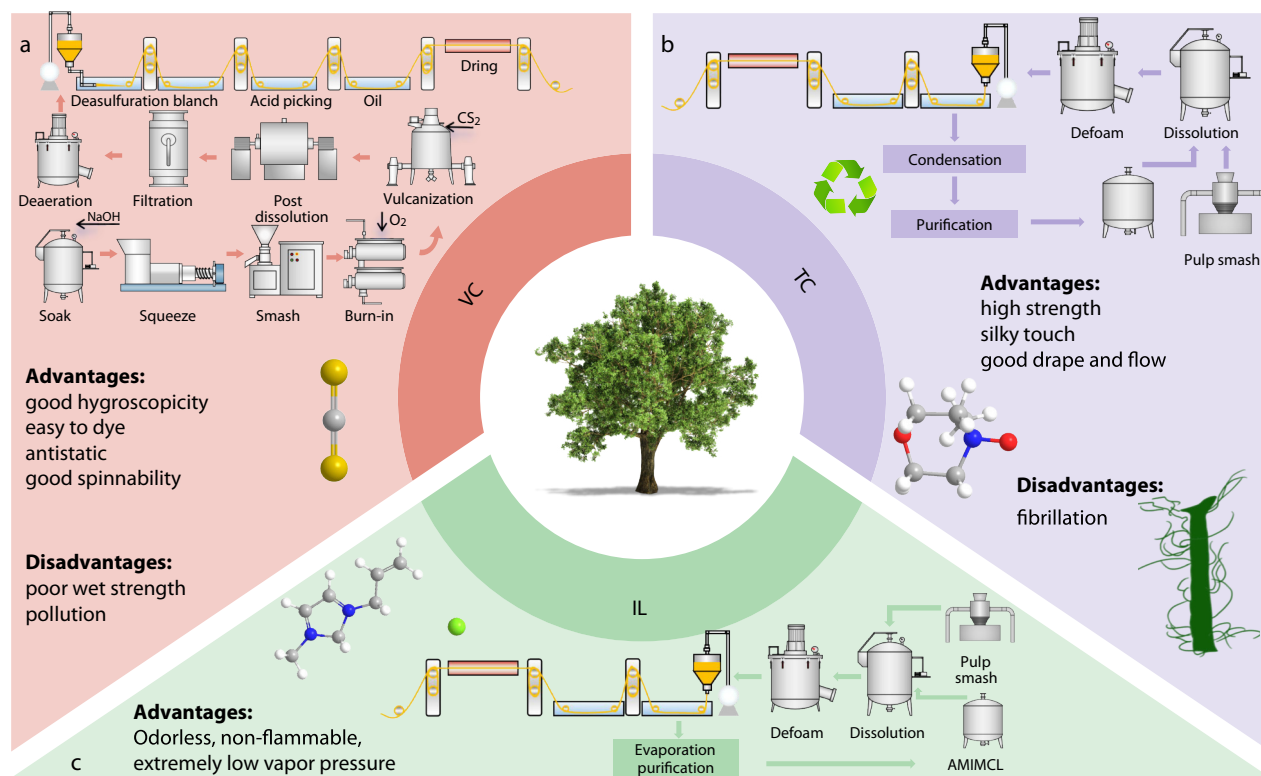


Fig. 1 Process flow for (a) viscose, (b) lyocell and (c) ionic liquid regenerated cellulose fibers.

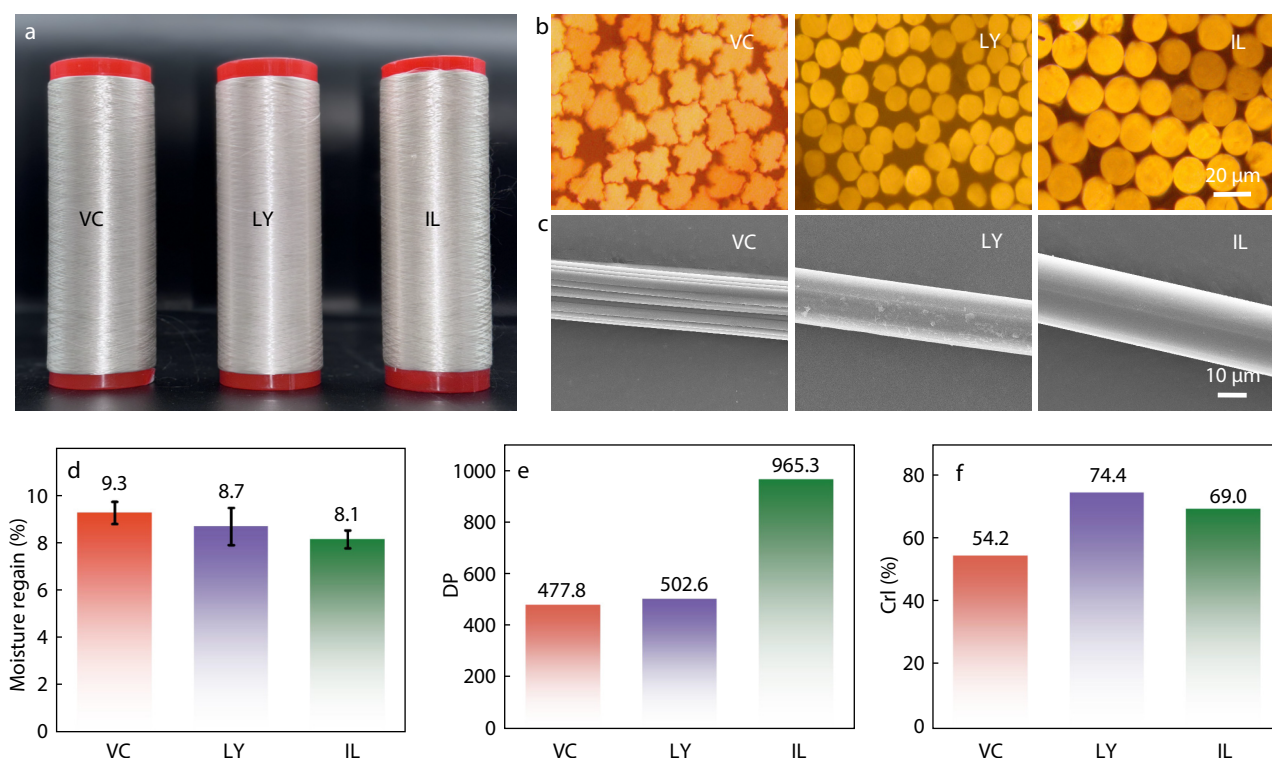


Fig. 2 (a) Photograph, (b) microscopic cross-sectional morphology, (c) surface SEM images, (d) moisture regain, (e) degree of polymerization and (f) crystallinity index of VC, LY and IL fibers.

ences in micromorphology have an impact on other fiber characteristics. Moisture regain in textiles refers to the amount of moisture a fiber or fabric can absorb and remain under standard atmospheric conditions, which influences its properties and performance in textile applications. The results of the moisture regain test of the fibers are shown in Fig. 2(d). The moisture regain rates of the three fibers, VC (9.3%), LY (8.7%), and IL (8.1%), were comparable to each other and closely matched that of the natural cotton fiber (about 8.5%). This similarity suggests that these regenerated cellulose fibers can provide comfortable properties comparable to cotton in fabric applications.^[20] As shown in Fig. 2(e), the degrees of polymerization (DP) for the VC, LY, and IL fibers, respectively. This was closely related to the preparation process of the three fibers. Because of the different spinning processes, the crystallinity index (CrI) of the LY and IL fibers was higher than that of the VC fibers (Fig. 2f).

Dyeing Properties

In addition to their mechanical properties, the high-quality dye characteristics and color strength of fibers play crucial roles in their textile industry applications.^[21] Fig. 3 presents a comparative analysis of the dye exhaustion ratio, dye fixation ratio, K/S values, and color indices of the fibers. When reactive red 195 (RR), reactive yellow 176 (RY), and reactive blue 194 (RB) were used as dyes, the dye exhaustion of the IL fibers was 74%, 90%, and 85%, respectively (Fig. 3a), higher than that of the LY fibers (65%, 86%, and 85%), and lower than that of the VC fibers (83%, 92%, and 88%, respectively). The fixation ratios of the IL fibers were also higher than those of the LY fibers and lower than those of the VC fibers for all reactive dyes (Fig. 3b). These differ-

ences in performance likely stem from their distinct structural characteristics (Fig. 2). The LY fibers exhibited the highest crystallinity, which facilitated the penetration of dye molecules into the fiber interior. The K/S value measurements (Fig. 3c) further confirm the enhanced dyeability of the IL fibers, with all three reactive dyes producing deeper, more vibrant colors than in the LY fibers. As seen from the values of a^* and b^* in Fig. 3(d), the three groups of fibers were still relatively similar in terms of hue, only the difference in color shades. This superior dye performance is visually evident in Fig. 3(e), where the dyed cellulose fiber displays noticeably brighter coloration than its VC counterpart. Notably, the surface functional groups or surface charge of regenerated cellulose have a significant impact on the dyeing process.^[22] The VC, LY, and IL fibers used in this study were pure cellulose without any modification, so the surface functional groups were only hydroxyl groups; thus, the surface charges of the three fibers were indistinguishable. The differences in the dyeing performances of the VC, LY, and IL fibers can be attributed to their distinct structural characteristics.

Fibrillation Tendency

Fiber fibrillation is defined as the process of fiber processing or utilization, whereby the fiber surface of the microfibers is separated from the fiber body by mechanical force, chemical action, or other factors, resulting in the formation of a fine, fluffy, or filamentary structure. Compared with VC fibers, LY fibers have a high tendency for fibrillation because of the few lateral intermolecular forces between separate elementary fibrils, owing to their isolated and intact cellulose crystallites, high alkaline affinity, and excellent water retention capacity.^[23] During fibrillation, the external crystalline part of the fiber breaks and peels off

lengthwise from the fibers.^[24] Controllable fibrillation may endow fabrics with various special effects, such as peach skin, silk touch, and soft denim.^[25] Excessive fibrillation not only affects the smooth texture and appearance of the fabric, leading to a worn or aged appearance, but also diminishes its strength, durability, and overall wearability.^[26] As shown in Fig. 4, the fibrillation characteristics of the VC, LY, and IL fibers were com-

pared. The fibrillation behavior of VC and LY fibers represents two extremes: the former exhibits no fibrillation, whereas the latter demonstrates severe fibrillation. Notably, IL fibers display an intermediate level of fibrillation, offering potential for the development of controllable fibrillated fibers. In addition, dyeing improved the anti-fibrotic characteristics of all fibers to some extent (Fig. 4a). This is because the dyes (Fig. 4b) act as crosslink-

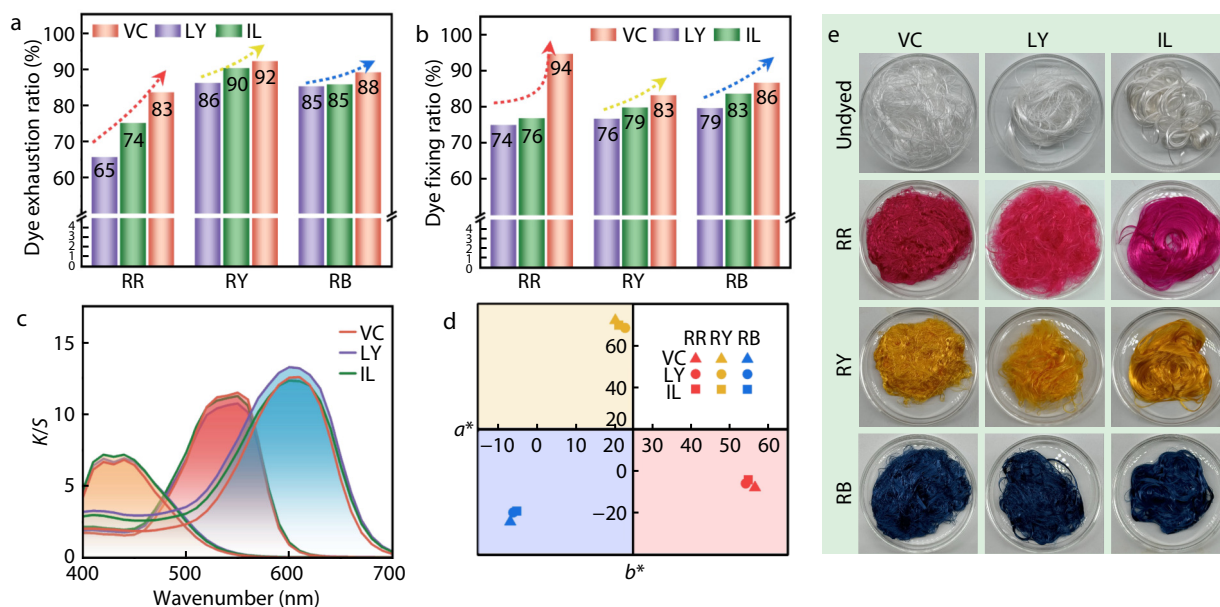


Fig. 3 (a) The dye exhaustion ratio, (b) dye fixing ratio, (c) K/S value and (d) color indices of VC, LY and IL fibers; (e) Photographs of cellulose fibers dyed with reactive dyes.

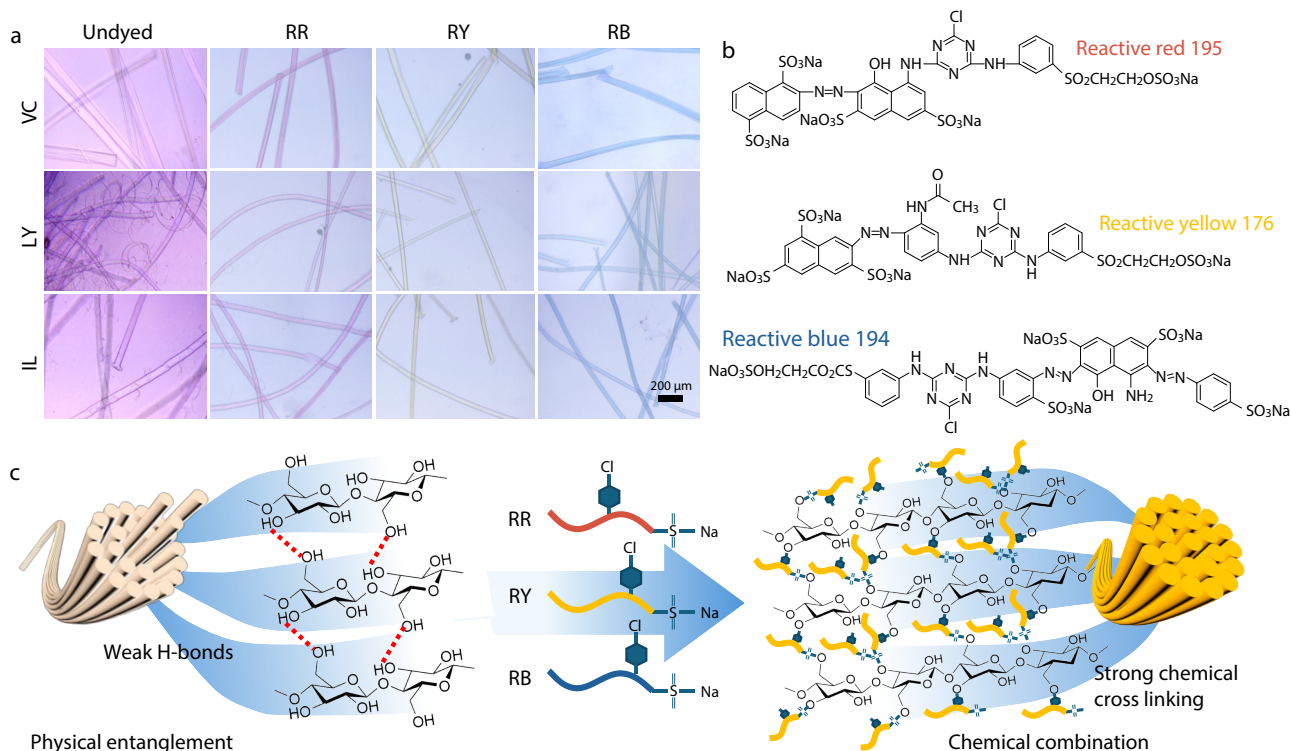


Fig. 4 (a) Photographs of fiber protofibrillation of each fiber before and after dyeing by ultrasonic vibration; (b) Chemical structures of reactive red 195, reactive yellow 176 and reactive blue 194; (c) Mechanism diagram of inhibiting fiber fibrillation after staining.

ing agents with the cellulose molecular chain during the dyeing process, which increases the interaction force between the microfibrils in the warp direction of the fibers (Fig. 4c). The two electrophilic species that participate in the cross-linking reaction are vinylsulfone and the chlorine atoms of the monochlorotriazine residue.^[27]

Mechanical Properties

The mechanical properties of fibers are the basis for determining whether they have commercial value. The tensile strength, elongation at break, Young's modulus, and toughness of the IL fibers were compared with those of the VC and LY fibers in the dry and wet states (Figs. 5a–5c). The average tensile strength of IL fibers is decreased from (814.2 ± 63.0) MPa in the dry state to (694.9 ± 64.4) MPa in the wet state, corresponding to a reduction about 14.7%, because the hydrogen bonds between cellulose chains of the fiber are partly destroyed by the interactions of infiltrated water molecules. As demonstrated by FTIR testing (Fig. S2 in ESI), a broad absorption band at approximately 3300 cm^{-1} was observed in the FTIR curve of the wet state, suggesting that intermolecular hydrogen bonds occurred between glucose residues and aqueous molecules during hydration.^[28] The tensile strength of the IL fibers was much higher than that of the VC and LY fibers (Fig. 5b). Moreover, the average modulus and toughness of IL fibers in the dry state were (21.5 ± 1.9) GPa and (60.2 ± 7.6) MJ/m², respectively, significantly higher than that of VC and LY fibers (Fig. 5c). After dyeing with reactive dyes, all regenerated cellulose fibers exhibited increased tensile strength (Figs. 5d–5f). The IL fibers showed the highest tensile strength, modulus, and toughness in both dry and wet states.

Fabric Performance

A textile yarn is a continuous strand of staple or filament fibers arranged in a form suitable for weaving, knitting, or other forms of fabric assembly. As the VC, LY, and IL multi-filaments were all

ready yarns, some twists were added to make them easier for subsequent textile processing (Fig. 6a). All of these twisted yarns exhibited the same diameter without any hairiness (Fig. 6b), which would reduce pilling or hooking problems while improving wrinkle resistance and stability, unlike cotton yarns.^[29] Compared with the VC and LY fiber yarns, the IL fiber yarns exhibited a loose structure because of the highest modulus. Fig. 6(c) illustrates the corresponding coefficients of variation (CV%) of the yarns as determined from the longitudinal electron micrographs of the three yarns. Among the variables under consideration, IL exhibited the highest coefficient of variation, followed by VC, while LY demonstrated the lowest. The lyocell process is characterized by its ability to regulate the quality and properties of the fibers, resulting in a uniform fiber composition that minimizes the coefficient of variation of the yarn. IL yarns exhibit a substantial coefficient of variation, which is attributable to the substandard uniformity of the fibers in terms of thickness and length during the manufacturing process. This is a consequence of the inherent characteristics of the raw materials and manufacturing process, resulting in a high inhomogeneity rate of the yarn. Subsequent adjustments to the spinning process parameters are required to obtain fibers with enhanced performance. The number of hairs in VC and LY exhibited a marked increase at a shorter length of hairs, subsequently decreasing with an increase in hair length. Conversely, the number of hairs of IL was lower at shorter hairs. IL yarns exhibited a reduced number of hairs at shorter plume lengths, and as the plume length increased, the number of hairs decreased more gently yet remained at a higher level compared to VC and LY (Fig. 6d), further validating the observed coefficients of variation. The yarns showed different linear densities of 22.3, 21.3 and 19.4 tex for VC, LY, and IL yarns, respectively (Fig. 6e). As shown in Fig. 6(f), VC yarns exhibit the best mechanical properties. The performance of IL yarns is similar to that of LY yarns, suggesting their

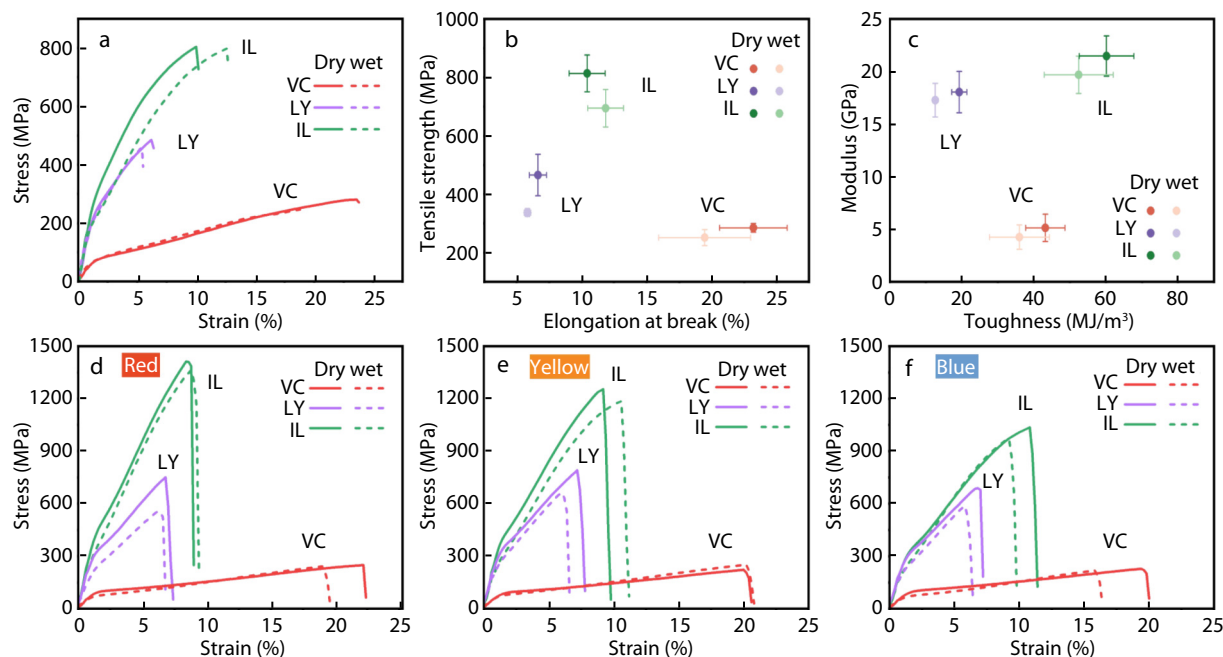


Fig. 5 (a) Strength-strain curves, (b) tensile strength and elongation at break, and (c) toughness and modulus of the fibers under dry and wet state; Strength-strain curves of the fibers after dyeing by (d) reactive red 195, (e) reactive yellow 176 and (f) reactive blue 194.

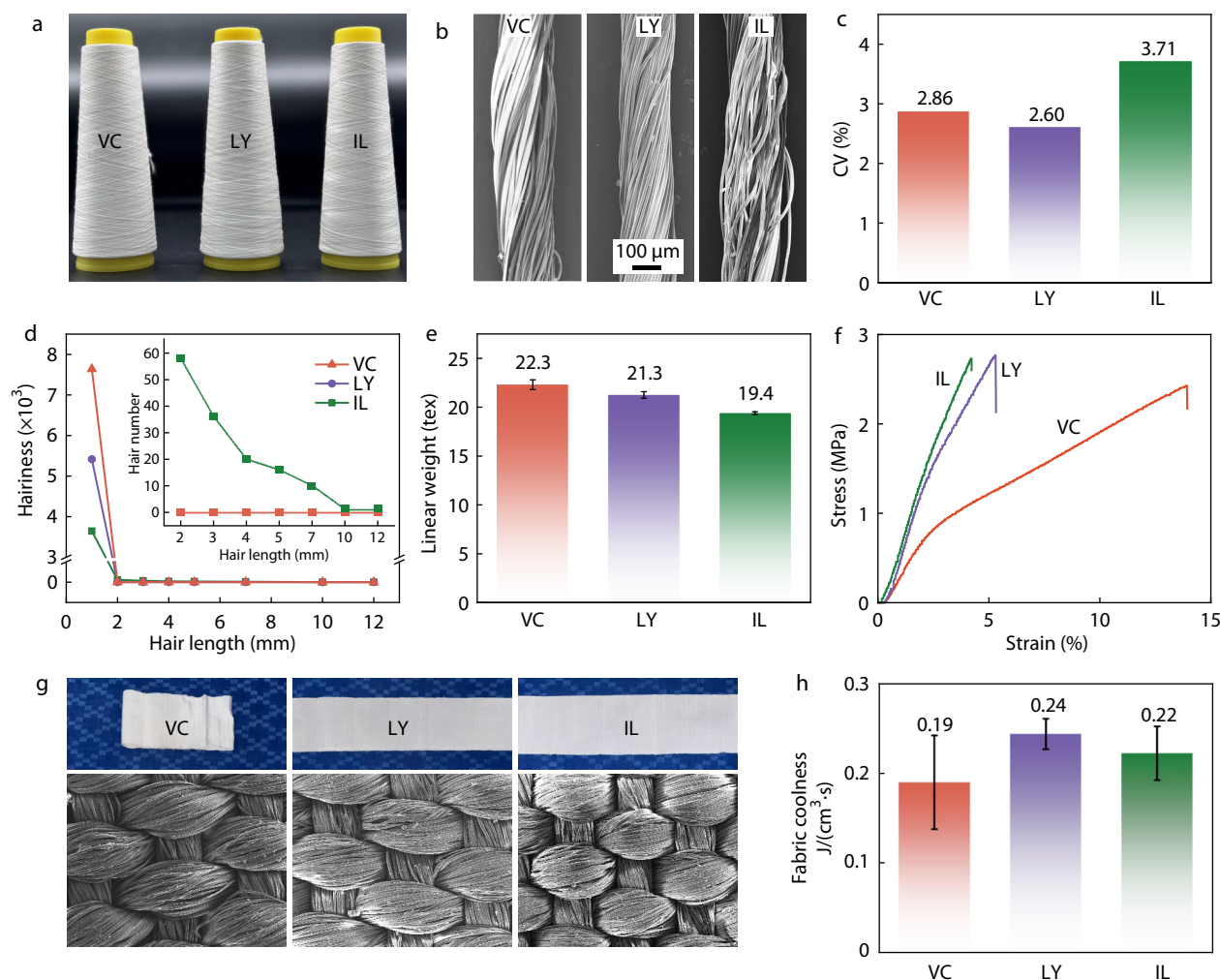


Fig. 6 (a) Photograph, (b) SEM images, (c) coefficients of variation, (d) hairiness, (e) linear density and (f) stress-strain curves of VC, LY and IL fiber yarns. Photograph and SEM images (g), and fabric coolness (h) of VC, LY and IL fiber fabrics.

practical application potential. Compared with laboratory-produced IL yarns, VC and IL demonstrated excellent stability and a relatively regular structure during the spinning process. IL fibers can be used to prepare fabrics with uniform texture and dense microstructures, such as VC and LY fibers (Fig. 6g). The coolness of IL fiber fabric is measured to be $0.22 \text{ J}/(\text{cm}^2 \cdot \text{s})$, between the VC and LY fiber fabrics (Fig. 6h). Fabric coolness is the sensation of a temperature drop when the skin contacts cooler fabric. This occurs as heat is transferred from the skin to the fabric, triggering a cooling response *via* nerve endings. This effect is primarily due to the thermal conductivity of the fabric (how well it transfers heat) and its moisture-wicking properties.^[30]

CONCLUSIONS

In summary, this study systematically compared ionic liquid fibers with viscose and lyocell fibers in terms of micromorphology, dyeing properties, fibrillation, mechanical properties, yarn properties, and fabric performance. The experimental results showed that the ionic liquid fiber outperformed the viscose and lyocell in terms of tensile strength, and its dyeing properties and anti-fibrillation were intermediate between the two fibers. Giv-

en their green processing characteristics, ionic liquid fibers have the potential to become the next generation of regenerated cellulose fibers after viscose and lyocell fibers.

Conflict of Interests

The authors declare no interest conflict.

Electronic Supplementary Information

Electronic supplementary information (ESI) is available free of charge in the online version of this article at <http://doi.org/10.1007/s10118-025-3465-9>.

Data Availability Statement

Data substantiating the findings of this study can be accessed from the corresponding author upon reasonable request.

ACKNOWLEDGMENTS

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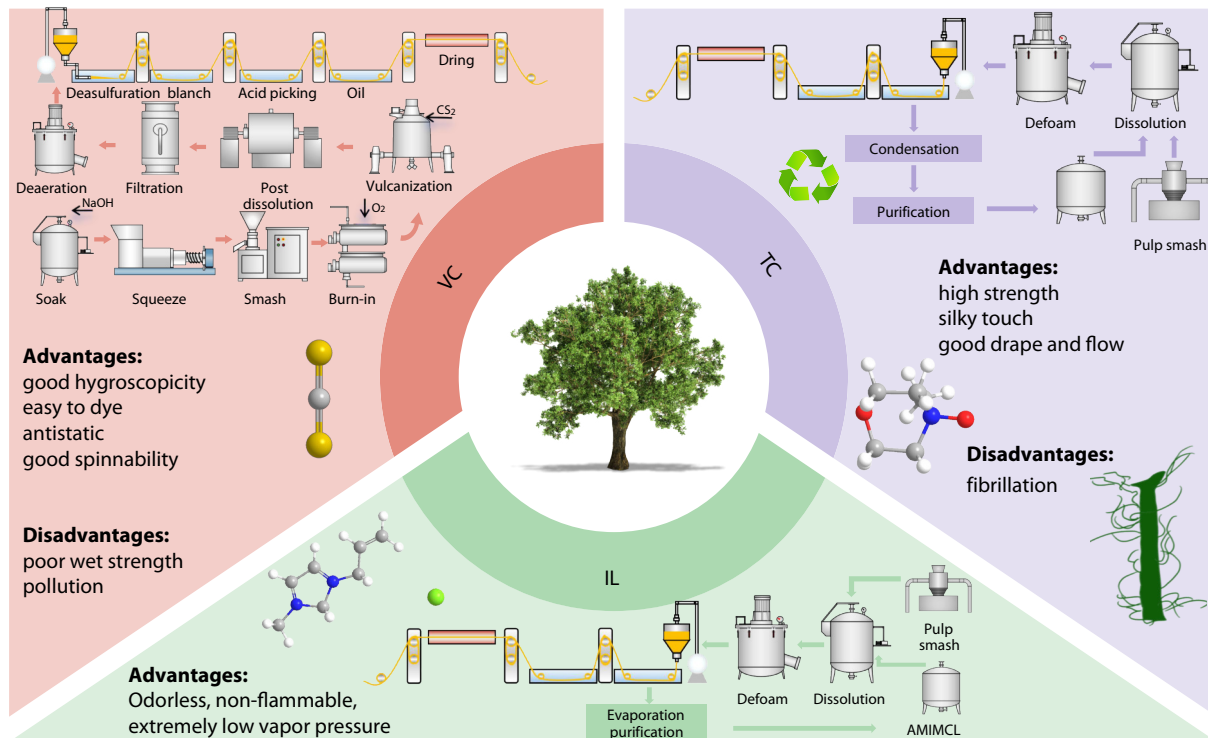
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Graphical Abstract

Ionic Liquid-regenerated Cellulose Fiber: A Promising Next Generation Regenerated Cellulose Fiber

Xin-Yu Wang, Kun-Kun Zhu, Min Zhang, Yong-Xin Wei, Jing-Jing Yuan, Jin-Ming Zhang, Jin-Feng Wang and Jun Zhang
Wuhan Textile University; Institute of Chemistry, Chinese Academy of Sciences (CAS)

Ionic liquid (IL)-made regenerated cellulose fibers show smooth surface, circular cross-section, highest tensile strength, moderate dyeing/fibrillation, good spinning/weaving, guiding their commercial use.



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