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Chemical recycling of cellulosic textile waste by dry-jet wet spinning

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Introduction

Europe has an extensive textile waste problem - annually 7 –7.5 million tons of textile waste is generated. EU-wide separate collection for textiles comes into effect at the beginning of the year 2025. Today, only about 30 to 35 percent of the generated textile waste is collected separately, and less than 1 percent is recycled into new clothing. [1] The recycling of textiles will significantly decrease the environmental impacts associated with textile production.

By chemical recycling, the downsides of mechanical recycling such as inadequate fibre lengths or colour differences of recyclable feedstocks can be overcome. On the other hand, the processability of feedstock and the final properties of recycled fibres strongly depend on the intrinsic viscosity and molecular weight of textile waste.

The goal of these trials was to evaluate the recyclability of different cellulosic textile waste feedstock in terms of numbers of chemical recycling cycles and to determine the extent of changes in intrinsic viscosities of feedstocks and mechanical properties of produced fibres after subsequent cycles of chemical recycling.

Materials and methods

In this study, coloured lyocell, viscose, and white cotton-containing waste textile feedstocks were used as raw materials (Figure 1). All the raw materials were mechanically refined to decrease the particle size and improve dissolution.

Figure 1. Lyocell, viscose, and cotton feedstocks before the pre-treatments (from left to right).

Chemical pre-treatments were conducted for the removal of metals and dyes, and if needed also to lower the intrinsic viscosity of cellulosic materials (Figure 2). The resulting intrinsic viscosities for viscose and lyocell-containing feedstocks were 110 ml/g and 130 ml/g, respectively. Cotton was adjusted to three intrinsic viscosity levels of high, medium and low molecular weights (650 ml/g, 440 ml/g, 240 ml/g).

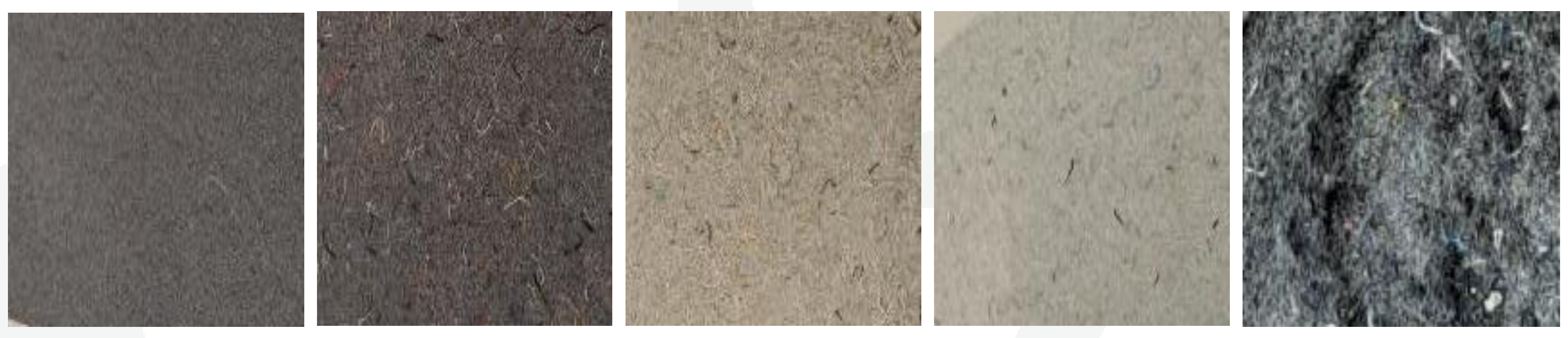

Figure 2. Lyocell-containing feedstock at different pretreatment stages and reject polyester fibres filtrated from the lyocell feedstock after the final stage of bleaching. From left to right mechanically refined lyocell, acid-treated lyocell, dithionite-treated lyocell, peroxide-treated lyocell, and polyester fibre reject from the lyocell-containing feedstock.

Spinning dopes were prepared by dissolving individual feedstocks into ionic liquid [EMIM][Oac] and using them as such or as a 50/50 mixture of viscose/cotton or lyocell/cotton. Fibres were spun by dry-jet wet-spinning in a water bath and freeze-dried for the next round of recycling (Figures 3-4).

Intrinsic viscosities and mechanical properties of regenerated fibres were measured after each subsequent cycle of recycling to determine the extent of changes caused by recycling.

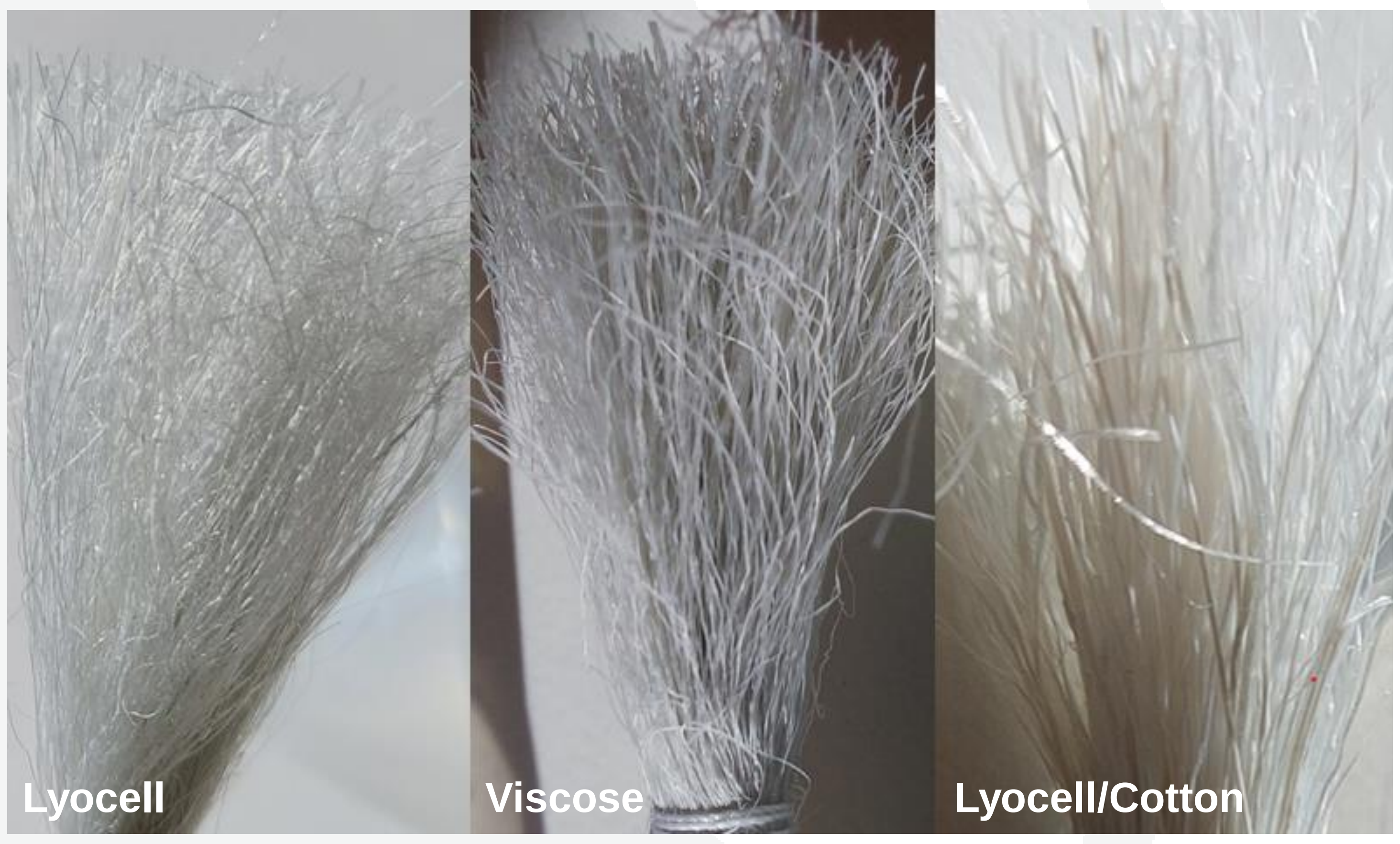

Figure 3. Recycled fibres spun by dry-jet-wet-spinning.

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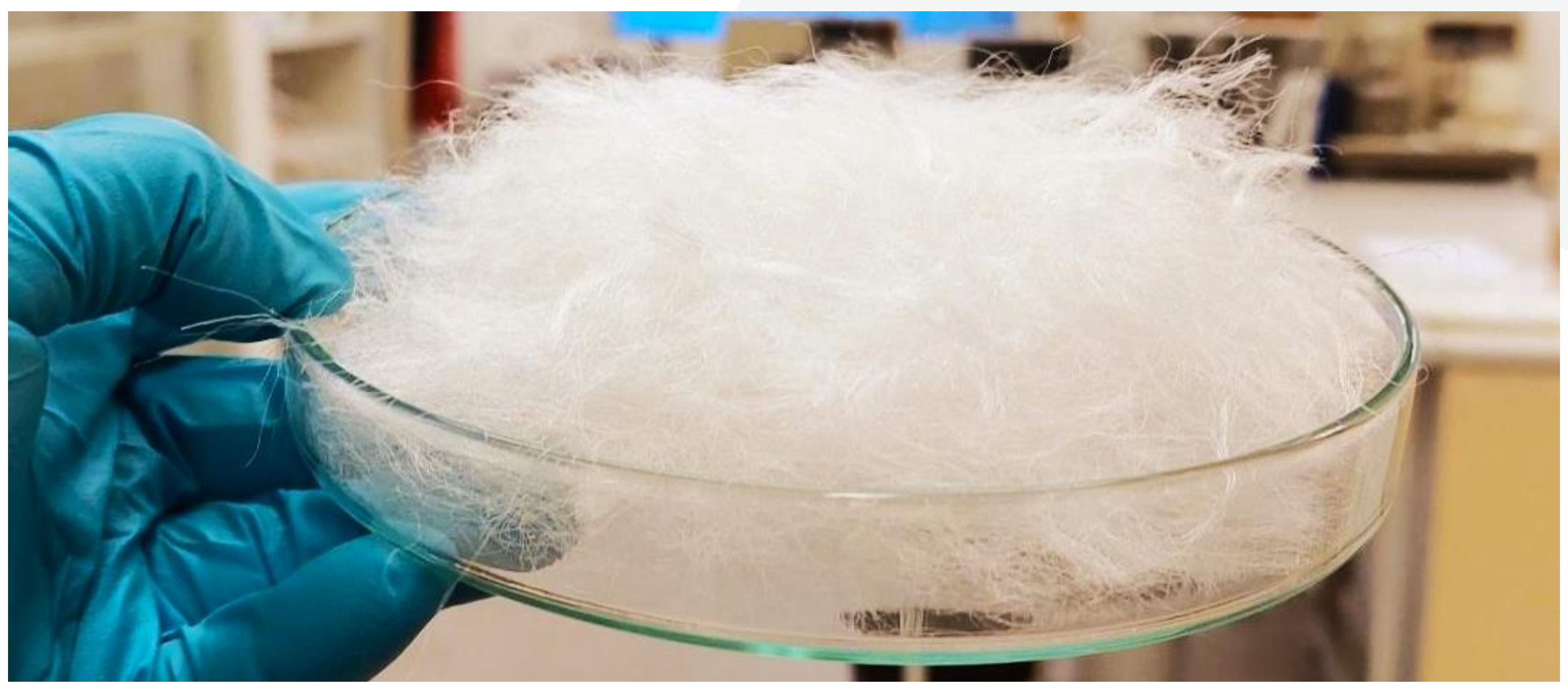

Figure 4. Twice recycled cotton fibres spun by dry-jet-wet-spinning.

Results and discussion

Each sample had excellent solubility into ionic liquid [EMIM][Oac], even though filtration of viscose- and lyocell-containing feedstocks resulted in relatively high amounts of undissolved reject, mainly consisting of polyester (Figure 2).

Recycled fibres from individual feedstocks exhibited significantly improved mechanical properties and higher linear densities compared to commercial reference fibres (table 1), medium molecular weight cotton having the highest values for both tenacity and elongation (figure 5). Mixing cotton with either viscose or lyocell resulted in significantly decreased spinnability and lower drawing ratio, leading to fibres with excessively high linear densities, and relatively low values for tenacity and elongation.

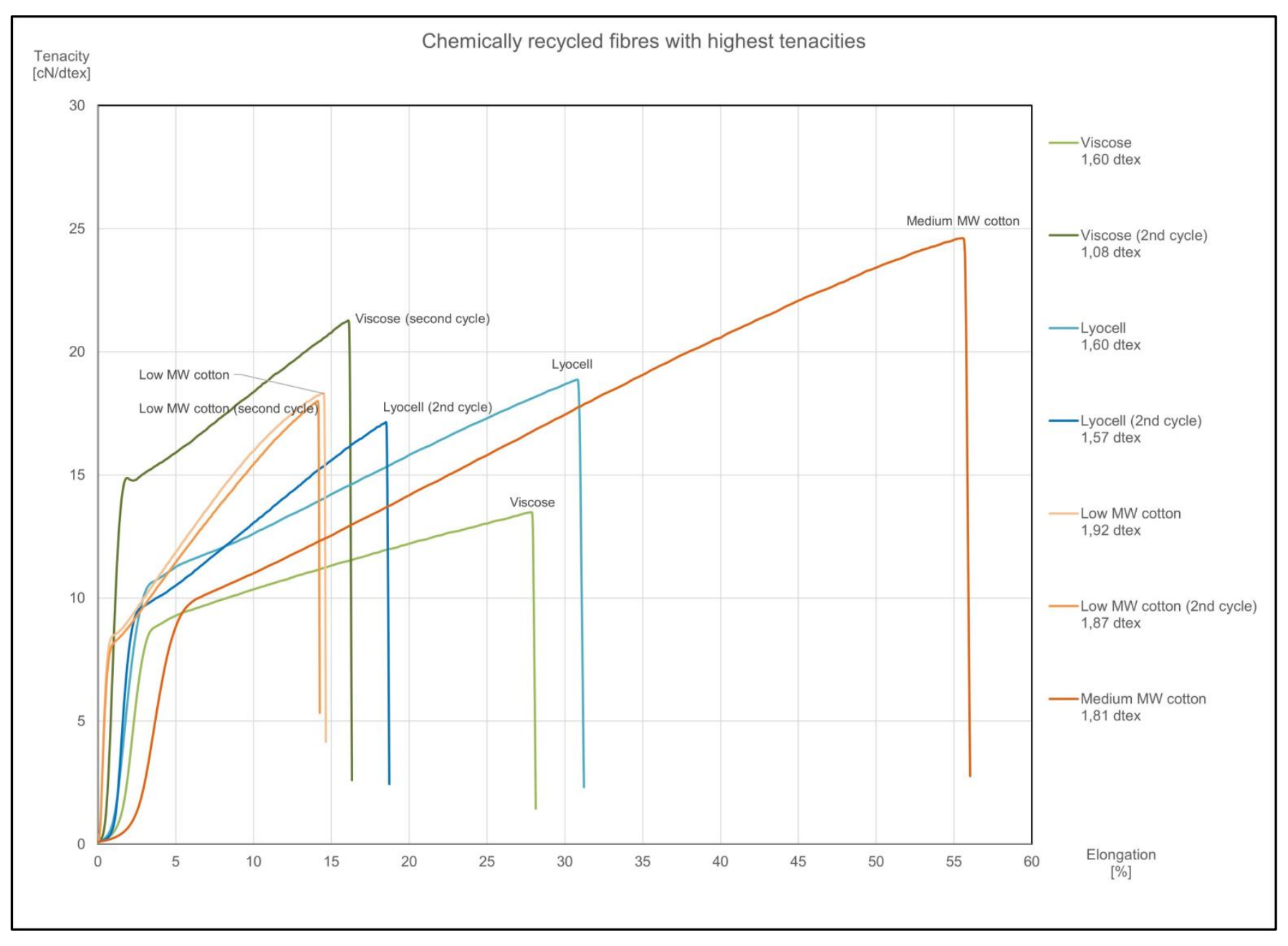

Figure 5. Mechanical properties of chemically recycled fibres.

Recycled sample	Elongation [%]	Tenacity [cN/dtex]	Linear density [dtex]	Minimum linear density [dtex]
Viscose	29.0 ± 7.7	12.4 ± 2.4	1.7 ± 0.3	1.6
Viscose (second cycle)	39.2 ± 7.6	12.6 ± 3.9	1.8 ± 0.5	1.1
Lyocell	38.2 ± 7.7	8.8 ± 3.8	2.1 ± 0.5	0.9
Lyocell (second cycle)	32.2 ± 7.2	12.1 ± 2.1	2.1 ± 0.6	1.2
Low MW cotton	46.6 ± 5.2	12.6 ± 4.1	3.1 ± 1.3	1.9
Low MW cotton (Second cycle)	41.2 ± 6.1	12.6 ± 2.7	2.5 ± 1.0	1.7
Medium MW cotton	48.0 ± 9.3	17.7 ± 3.3	2.1 ± 0.3	1.5
Viscose + high MW cotton	30.2 ± 10.5	4.9 ± 22.0	46.4 ± 46.0	10.9
Viscose + medium MW cotton	29.8 ± 14.4	1.2 ± 30.2	61.3 ± 22.9	34.1
Viscose + low MW cotton	25.4 ± 3.8	1.8 ± 1.2	62.5 ± 29.5	29.2
Lyocell + low MW cotton	30.1 ± 13.5	3.9 ± 6.3	49.6 ± 38.4	13.6
Commercial viscose reference	18.0 ± 1.6	2.7 ± 0.2	1.2 ± 0.1	1.0
Commercial lyocell reference	10.9 ± 2.9	3.5 ± 0.7	1.45 ± 0.3	1.2

Table 1. Average mechanical properties of chemically recycled fibres.

In general, subsequent cycles of recycling resulted in slightly increased tenacity and decreased elongation, with the exception of fibres spun from viscose–containing feedstock having both increased tenacity and elongation after the second cycle.

Losses in intrinsic viscosities by subsequent chemical recycling seemed to be mainly focused on the first cycle of dissolution and regeneration (Table 2). In the case of viscose and lyocell, this might be at least partly due to the removal of undissolved reject from filtration. Higher losses in intrinsic viscosity were observed with fibres spun from cotton-containing feedstocks though, which were nearly free of impurities (Figure 6). However, the tenacities were not decreased with any of the samples despite the viscosity loss.

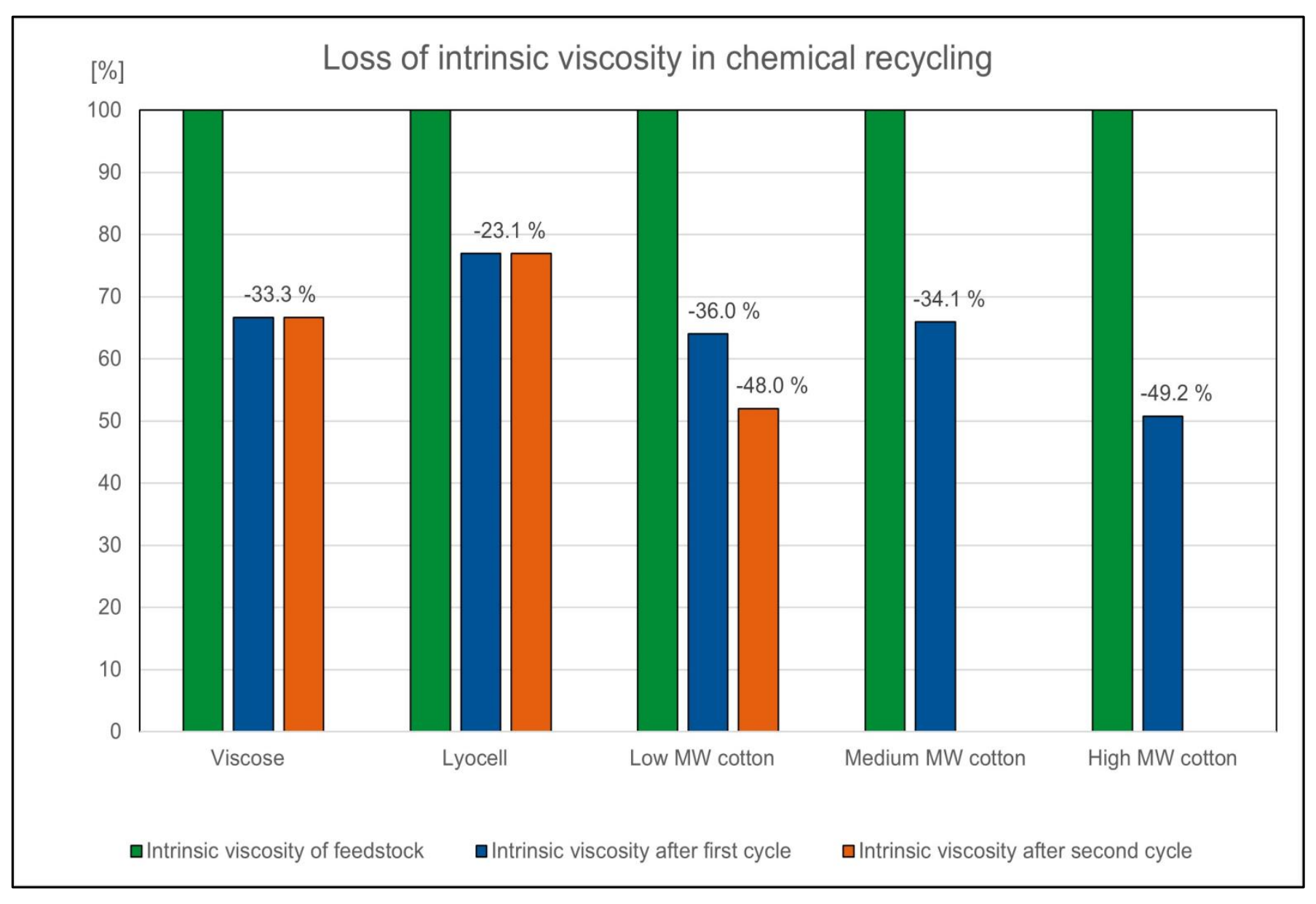

Figure 6. Decrease of intrinsic viscosity in chemical recycling cycles.

Recycled sample	Intrinsic viscosity of feedstock [ml/g]	Intrinsic viscosity after chemical recycling [ml/g]	Decrease [%]
Viscose	120	80	33.3
Viscose (second cycle)	80	80	0.0
Lyocell	130	100	23.1
Lyocell (second cycle)	100	100	0.0
Low MW cotton	250	160	36.0
Low MW cotton (second cycle)	160	130	18.8
Medium MW cotton	440	290	34.1
High MW cotton	650	330	49.2

Table 2. Decrease of intrinsic viscosity in chemical recycling cycles.

Conclusion

- **Cellulosic textile waste feedstocks can be recycled for several cycles, even with feedstocks of low intrinsic viscosity such as viscose rayon, or lyocell**
- **Decrease in intrinsic viscosities decreased with subsequent cycles of recycling**

References:

- [1] The European Apparel and Textile Confederation. Rehubs 2022: Circulating textile waste into value. Retrieved from https://euratex.eu/139/rehubs-2022-circulating-textile-waste-into-value/

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