

Interactions of fibres and textile dyes

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Contextualization

The Textile Sector is...



3rd highest source of pressure on water and land use in the EU



5th highest use of raw materials and greenhouse gas emissions

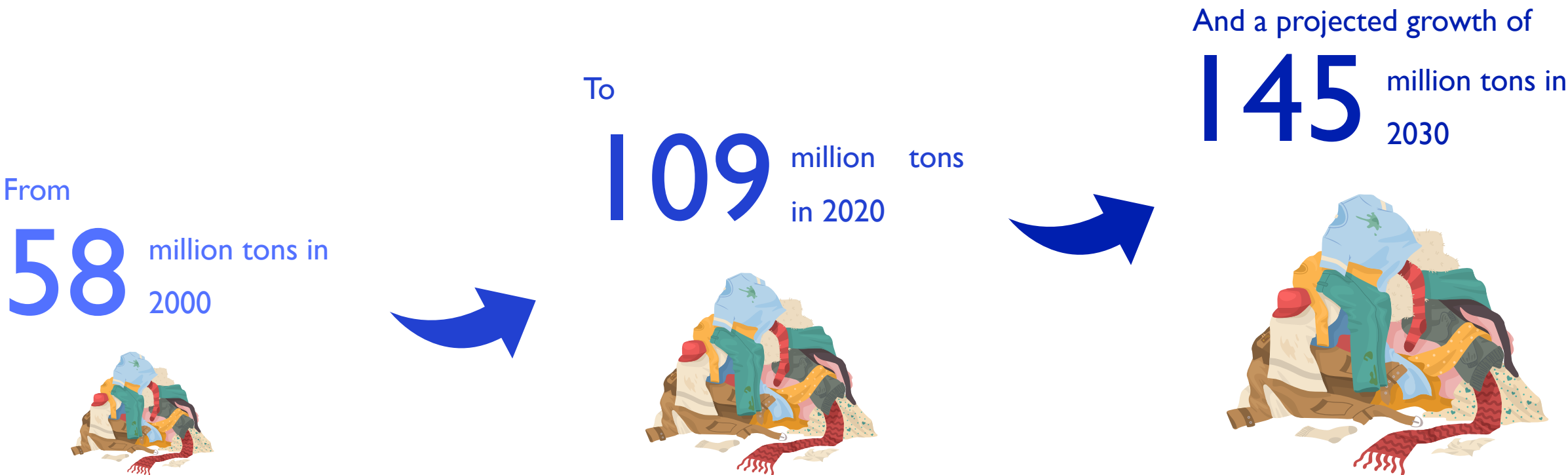


3rd largest employer worldwide, after food and housing

On the European Union alone...

- **4–9%** of textiles are destroyed without being used.
- In 2020, each person used **16 kg of textiles**.
- Only **4.4 kg** per person were reused/recycled; **11.6 kg** went to household waste.
- **60–70%** of textiles are made from plastic, mainly polyester.
- Per average person, textile consumption required **9m³ of water**, **400 m² of land**, **391 kg of raw materials**, and caused a **carbon footprint of about 270 kg**.
- **Less than 1%** of textiles worldwide are recycled into new products (data from 2017).
- EU exports of used textiles **tripled** from 2000 (550,000 tonnes) to 2019 (1.7 million tonnes).

Textile Industry Overview



10-15%
of all dyes used
remain unfixed

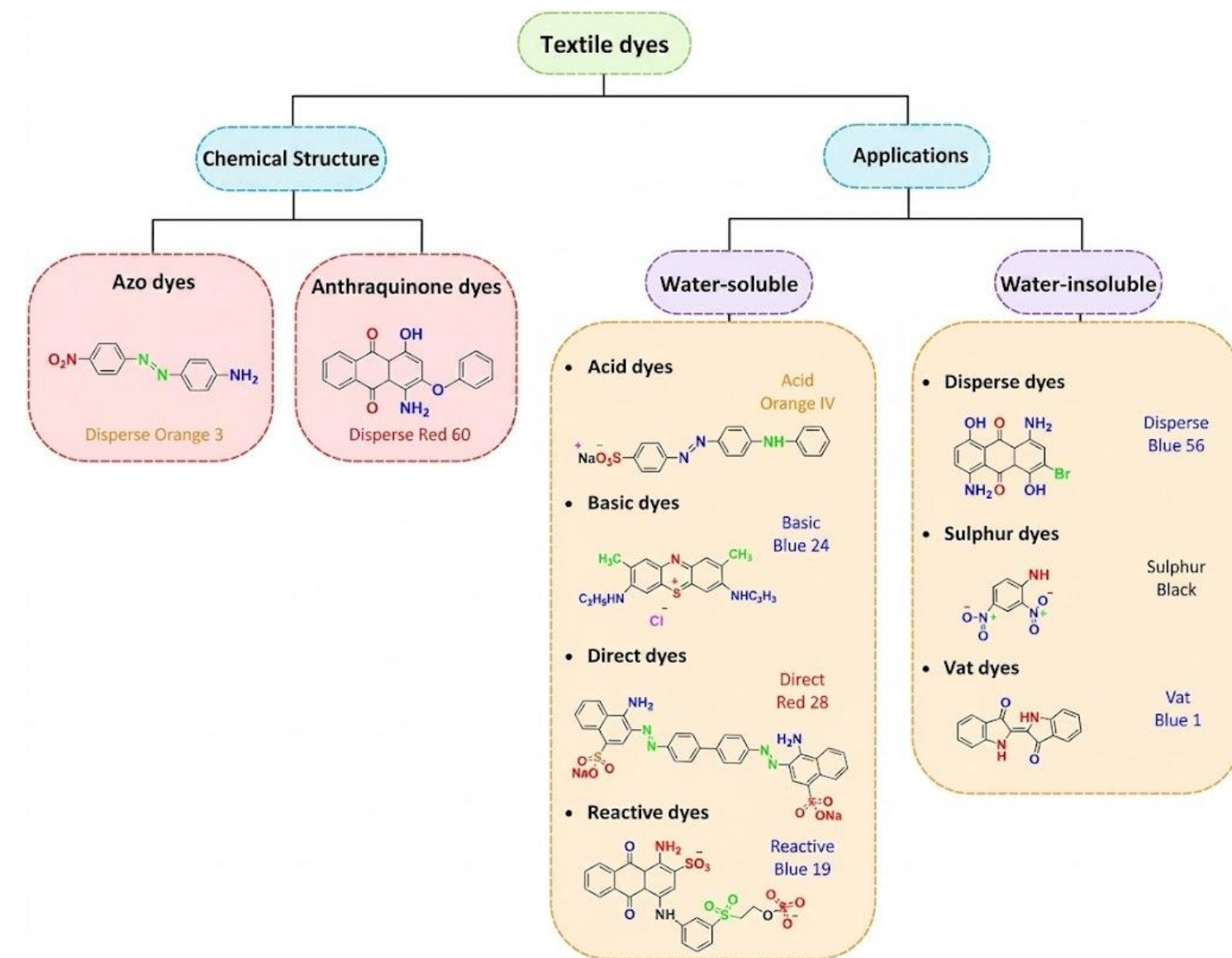
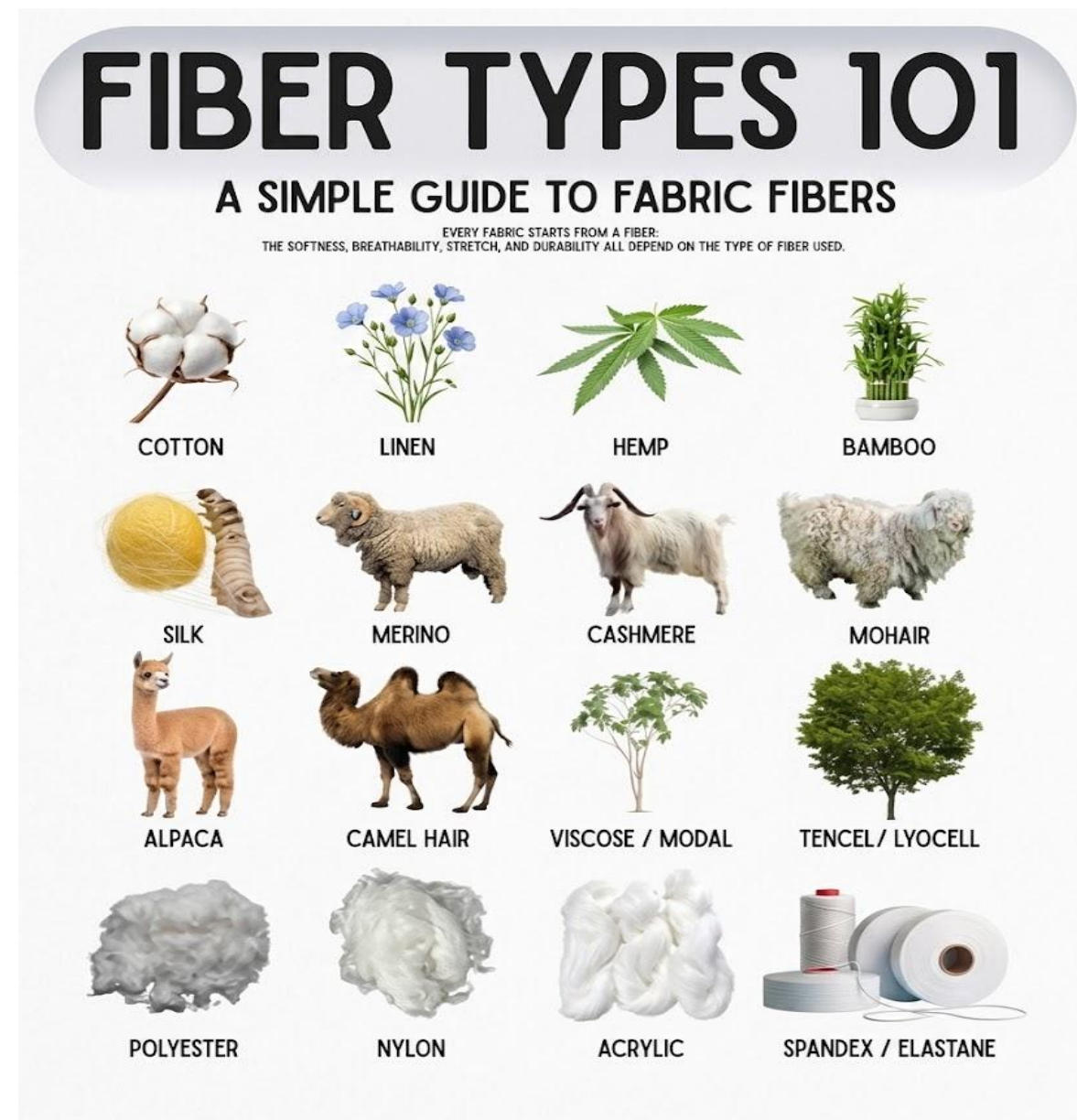
54%
of all effluent containing dyes
originate in textile industries

≈280,000
tons/yr of textile dyes
are released to the
environment



Contextualization

Classification of Textile Fibers and Dyes



❑ The global market relies on **dozens of commercial fiber types** grouped into broad **natural** and **synthetic** categories

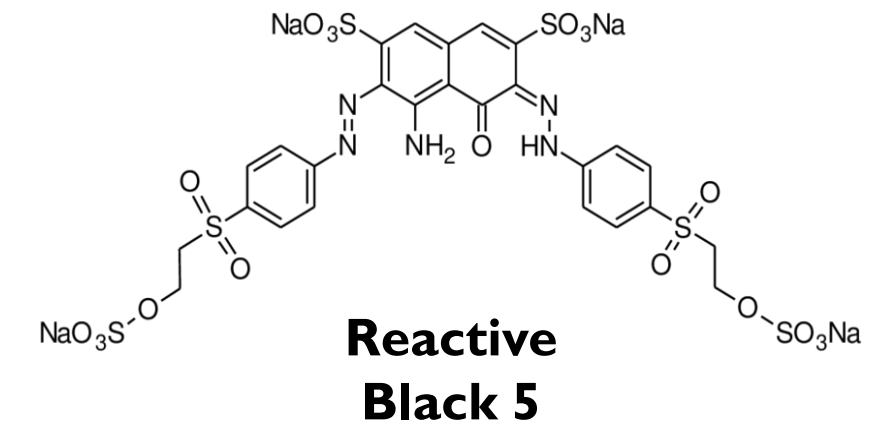
❑ **Polyester** alone accounts for about **59%** of total global fiber production, with **cotton** making up **roughly 19%**

❑ There **are more than 10,000 different types of synthetic textile dyes** available for commercial and industrial used, with over **40,000 distinct trade names**

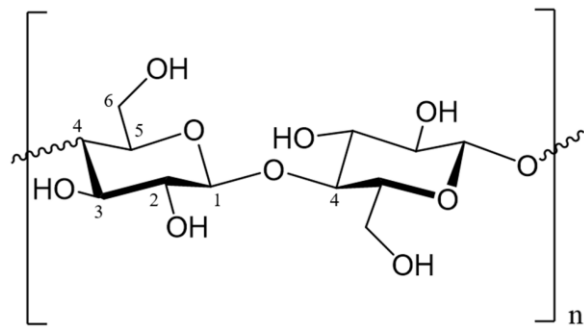
❑ **Azo dyes** represent **60% to 70% of all organic dyes** produced globally

Contextualization

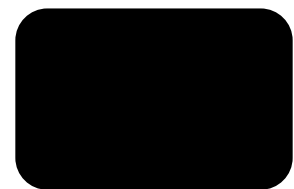
Why can't we just dye every textile with the same dye?



Cotton (Cellulose)

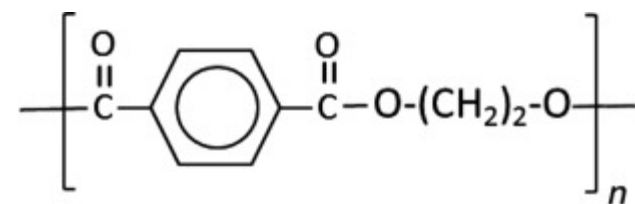


Outcome with this dye



Strong covalent dye-fiber bonding

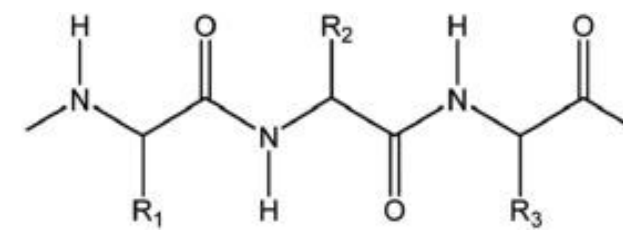
Polyester (PET)



Outcome with this dye

Very weak interaction
(no reactive group and very hydrophilic dye + very hydrophobic fiber)

Wool (Protein)

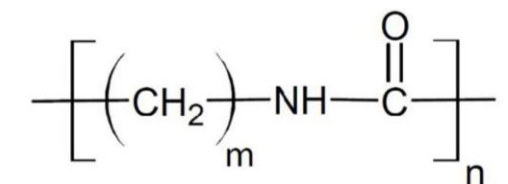


Outcome with this dye

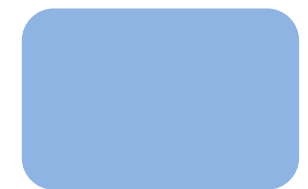


Good ionic and hydrogen bonding

Nylon (Polyamide)



Outcome with this dye



Moderate ionic and hydrogen bonding

Different fibers have different chemical composition, presenting **different dye affinity and dyeing performance!**

Classes of textile dyes and their application

DYE CLASS	DESCRIPTION	TYPE OF FIBER	DYE FIXATION (%)	DYE AND FIBER INTERACTION	TYPICAL POLLUTANTS
ACID	Anionic, Water soluble	Wool, Silk, Nylon	80-95%	Dye $-$ ----- $+$ Fiber H-bonding, ionic bonding, van der Waals forces	Colour; unfixed dyes; organic acids
BASIC	Cationic, Water soluble	Acrylic, Some polyesters	95-100%	Dye $+$ ----- $-$ Fiber H-bonding, ionic bonding	N/A*
DIRECT	Anionic, Water soluble	Cotton, Rayon, and Other cellulosic	70-95%	Dye $\text{H}-\text{H} \cdots \text{O}$ Fiber $\text{O} \cdots \text{H}$ H-bonding, van der Waals forces	Colour; unfixed dye; salts; cationic fixing agents; surfactants; others
DISPERSE	Non-ionic, Water insoluble	Polyester, Acetate, and other synthetics	90-100%	Van der Waals, H-bonding	Colour; organic acids; levelling agents; phosphates; lubricants; dispersants; diluents
REACTIVE	Anionic, Water soluble	Cotton, Other cellulosic, Wool	50-90%	Dye ---X + HO---Fiber Dye ---O---Fiber Covalent bonding, H-bonding	Colour; salt; alkali; unfixed dye; surfactants; defoamer; diluents; finishing agents
SULPHUR	Non-ionic, Water insoluble	Cotton, Other cellulosic	60-90%	Dye $\text{---H} \cdots \text{O}$ Fiber H-bonding, van der Waals forces	Colour; alkali; oxidizing agents, reducing agents, unfixed dyes
VAT	Non-ionic, Water insoluble	Cotton, Other cellulosic	80-95%	Dye $\text{---H} \cdots \text{O}$ Fiber H-bonding, van der Waals forces	Colour; alkali; oxidizing agents, reducing agents, unfixed dyes

$-$ Negative charge

$+$ Positive charge

$\text{H} \cdots \text{O}$ Hydrogen bond

X Reactive group (leaving group)

$\bullet$ Dye molecule

$\longrightarrow$ Diffusion into fiber

*N/A: Not applicable

- Dye–fiber affinity:

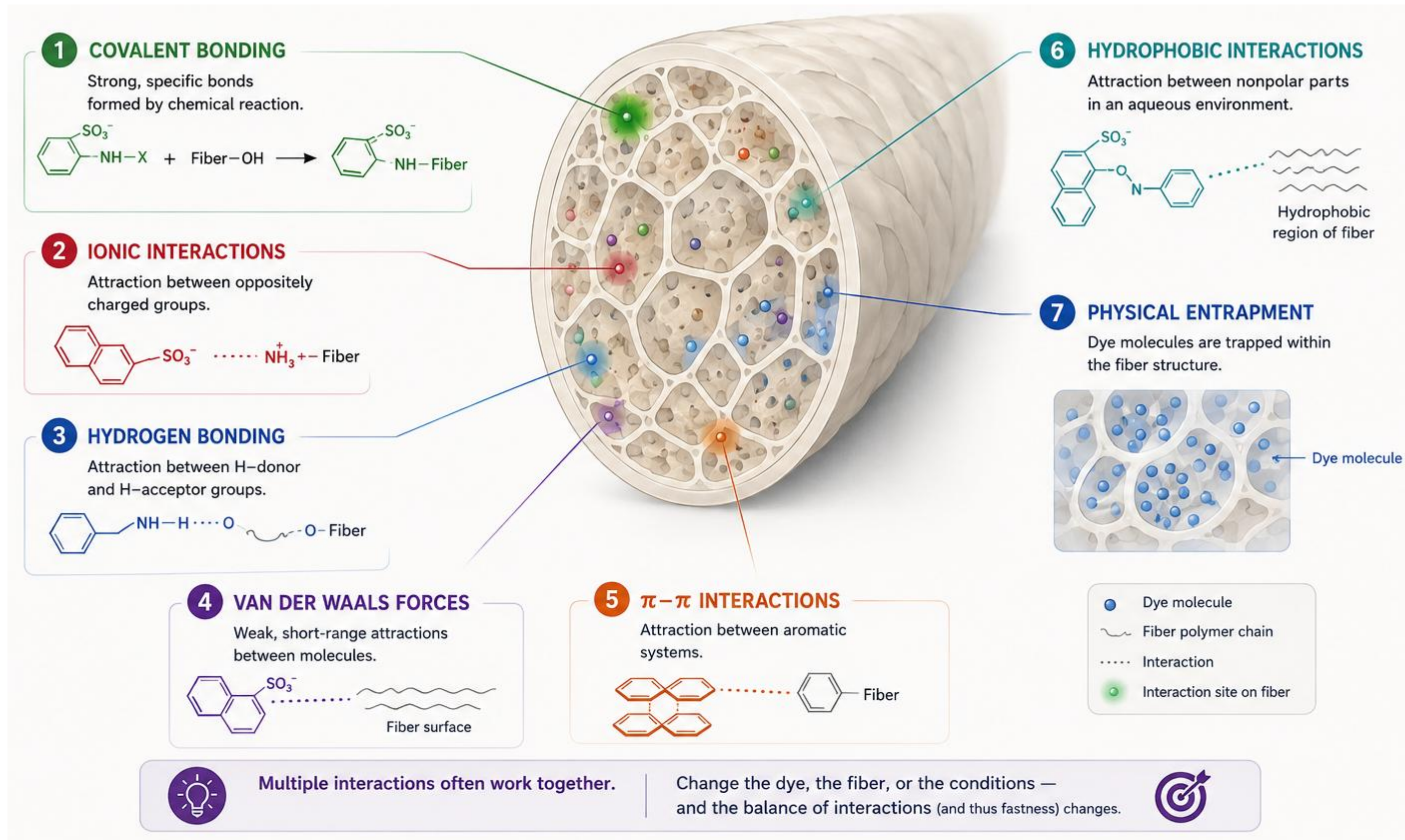
Each type of dye has a greater affinity for specific types of fibers.
- Intermolecular interactions:

Dyes interact with fibers through different intermolecular mechanisms.
- Main pollutants:

Unbound dyes remaining in the dye bath.

Auxiliary agents that do not bond to the fibers.

The “interaction toolbox”



Industrial Case Study – Reactive Cotton Dyeing Process

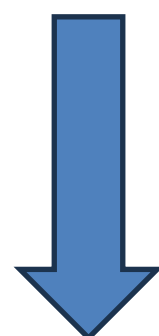
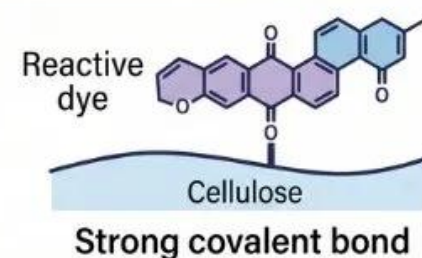
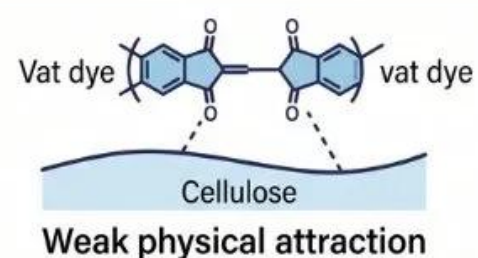
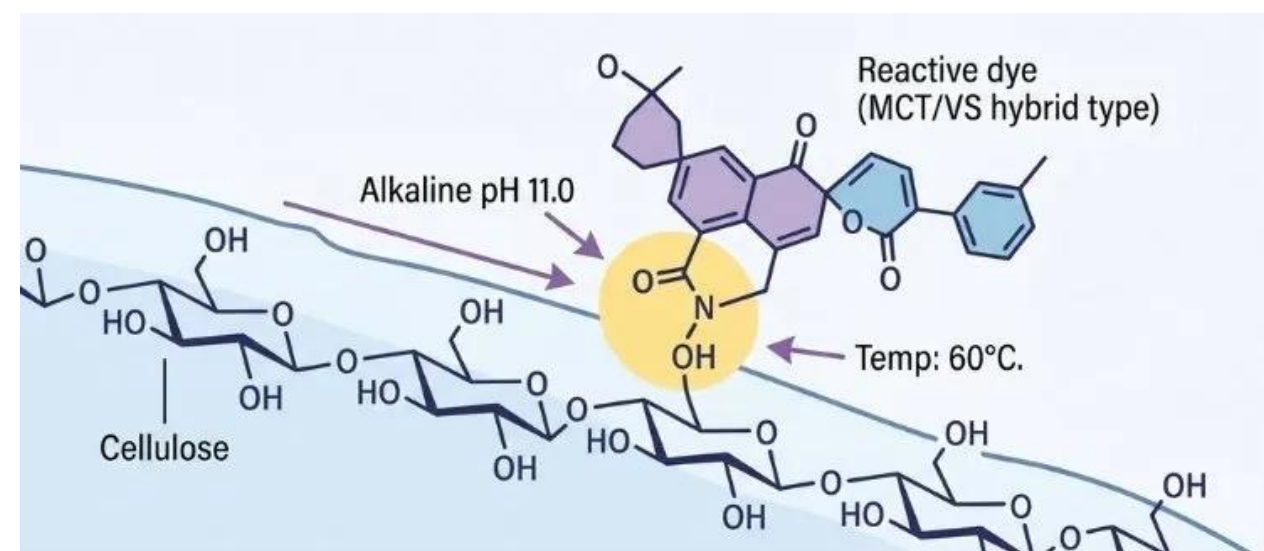


A	1g/L	400g
B	1g/L	400g
C	1.0%	255g
D	7.0%	1.785kg
E	100g/L	40kg
F	10g/L	4kg
G	2g/L	800g

Contextualization

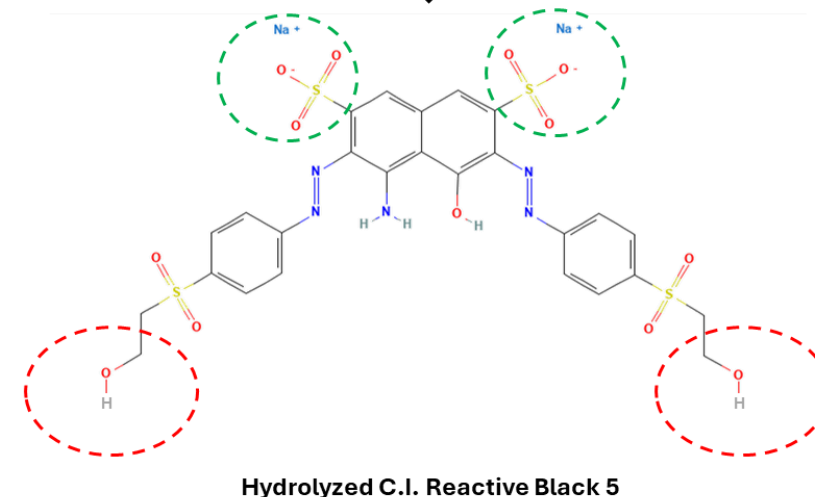
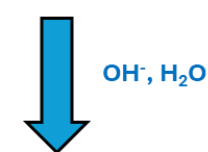
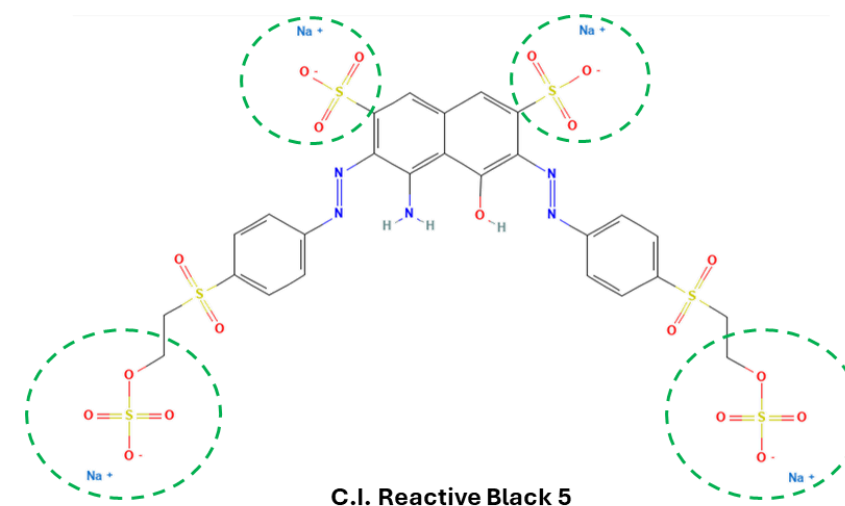
Industrial Case Study – Reactive Cotton Dyeing Process

Covalent dye-fiber binding



PRIMARY REACTION WE SEEK!

Dye hydrolysis in the dye bath



UNWANTED SECONDARY REACTION!



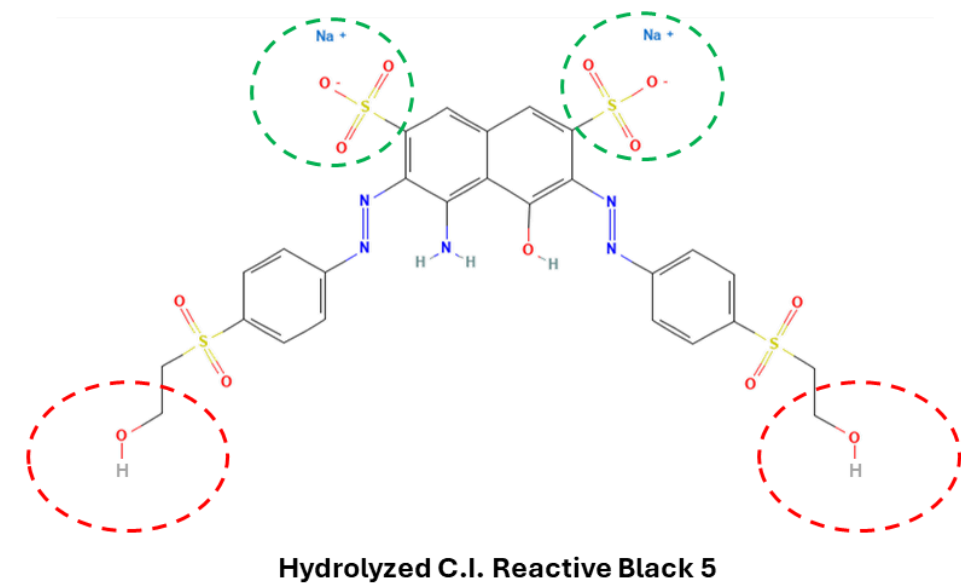
Effluents containing hydrolyzed reactive dyes



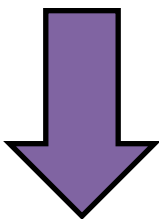
- ☐ Colored effluents difficult to treat
- ☐ High organic and inorganic content
- ☐ Complex and costly treatment
- ☐ Focus on dye degradation

Contextualization

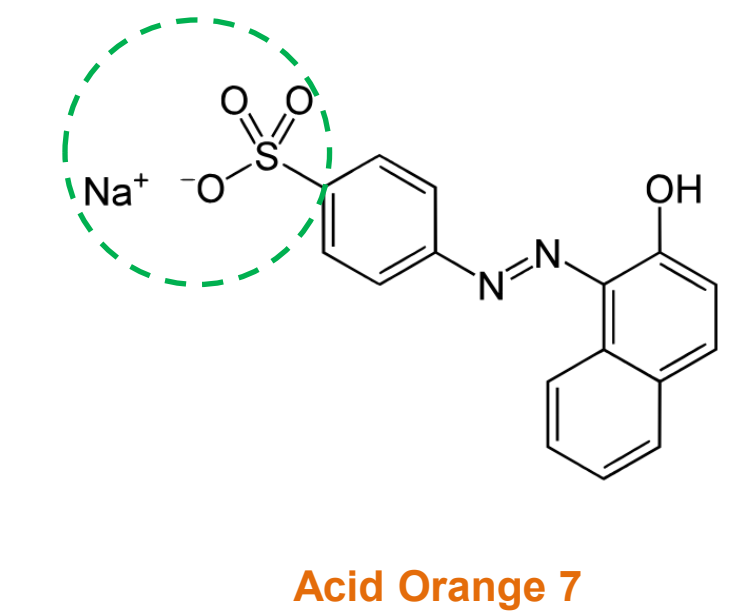
Industrial Case Study – Reactive Cotton Dyeing Process



Hydrolysed reactive dyes have the same binding sites as **acid dyes**

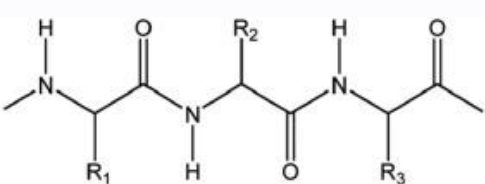


Can dye the **same type of fibers as acid dyes!**

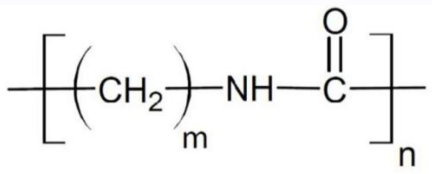


Fibers dyed using Acid Dyes

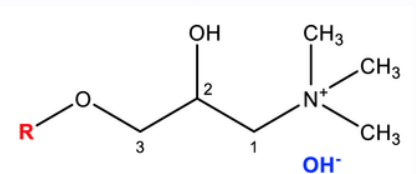
Wool (Protein)



Nylon (Polyamide)



Cationized cotton



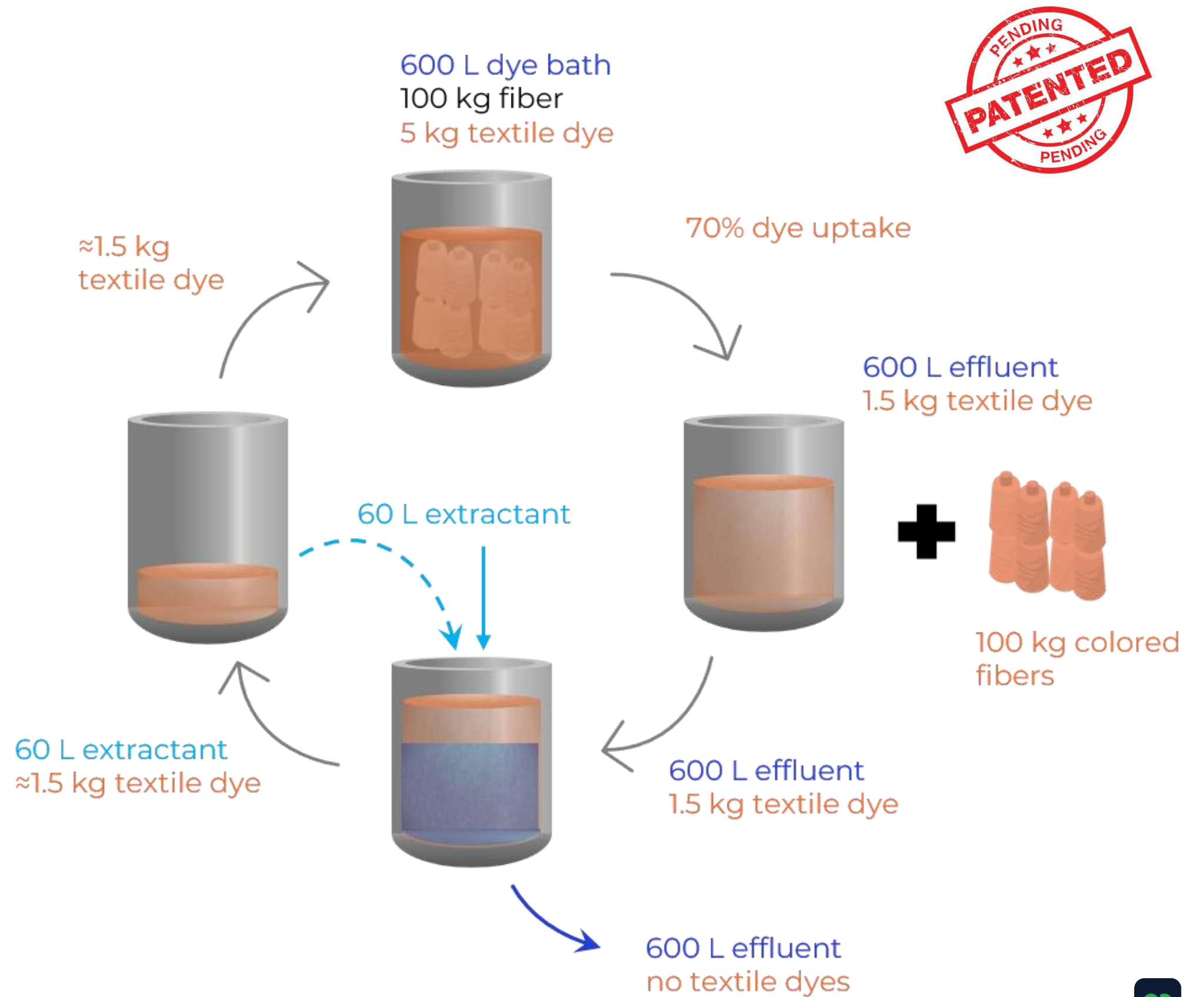
Our Solution

DyeLoop technology



A circular and integrative process that **uses low-cost and eco-friendly extractants to concentrate and recover dyes and water** from textile effluents.

The **complete recovery of dyes and extractants**, which were efficiently reused in successive dyeing and extraction cycles, reducing organic load and overall cost and complexity of wastewater treatment.



DyeLoop – Circular technologies for textile dyeing



The widespread attention from society, commercial interest from stakeholders, and recognition from innovators and researchers, coupled with the lack of commercial circular technologies for textile dyeing, drive us to advance the DyeLoop technology.

The merit of the project was recognized and the **DyeLoop Project—Circular technologies for textile dyeing** was granted with 1.4M EUR.

The project tasks are scheduled over a 36-month period, **from Jan'25 to Dec'27**.



1. To optimize, mature, and scale-up the technology, culminating in a prototype to validate its efficiency in a relevant environment.
2. To explore new areas of research to enhance the circularity and sustainability of dyeing processes using natural dyes and biotechnological strategies.
3. To obtain results for business consolidation of the DyeLoop-related roadmap within a circular economy framework.

Objectives

DyeLoop – Circular technologies for textile dyeing

Achievements

Several performance indicators outline the expected results and achievements, reflecting its anticipated **contributions to innovation, technology adoption, and operational improvements.**



70-90%



Dye recovery & reuse efficiency from real textile effluents.

50%



Cost reduction in textile dyeing processes compared to conventional approaches.

3+ Patents filed for the DyeLoop technology and related innovations.

2+ Trademarks registered.

2+ Validation tests of the DyeLoop prototype in relevant environments.

7+ National and 1 International Partnerships with Industrial Stakeholders.

1 IP, business and market valorization roadmap.

... Establishment of agreements for testing and validation of prototype with industrial partners.

Project

DyeLoop – Circular technologies for textile dyeing

Impact

The DyeLoop project is positioned to **deliver long-term impacts**, aligning with its core objectives, fostering the transformation in the textile dyeing sector, while also advancing the **transition toward a bioeconomy in the textile sector**.



Environmental



- **40-60% reduction in water usage & 70-90% dye recovery and reuse**, significantly lowering raw material demand
- **Wastewater contamination** could be **reduced by 50-70%**, alleviating environmental burdens.
- If widely adopted across Portugal, DyeLoop could save millions of liters of water annually and reduce CO₂ emissions by thousands of metric tons.

Economic



- competitive advantages through **cost savings, resource efficiency**, and **compliance** with stringent **environmental regulations**
- These innovations foster job creation, open new business opportunities, and **aligning with EU strategies** for re-industrialization, circularity, and decarbonization.
- Enhanced resource-efficiency could lead to **50% cost savings**, enabling companies to reinvest in more sustainable practices and innovative value-added technologies.

DyeLoop – Circular technologies for textile dyeing



- ❑ **DyeLoop™ achieved >98% dye extraction efficiency** from cotton dyeing effluents.
- ❑ **Phase separation were completed in <10 min**
- ❑ **Alcohol recovery of 99% in <30 min.**
- ❑ **Salt recovery exceeded 90%** through selective precipitation.
- ❑ **Process optimisation reduced dye-contaminated water volumes by ~67%.**

The integrated DyeLoop™ process enables circular resource recovery with ~99% alcohol recovery, and >90% salt and water recovery.

Experimental

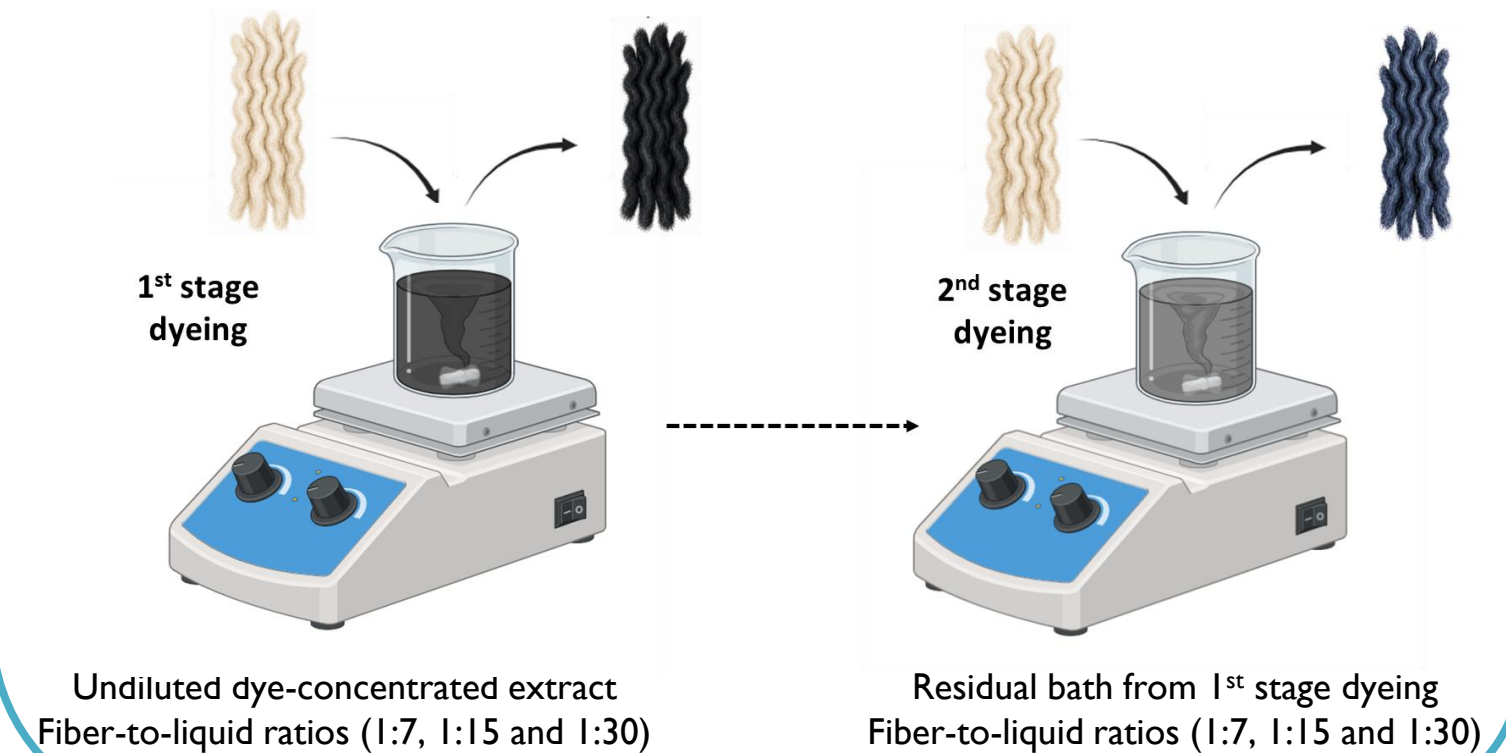
Dye recovery using DyeLoop™ technology



Recovered dye adsorption studies

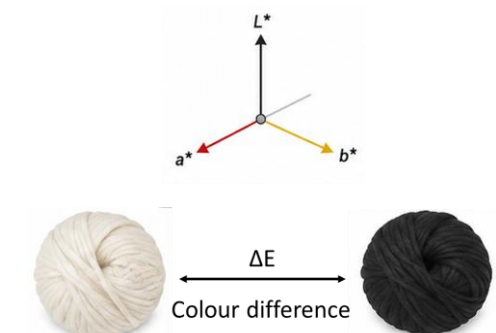
- Dye extract diluted and acidified to pH $\approx$ 4
- Assessed at 60°C and 90°C using 10 mL of diluted dye solution

Wool dyeing process



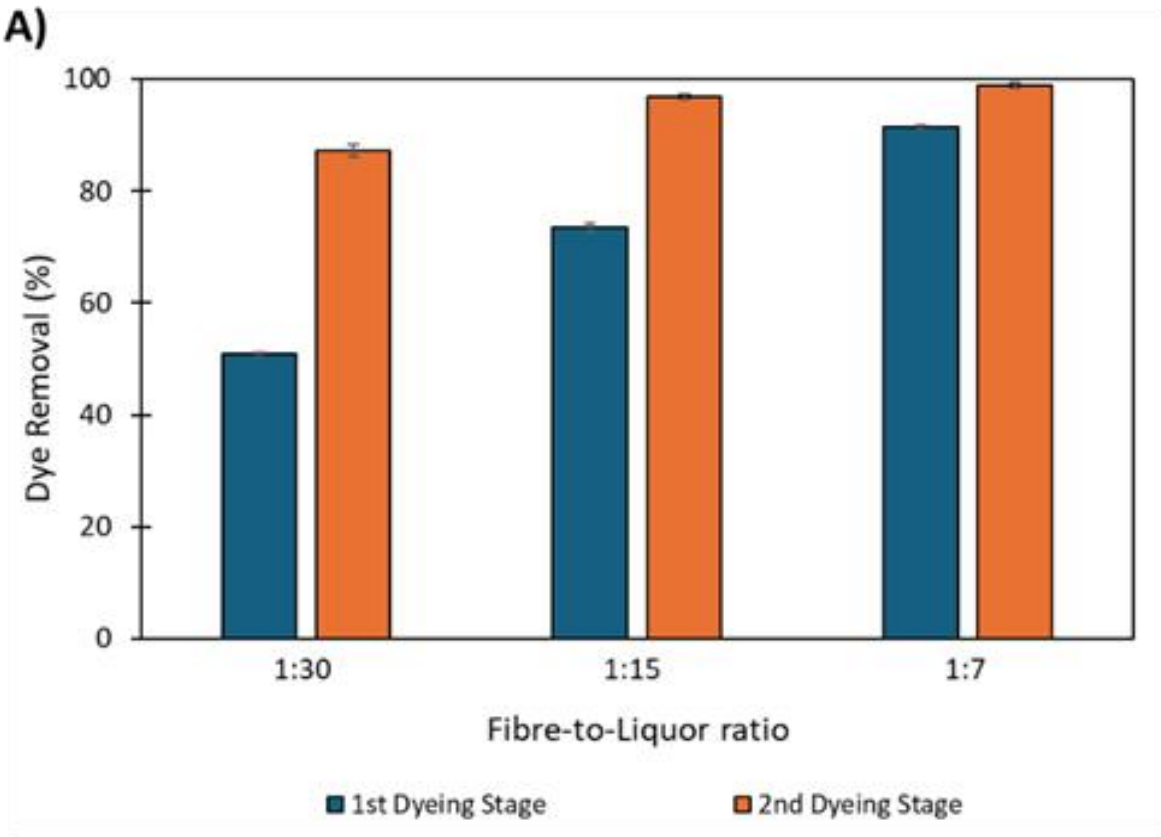
Evaluation of dyeing performance

- **Washing fastness:** ΔE comparison before/after washing comparison and dye bleeding assessment
- **Colour measurements**



Results

Dyeing Experiments



- Higher wool dosage increased **dye removal up to ~90%**
- Strong dyeing in both stages (high ΔE)**, with greater 2nd stage variability due to residual dye concentration
- Distinct shades were generated from a single dye bath while achieving **near-complete dye removal and reuse**



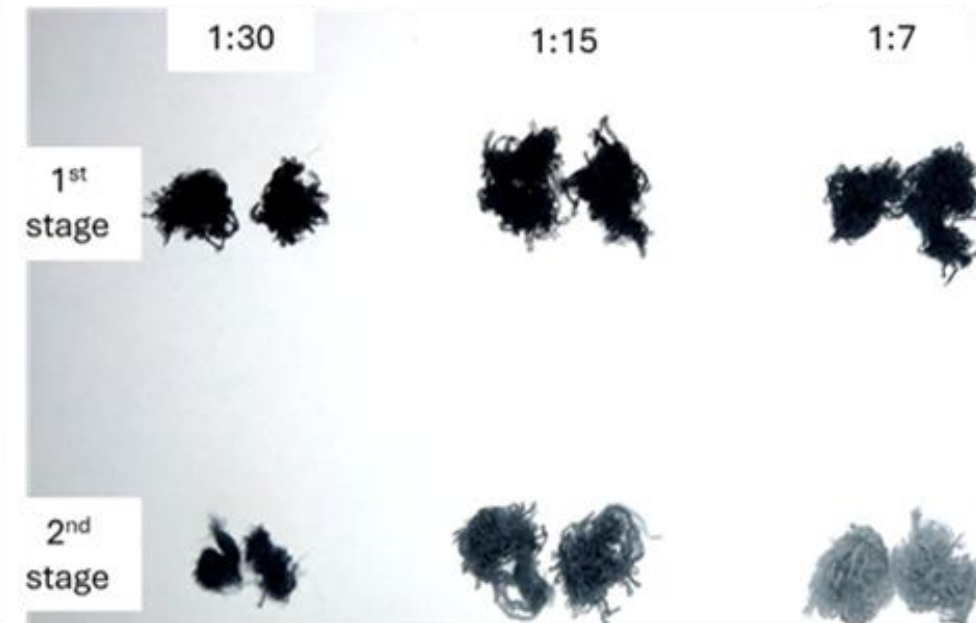
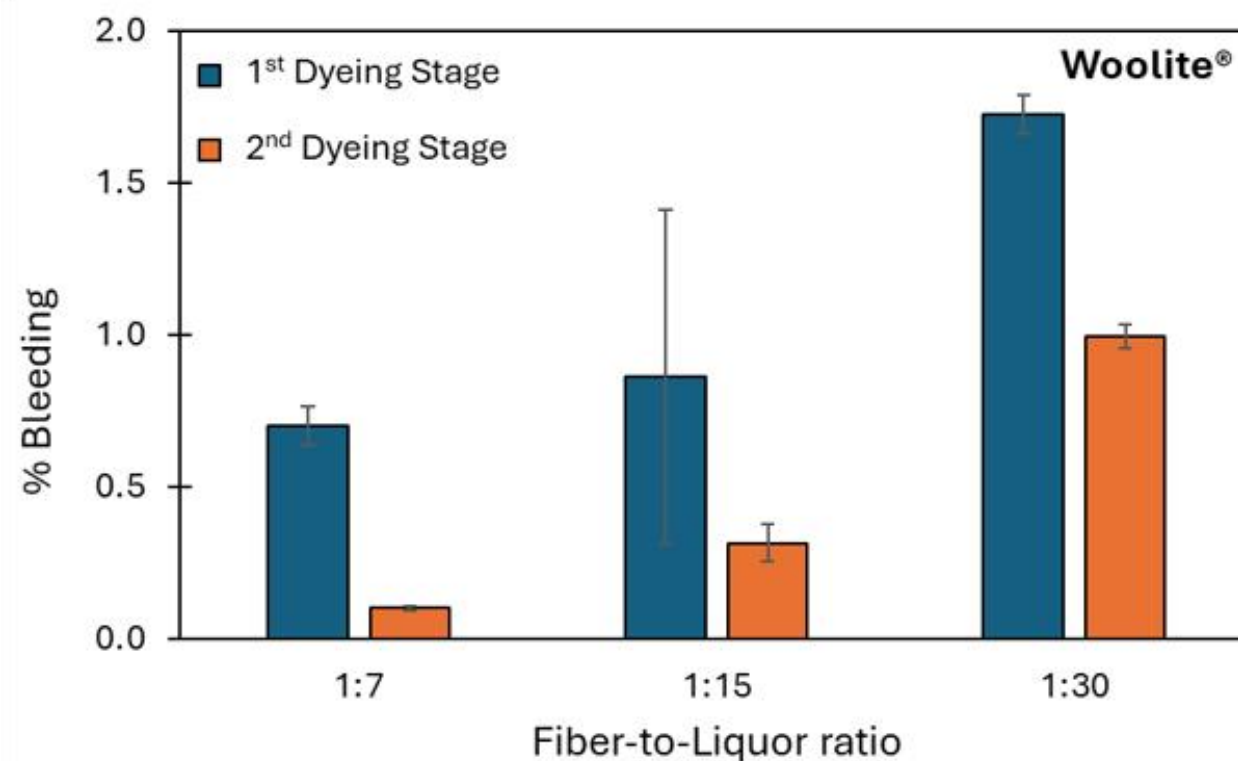
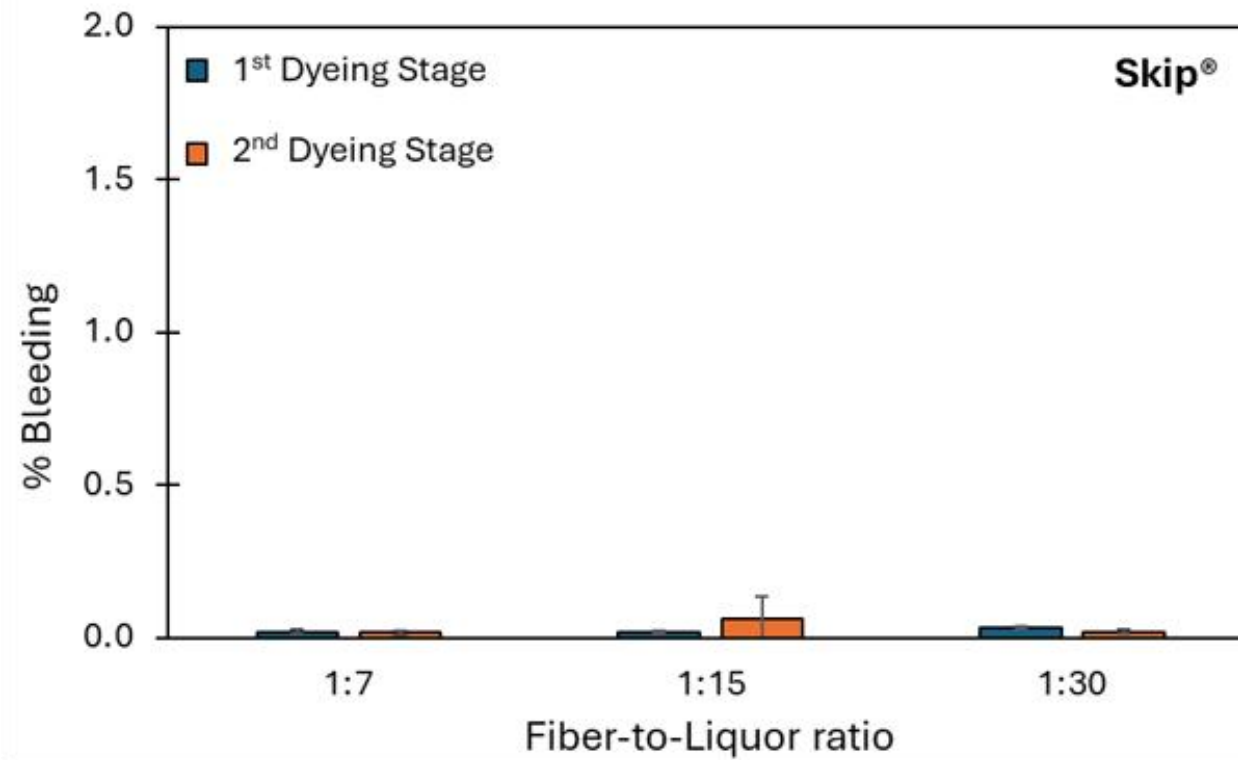
$$\Delta E = 72.04-75.06$$

$$\Delta E = 44.05-71.87$$

Two-stage dyeing combined efficient dye removal with the production of commercially relevant dyed fibres

Results

Washing fastness experiments



- ☐ Colour was preserved after washing
- ☐ Better colour fixation under near-neutral washing conditions
- ☐ Alkaline washing weakened dye-wool interactions
- ☐ **Dye bleeding < 2%** in both cases
- ☐ Good fixation of the recovered dye in wool fibers

Results

Economic Potential of DyeLoop™ Technology at Industrial Scale

Metric	Value
Wastewater treated	20,000 m³/month
Scale-up factor (vs prototype)	27 ×
Industrial CAPEX	€2.66–3.32 M
Net annual operating cost	€522,920/year
Additional annual revenue	€1.06 M/year
Net annual profit	€533,080/year
Payback period	6.24 years
ROI	16%
Value creation (15 years)	€4.66 M

Main Conclusion:

DyeLoop™ transforms wastewater treatment from a **€567k/year cost** into a circular process generating ~€533k/year profit, with a payback period of only 6.2 years.



Wool fabrics dyed
recovered dyes with
DyeLoop™ technology

Results

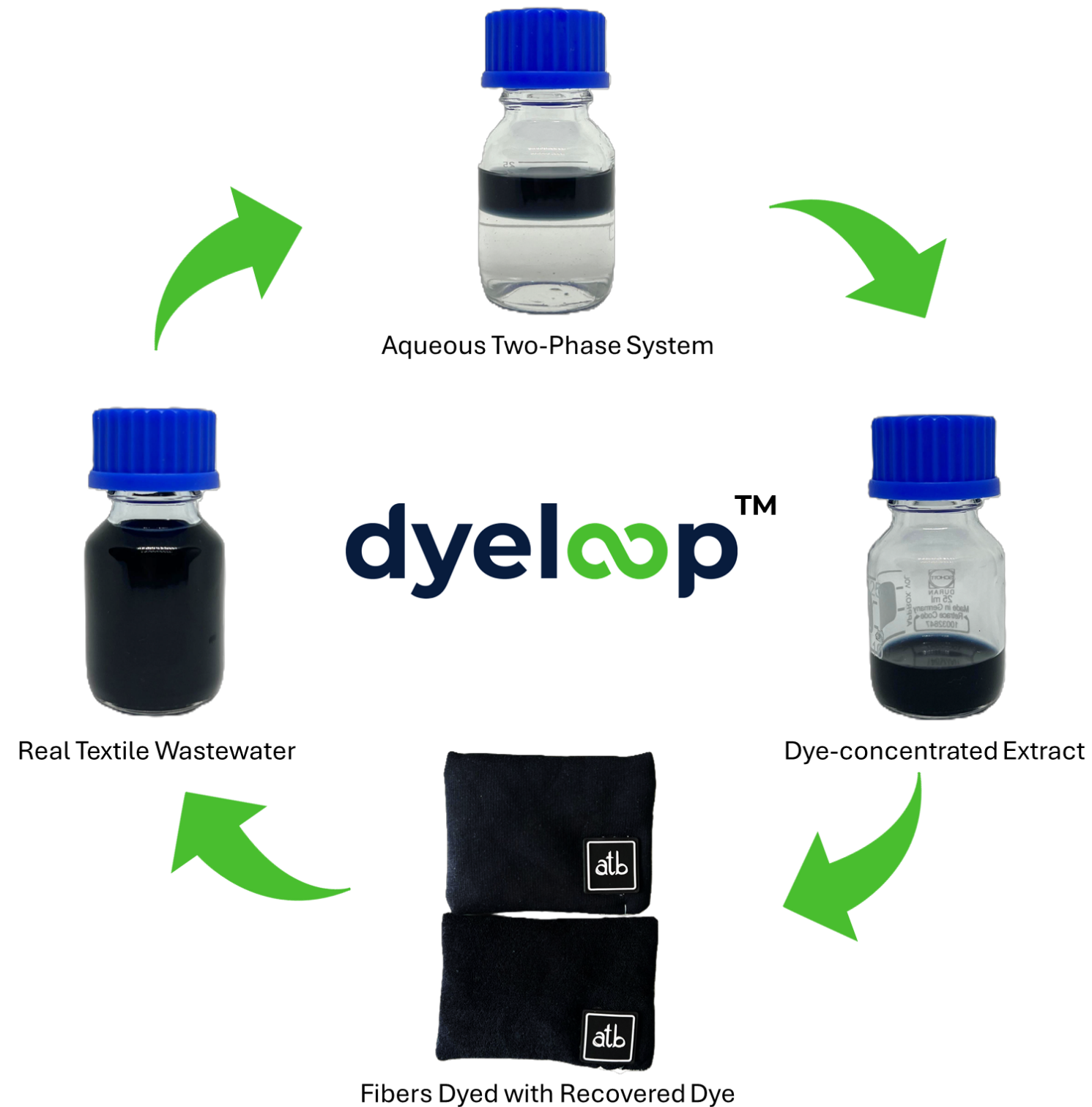
Environmental Benefits of DyeLoop™ Technology

Indicator	Conventional Process	DyeLoop™
Water consumption	100–150 L/kg textile	90% reduction
Wastewater discharge	100–150 L/kg textile	10–15 L/kg residual discharge
Salt consumption	0.6–1.0 kg/kg textile	>90% recovered
Solvent consumption	-	~99% recovered
COD load	High	30–70% reduction
Sludge generation	High	30–60% reduction
Energy demand	Baseline	10–40% reduction

Circular Economy Benefits

- ❑ Near-zero water discharge (90% water reuse)
- ❑ Recovery and reuse of water, salt, alcohol and dyes (>90% salt recovery, ~99% alcohol recovery and significant reductions in wastewater discharge)
- ❑ Lower CO₂ emissions from water and chemical production
- ❑ Reduced environmental burden on WWTPs

DyeLoop™ Technology



Problem: Water pollution with hydrolysed reactive dyes

Understand the structure of hydrolysed reactive dyes

Similar structure as acid dyes (similar type of interactions), so they can color the same types of fibers

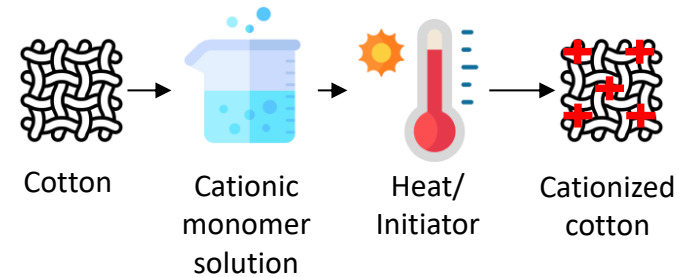
Limitation: We need to concentrate these dyes to facilitate industrial application

We developed the **DyeLoop™ technology** and validated its industrial potential

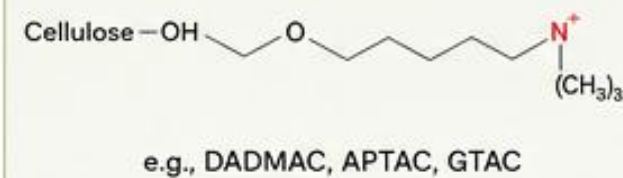
Industrial implementation and improved circularity and sustainability for textile industries

Strategy #1: Fiber cationization

1. Chemical grafting

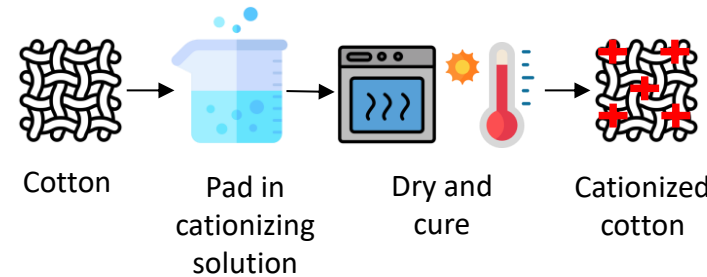


Typical chemistry (example)

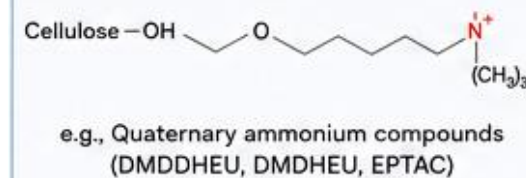


Cationic monomers are grafted onto cellulose backbone through covalent bond (radical or other mechanism)

2. Pad-Dry-Cure Method

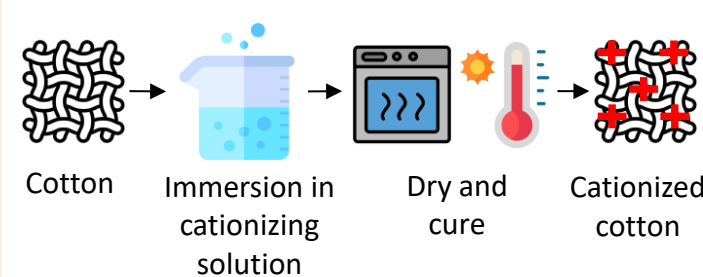


Typical chemistry (example)

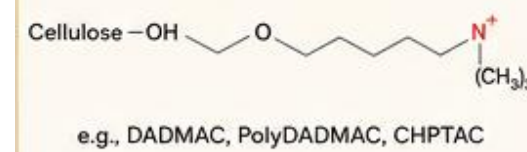


Cationizing agent is applied by padding, and the fabric is then dried and cured to react with cellulose and form cationic sites.

3. Exhaustion method

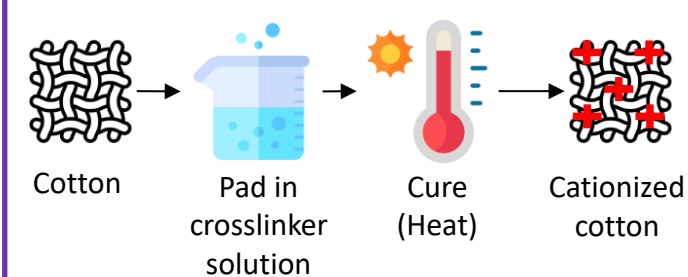


Typical chemistry (example)



Cotton is immersed in an aqueous cationizing bath; the fibers absorb the agent and are subsequently dried and cured.

4. Crosslinking / Reactive Cationization

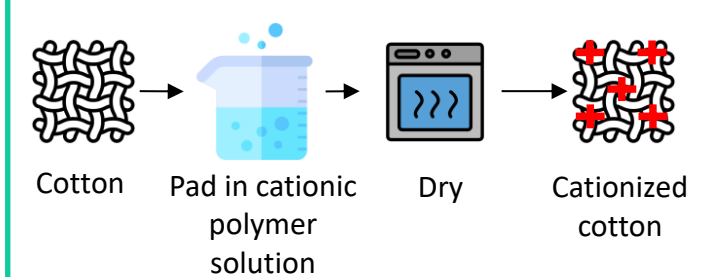


Typical chemistry (example)

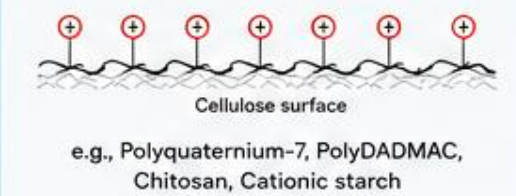


Bifunctional cationic reagents react with two cellulose chains to introduce cationic groups and form crosslinks.

5. Polymer Coating



Typical chemistry (example)



Cationic polymer is deposited/absorbed on the fibers' surface through electrostatic interactions or physical entanglement.

Advantages

- Covalent bonding gives excellent durability.
- High wash fastness and color yield.
- Good resistance to leaching.
- Wide choice of monomers for different performance.

Disadvantages

- Cost of cationic monomers can be high.
- Harsh conditions (heat, initiator) may damage fiber.
- Multi-step process.
- Possibility of yellowing or strength loss if overdone.

Advantages

- Simple and economical industrial process.
- Suitable for continuous padding.
- Good and reproducible results.
- Applicable to a wide range of cationizing agents.

Disadvantages

- Requires padding and curing equipment.
- Possible uneven cationization.
- Over-curing can cause brittleness/yellowing.
- Some agents release formaldehyde.

Advantages

- Uniform treatment of fabric (complete wetting).
- Suitable for exhaust machines.
- Good for bulky and thick fabrics.
- Less requirement of equipment.

Disadvantages

- High liquor ratio and chemical consumption.
- Longer process time than padding.
- Effluent treatment requirement.
- Possible strength loss if exposure is high.

Advantages

- Introduces cationic groups and improves dimensional stability.
- Better durability due to crosslinking.
- Improves wrinkle resistance and strength.
- Good for multifunctional finishing.

Disadvantages

- Chemicals may be expensive.
- Over-crosslinking can reduce strength and absorbency.
- Requires control of pH and temperature.
- May cause stiffness (high deposits).

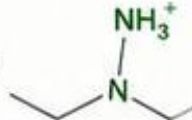
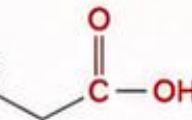
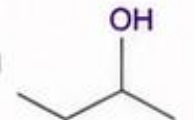
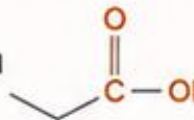
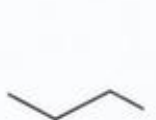
Advantages

- Mild conditions; suitable for heat-sensitive fibers/blends.
- Improves softness and handle.
- Can impart additional functional properties.
- Simple, short process time.

Disadvantages

- Durability lower than covalent methods.
- Possible loss of polymer by washing.
- May affect breathability if over-deposited.
- Some polymers are costly and may affect whiteness.

Strategy #2: Plasma Treatment (Cationization/Anionization)

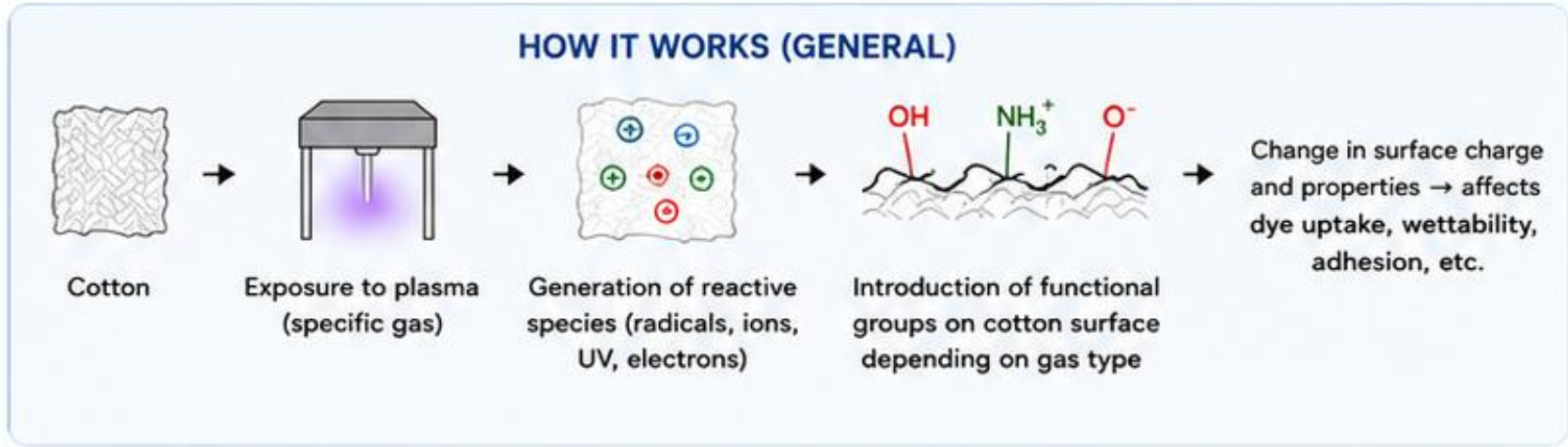
GAS TYPE DETERMINES THE SURFACE MODIFICATION				
1. AMMONIA / AMINE CONTAINING PLASMA (e.g., NH ₃ , N ₂ /H ₂ /NH ₃)	2. OXYGEN PLASMA (O ₂)	3. AIR PLASMA (O ₂ + N ₂)	4. CO ₂ PLASMA (CO ₂)	5. INERT GAS PLASMA (Ar, He)
Leads to CATIONIZATION (positive surface)	Leads to ANIONIZATION (negative surface)	Leads to ANIONIZATION (negative surface)	Leads to ANIONIZATION (negative surface)	Leads to PHYSICAL ACTIVATION (Neutral / no charge)
				
Introduces cationic groups such as -NH ₃ ⁺ , -NH ₂ ⁺ , -NR ₃ ⁺ etc.	Introduces oxygen-containing groups such as -OH, -COOH, C=O, -O-, etc.	Mainly introduces polar oxygen-containing groups (-OH, -COOH, C=O, etc.).	Introduces carboxyl and carbonate type groups (-COOH, -O-COO-, etc.).	Primarily cleans and activates the surface by etching; very little chemical functionality introduced.
Result: Surface becomes positively charged → Cotton CATIONIZATION	Result: Surface becomes negatively charged → Cotton ANIONIZATION	Result: Surface becomes negatively charged → Cotton ANIONIZATION	Result: Surface becomes negatively charged → Cotton ANIONIZATION	Result: Activated surface (Neutral). Further chemical steps needed for charge introduction.

 **ADVANTAGES**

- ✓ Dry process or minimal water usage.
- ✓ Short treatment time (seconds to minutes).
- ✓ Environmentally friendly (no liquid effluent).
- ✓ Precise control over surface chemistry by selecting gas, power and time.
- ✓ Improves dyeability, adhesion, wettability, antibacterial properties, etc.
- ✓ Can be combined with other finishing processes.

 **DISADVANTAGES**

- ✗ High initial cost of plasma equipment.
- ✗ Effectiveness depends on fabric type, structure and plasma parameters.
- ✗ Surface effects may be lost over time if not followed by stabilization (e.g., after-wash, curing).
- ✗ Limited penetration (mainly surface modification).
- ✗ Scale-up and continuous industrial application can be challenging.



 **KEY MESSAGE**

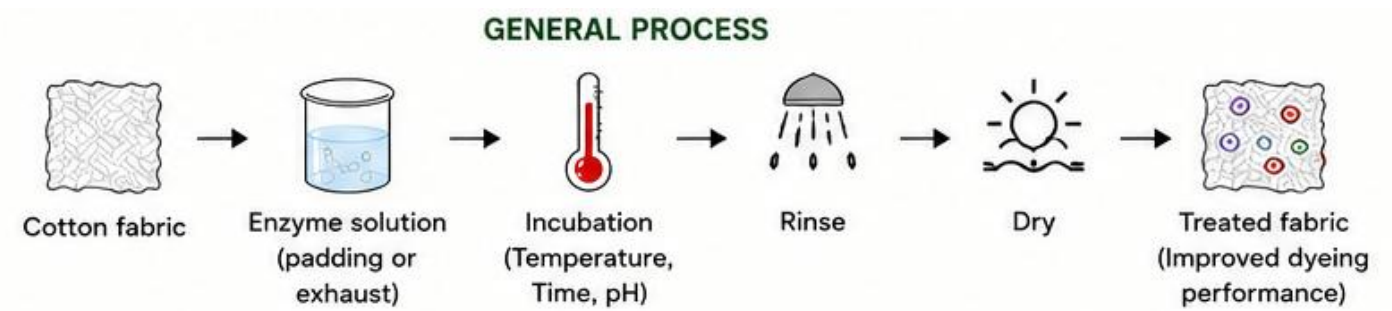
The gas used in plasma treatment is the deciding factor:

- Ammonia/amine plasma = CATIONIZATION (+)
- Oxygen/Air/CO₂ plasma = ANIONIZATION (-)
- Inert gas plasma = ACTIVATION (neutral)

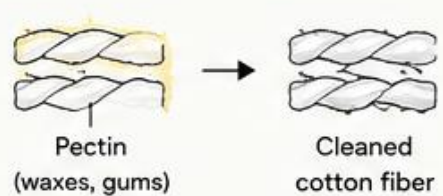
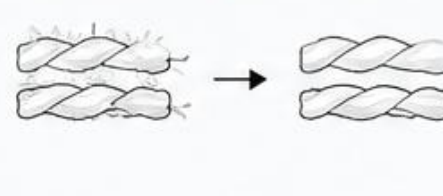
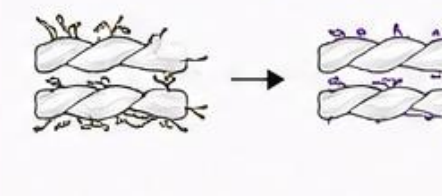
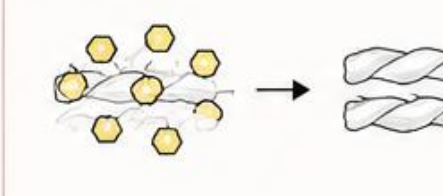
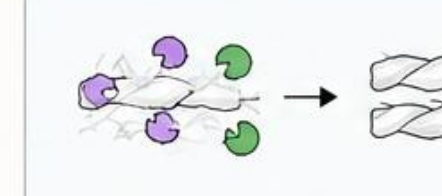
Note: Plasma treatment alone may only activate the cotton surface or introduce functional groups; for stable cationization, ammonia/amine plasma or subsequent chemical treatment may be required.

Strategy #3: Enzymatic treatment

Enzymatic treatment uses specific enzymes to modify the cotton surface and structure, improving absorbency, dye uptake, levelness and overall dyeing performance in an eco-friendly manner.



TYPES OF ENZYMES AND THEIR ACTION

1. PECTINASE (Pectin degrading enzyme)	2. CELLULASE (Cellulose degrading enzyme)	3. HEMICELLULASE (Hemicellulose degrading enzyme)	4. LACCASE (Oxidoreductase enzyme)	5. COMBINED ENZYME SYSTEMS (e.g., Pectinase + Cellulase)
				
• Removes pectin, waxes and hemicellulosic impurities. • Improves absorbency, wettability and dye penetration.	• Partially hydrolyzes protruding cellulose fibrils. • Improves smoothness, reduces pilling and improves dye levelness.	• Breaks down hemicelluloses. • Enhances dye uptake and brightness.	• Oxidizes phenolic impurities. • Helps in bleaching and improves whiteness and dye affinity.	• Synergistic action for better purification and modification. • Gives optimum dyeing results.



ADVANTAGES

- ✓ Environmentally friendly (biodegradable, non-toxic).
- ✓ Works under mild conditions (low temperature, neutral pH).
- ✓ Reduces use of harsh chemicals and energy.
- ✓ Improves absorbency, wettability and dye penetration.
- ✓ Enhances dye uptake, levelness, brightness and fastness.
- ✓ Improves handle, softness and reduces pilling.
- ✓ Reduces effluent load and pollution.
- ✓ Can be used in padding, exhaustion or continuous processes.



DISADVANTAGES

- ✗ Enzymes can be relatively expensive.
- ✗ Requires precise control of pH, temperature and time.
- ✗ Over-treatment can reduce fabric strength and cause fuzziness.
- ✗ Enzyme activity can be affected by hard water, heavy metals and unsuitable chemicals.
- ✗ Requires inactivation of enzymes after treatment.
- ✗ Storage and shelf life need to be properly managed.

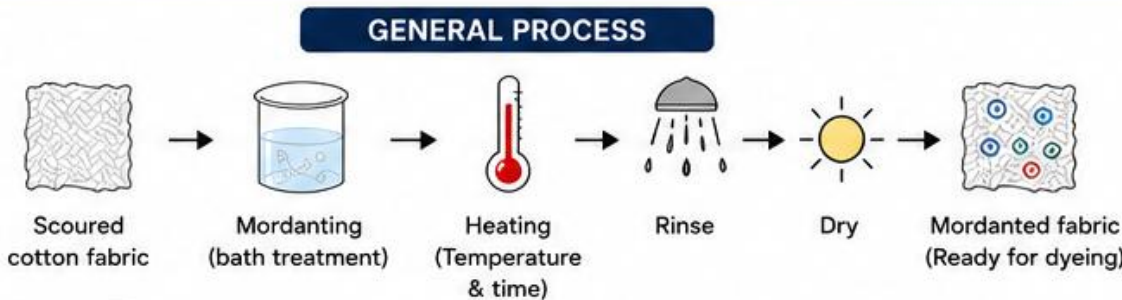
- ❑ Enzymatic treatment is most commonly used as a **pre-treatment before dyeing**.
- ❑ Optimum conditions depend on:
 - Enzyme type
 - Enzyme concentration
 - Fabric construction
 - Desired outcome
- ❑ Enzymes should be **inactivated before dyeing**, e.g., by:
 - Washing with hot water
 - Adjusting pH
 - Adjusting temperature

Innovative Strategies

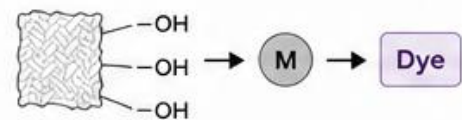
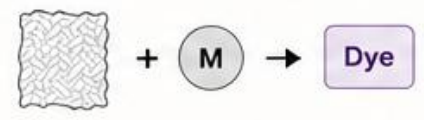
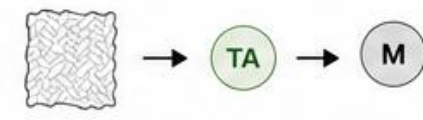
Strategy #4: Mordanting process

MORDANTING PROCESS FOR TEXTILE DYEING

Mordanting is the process of treating cotton fibres with metallic salts (mordants) to form coordination complexes with the fibre. This improves the affinity of the fibre for dyes, enhances dye uptake, and improves fastness properties.



TYPES OF MORDANTS AND THEIR ACTION

1. Metallic Salt Mordants (Commonly used)	2. Pre-mordanting (Before dyeing)	3. Meta-mordanting (With dye)	4. Post-mordanting (After dyeing)	5. Tannic Acid Mordanting (Natural mordant)
Alum (Potassium aluminium sulfate), Ferrous sulfate, Stannous chloride, Copper sulfate, Chromium salts, etc.  • Metal ions form coordination complexes with –OH groups of cellulose and dye molecules.	Fabric is first treated with mordant, then washed and dyed.  • Forms stable metal–fiber sites before dye application. • Gives good penetration and level dyeing.	Mordant and dye are added together in the dye bath.  • Metal ion reacts with both dye and fiber simultaneously. • Often gives brighter shades and better fastness.	Fabric is first dyed, then treated with mordant solution.  • Metal ion complexes with dye already on the fiber. • Useful for some dyes where pre-mordanting gives dull shades.	Tannic acid is applied before metal mordant to improve binding.  • Tannic acid acts as a bridge between fiber and metal ion. • Enhances dye uptake and fastness.

ADVANTAGES

- ✓ Improves dye uptake and exhaustion.
- ✓ Enhances fastness to washing, light and rubbing.
- ✓ Produces brighter, deeper and more level shades.
- ✓ Wider range of dyes can be used effectively.
- ✓ Improves overall durability of dyed fabrics.
- ✓ Essential for natural dyes which have low affinity for cotton.

DISADVANTAGES

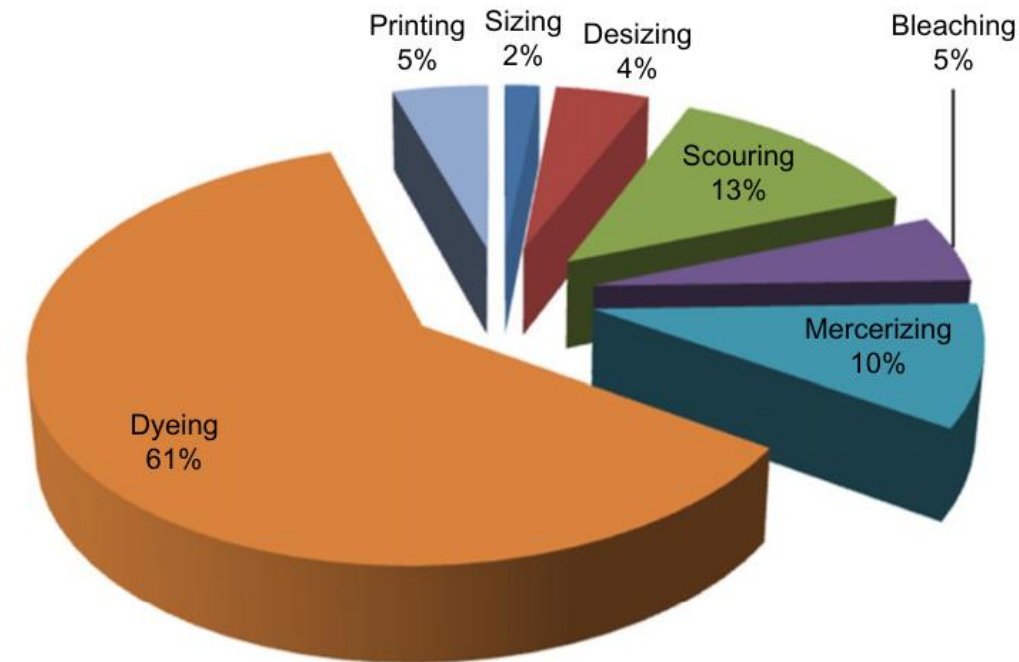
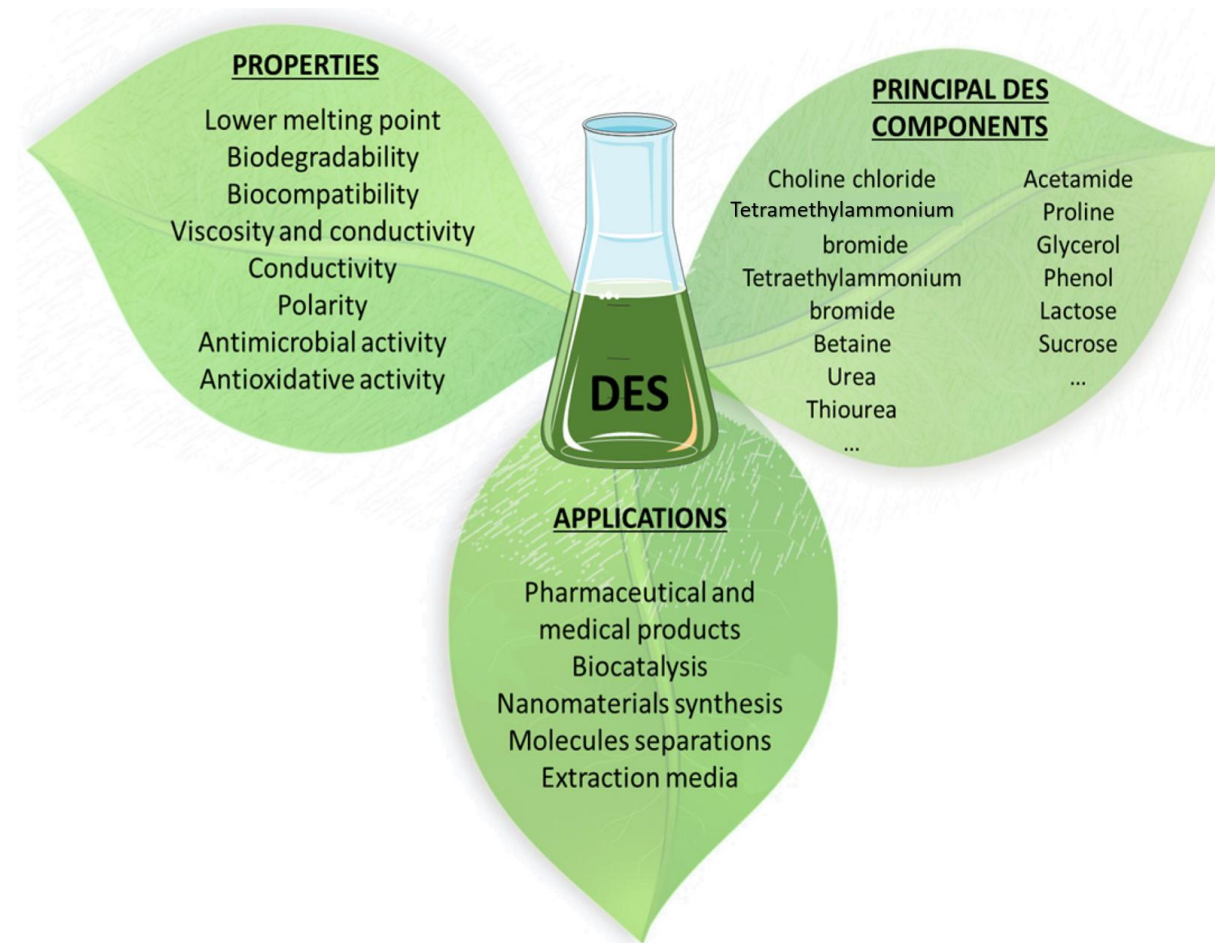
- ✗ Extra processing step increases cost, time and chemical consumption.
- ✗ Some mordants (e.g., chromium, tin) are toxic and cause environmental problems.
- ✗ May cause fabric yellowing or change in handle.
- ✗ Incorrect mordant concentration can lead to poor fastness or spotting.
- ✗ Requires proper washing to remove unfixed mordant.
- ✗ Disposal of metal-containing effluents needs proper treatment.

Common Mordants

- **Alum:** Most widely used; provides good overall fastness.
 - **Ferrous sulfate:** Produces darker shades and improves wash fastness.
 - **Stannous chloride:** Enhances tinctorial strength; suitable for direct dyes.
 - **Copper sulfate:** Produces brighter shades and provides good light fastness.
 - **Chromium salts:** Provide excellent fastness but should be used with caution due to toxicity.
- ☐ Mordant choice and treatment conditions depend on the **dye type, desired shade, and required fastness.**

Innovative Strategies

Strategy #5: Eutectic Solvent-based Dyeing



- **Deep eutectic solvents (DESs)** provide a greener alternative to conventional dyeing media, with **low melting points, biodegradability, and low volatility**.
- DESs can **enhance dye solubility and extraction**, enabling efficient coloration using **natural dyes from plant-based sources**.
- DES-based dyeing can potentially **reduce water and hazardous chemical consumption** compared with conventional textile dyeing.
- **Tunable DES composition** allows solvent properties such as viscosity, conductivity, and polarity to be optimized for different dyes and textile fibers.
- **Key challenges:** high viscosity, solvent recovery/reuse, dye–fiber compatibility, color fastness, and scaling up the process.
- **Overall:** DES-based dyeing offers a promising pathway toward **more sustainable, circular, and resource-efficient textile coloration**.

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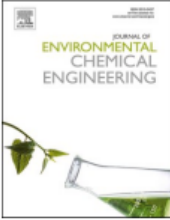
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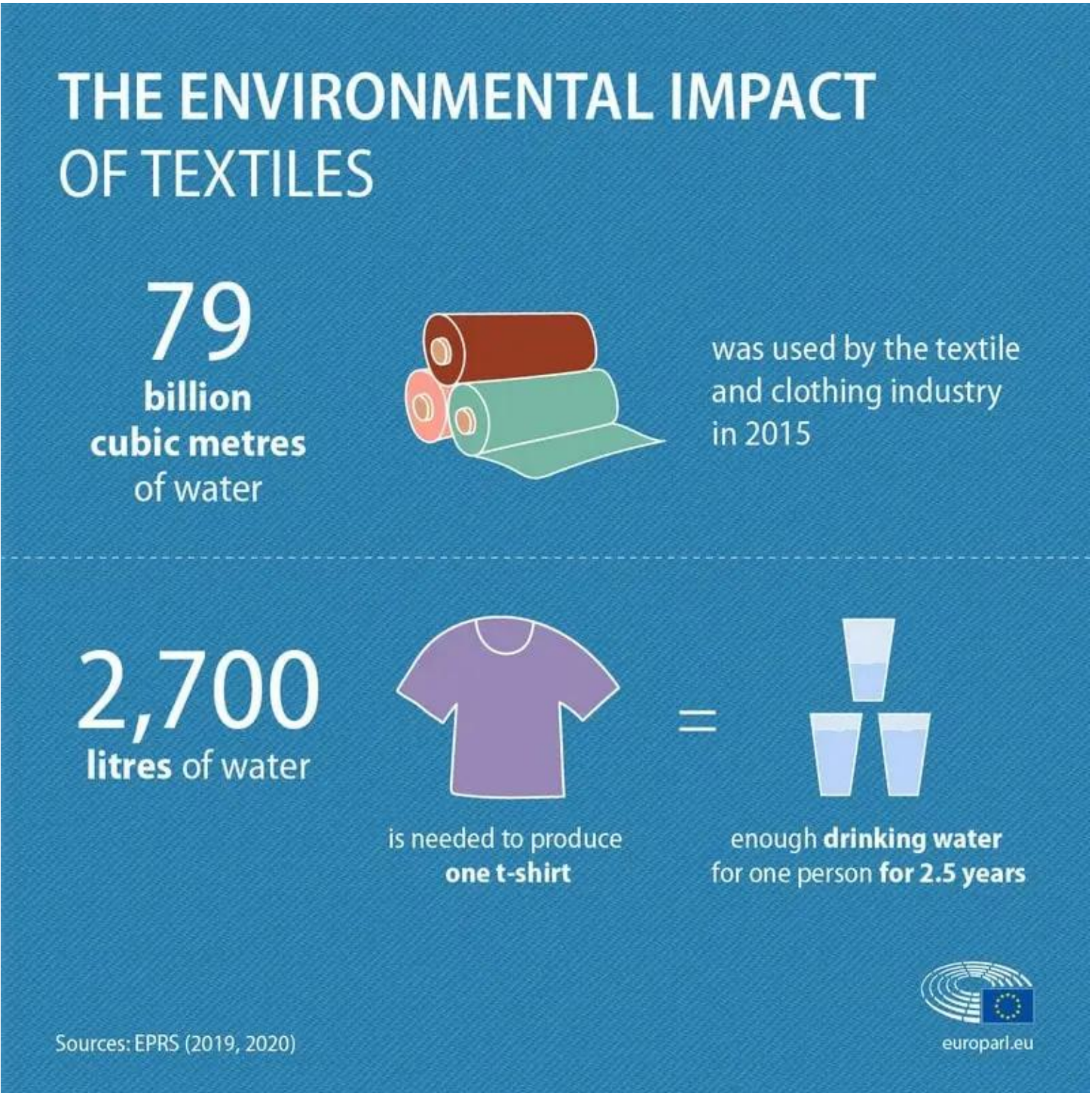
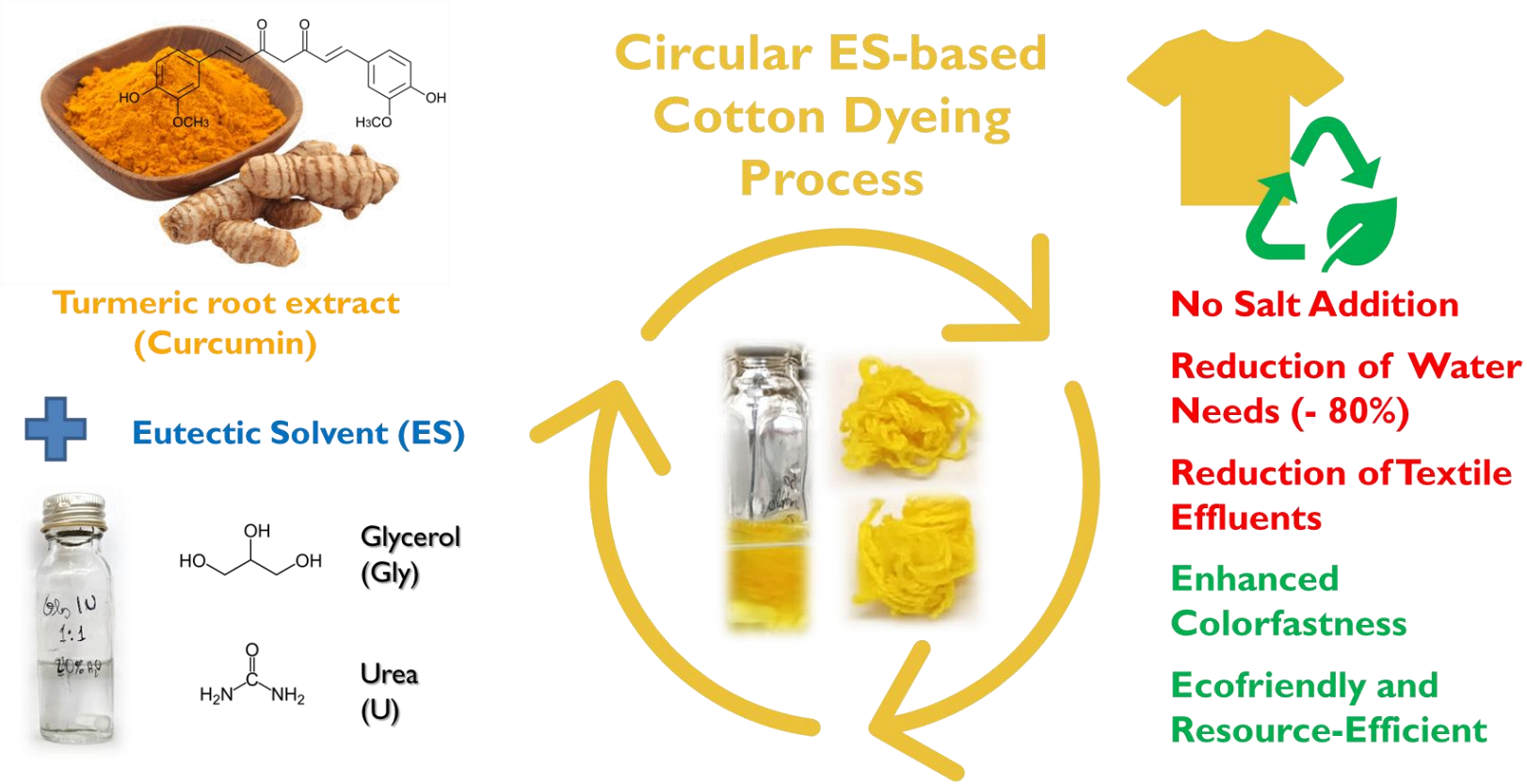
Natural and eco-friendly cotton dyeing process using eutectic solvents and curcumin

Alexandre M.S. Jorge^a, Helena F. Ribeiro^a, Jorge F.B. Pereira^{a,b,*}

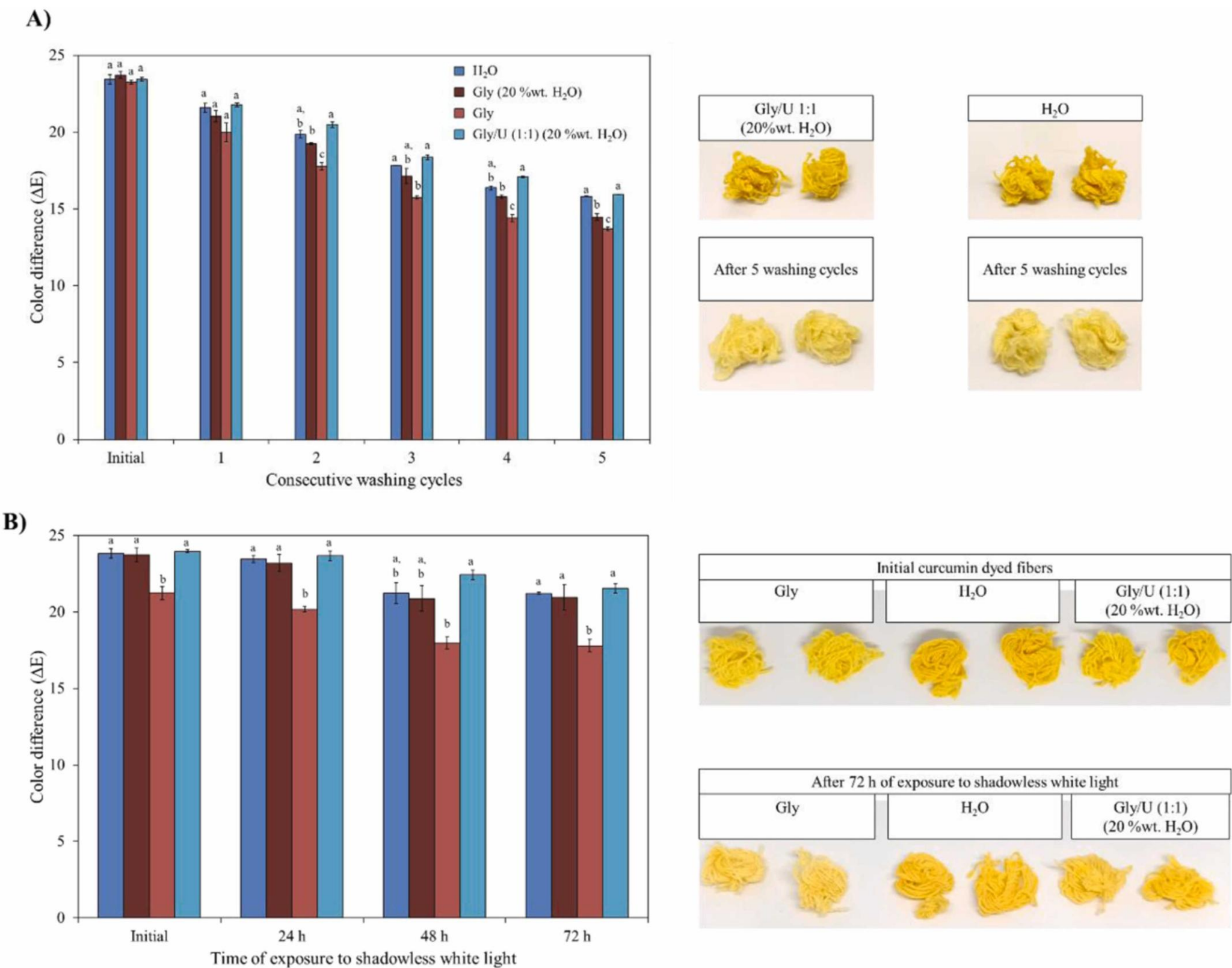
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^b Department of Chemical Engineering of the Faculty of Sciences at the University of Coimbra, Portugal





Comparison of Gly/U ES with control dye baths



ES-dyed fibers exhibited higher stability compared to the control samples, for both the wash fastness ([Fig. 5A](#)) and light fastness ([Fig. 5B](#)) tests.

The **higher solubility of curcumin in ES-based solutions**, facilitated by the intrinsic hydrogen-bonding networks formed between the ES constituents, results in **lower curcumin exhaustion from the dye bath** but with **comparable fiber coloration to water-based dye baths**

Fig. 5. Wash (A) and light (B) fastness of the curcumin dyed cotton fibers using different dye baths: 100 %wt. H₂O, Gly (20 % wt. H₂O), 100 %wt. Gly and Gly/U 1:1 (with 20 %wt. H₂O). Each dyeing cycle was carried out at 60 °C for 60 min, using 0.1 g of cotton fiber was dyed using a dye bath prepared by adding 0.1 mL of curcumin stock solution to 3 mL of ES.

Design of circular ES-based cotton dyeing platform

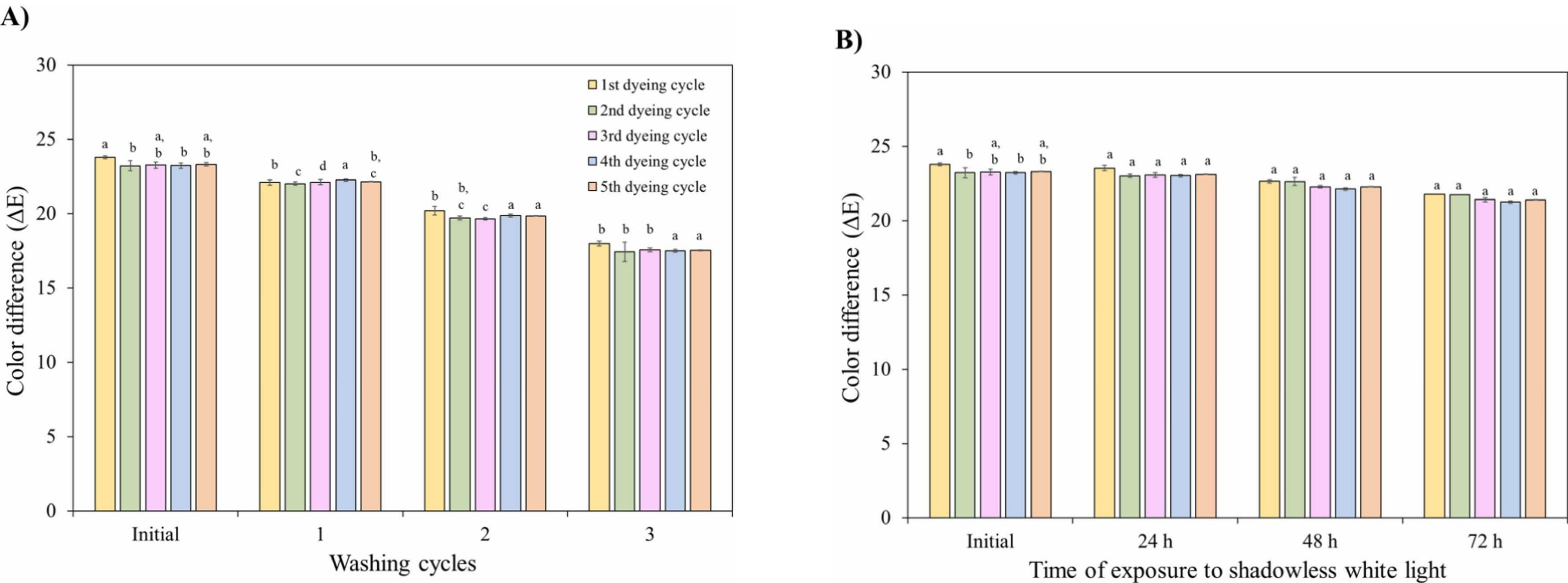
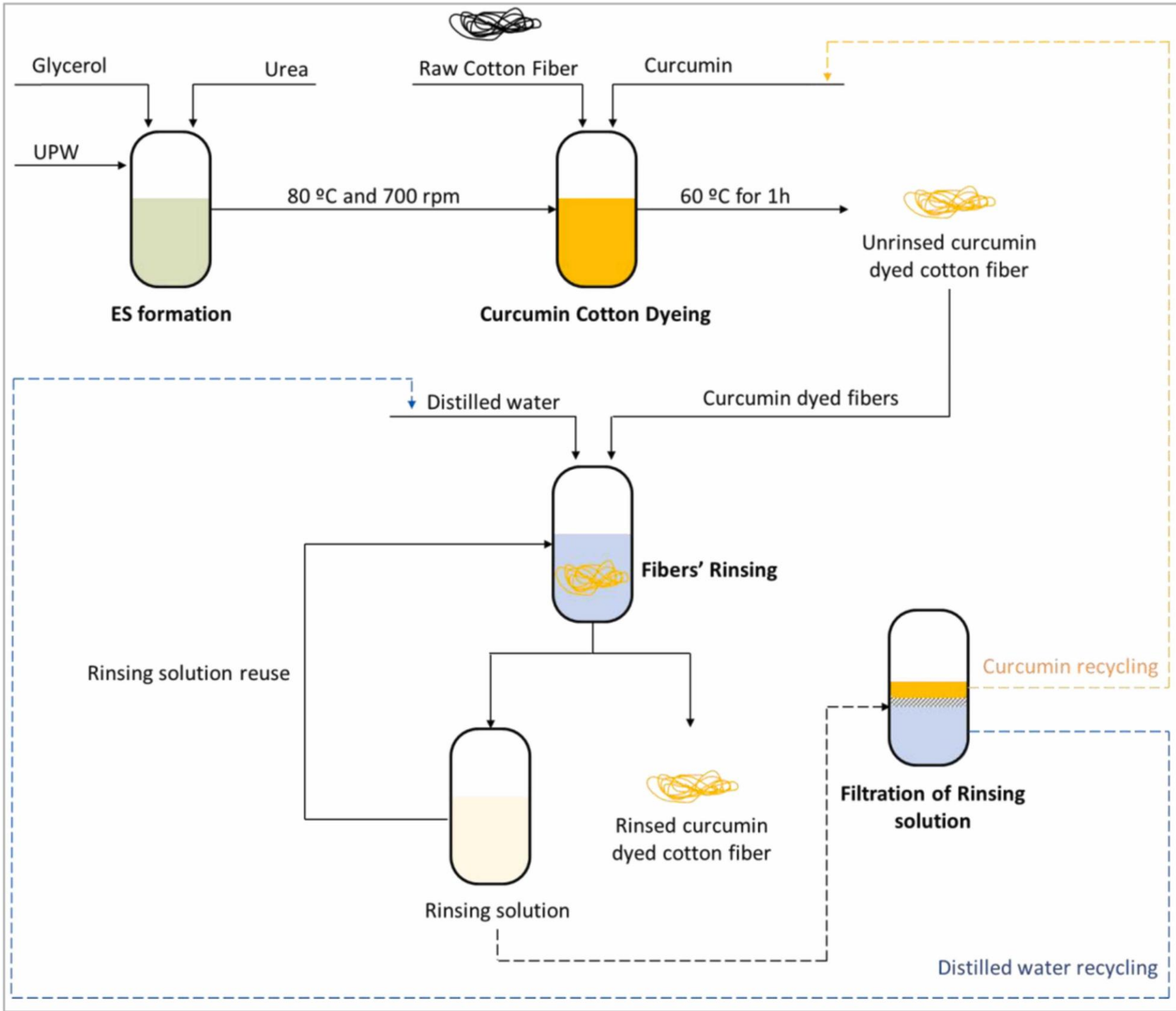


Fig. 7. Wash (A) and light (B) fastness of the curcumin dyed cotton fibers obtained in each dyeing cycle of the circular ES-based dyeing process. Each dyeing cycle was carried out at 60 °C for 60 min, using 0.1 g of cotton fiber was dyed using a dye bath prepared by adding 0.1 mL of curcumin stock solution to 3 mL of ES. To keep the fiber-to-liquor ratio and curcumin concentration consistent throughout consecutive dyeing cycles, adjustments to the ES and curcumin masses were made to the dye bath after each dyeing cycle. Columns with different letters differ significantly ($P < 0.05$) by Tukey test.

Design of circular ES-based cotton dyeing platform

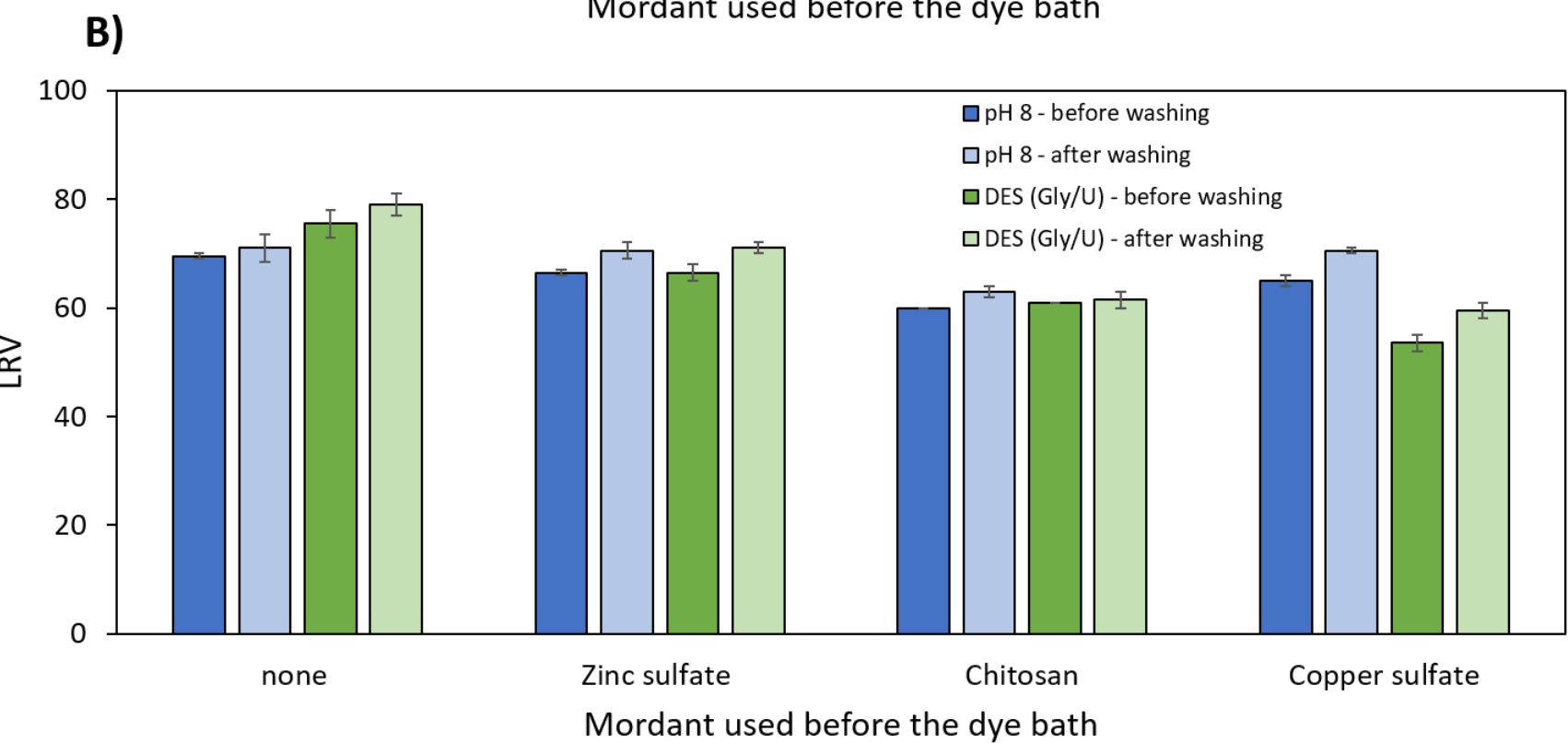
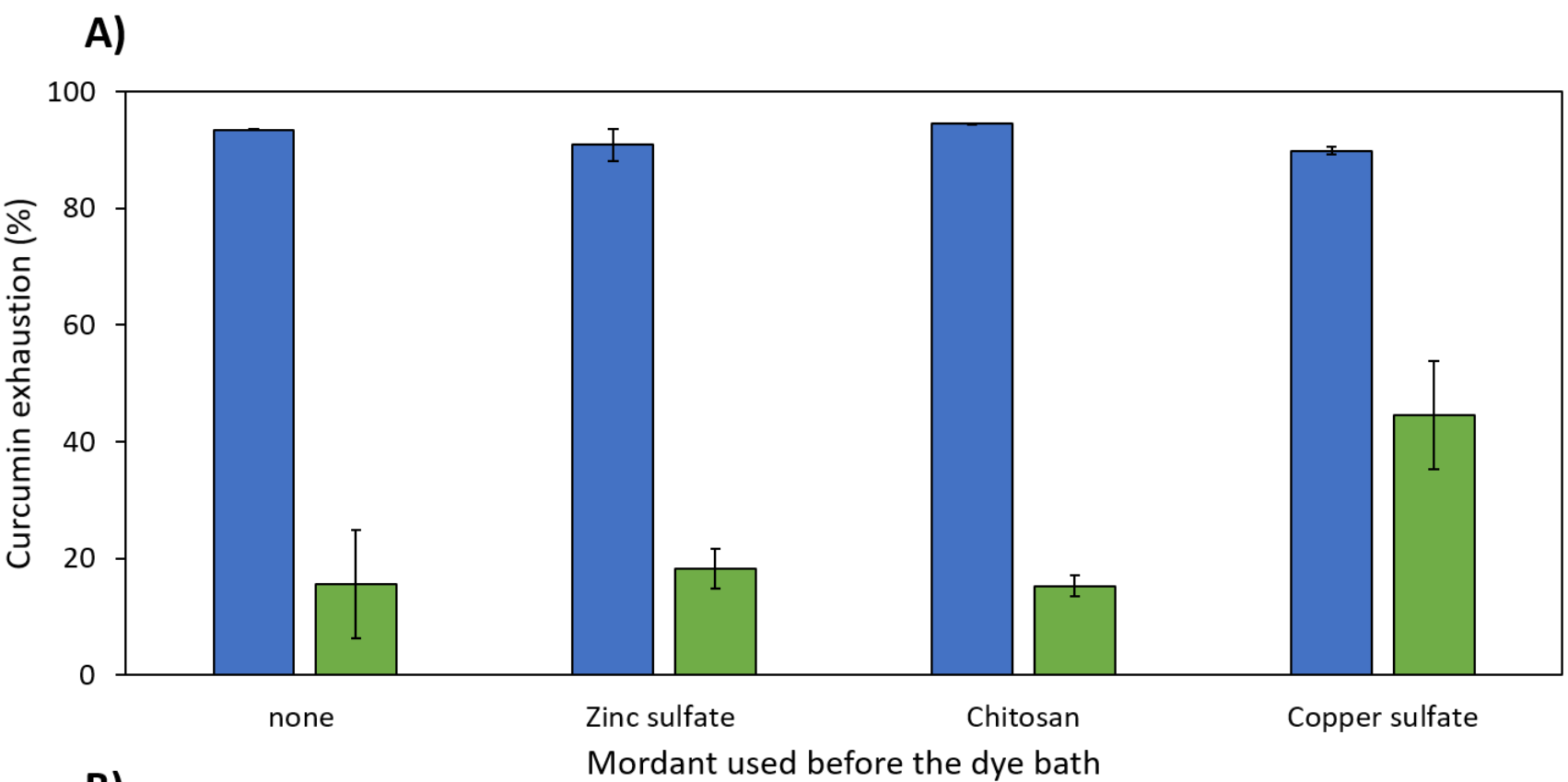


Summary:

- **Gly:U (1:1) (20 %wt. H₂O)** - most cost-effective ES for curcumin-based cotton dyeing
- **ES demonstrated superior dyeing performance and fastness properties** compared to control samples
- **Curcumin exhaustion was reduced by 10–50 %** compared to water-based dye baths while maintaining similar **colorimetric properties** ($\Delta E \approx 24.0$)
- The designed process has the potential to **reduce water usage by 80 % and eliminate the need for salts** as additives to enhance textile dyeing.

Fig. 8. Schematic representation of the circular and sustainable curcumin-based cotton dyeing process designed using the Gly/U 1:1 (20 %wt H₂O), including key findings and outcomes of the study..

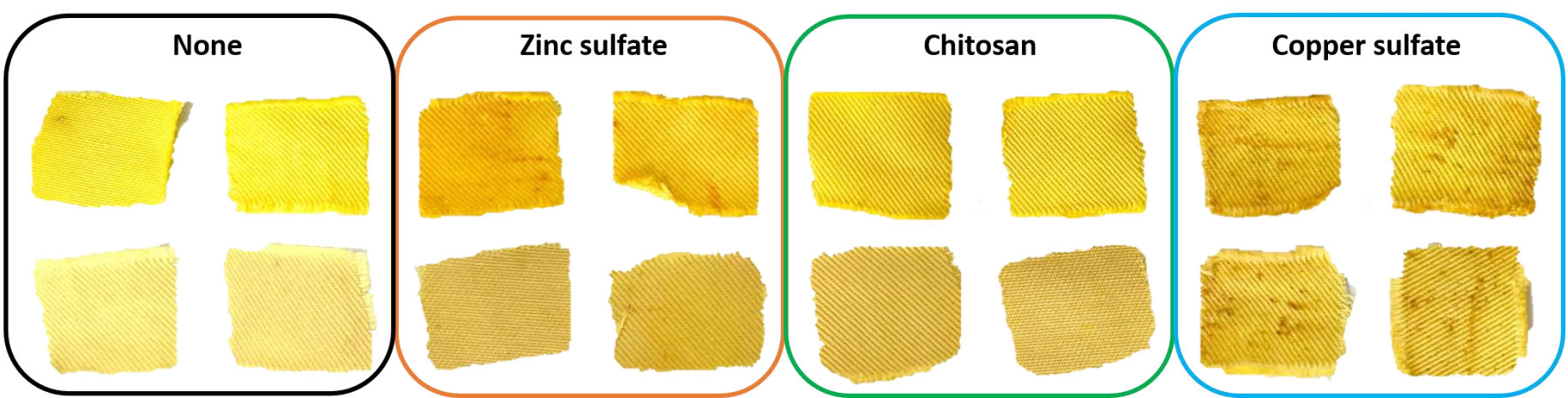
Water-based vs ES-based Dye bath with mordants



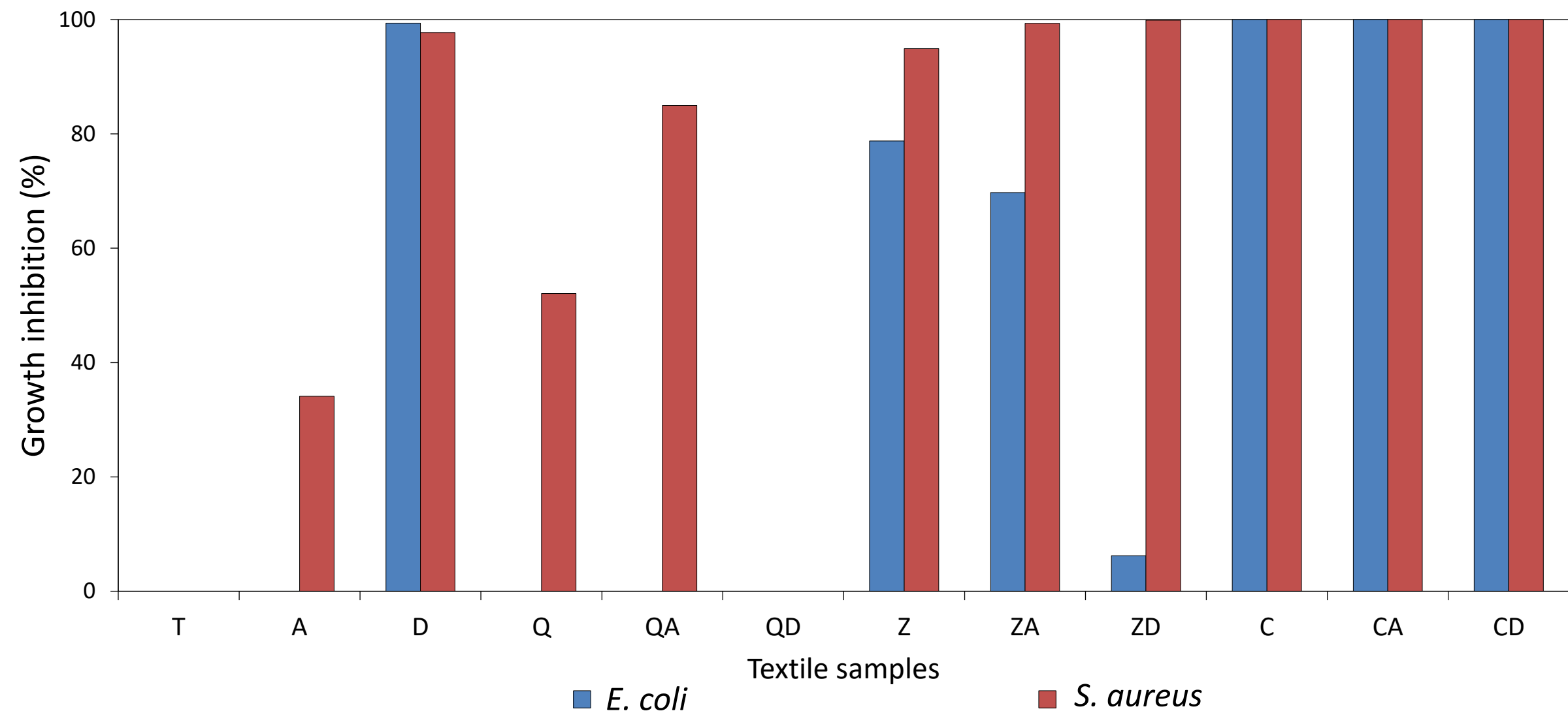
Before and after washing
(pH 8)



Before and after washing
(DES (Gly/U))



Antimicrobial activity of the dyed fabrics

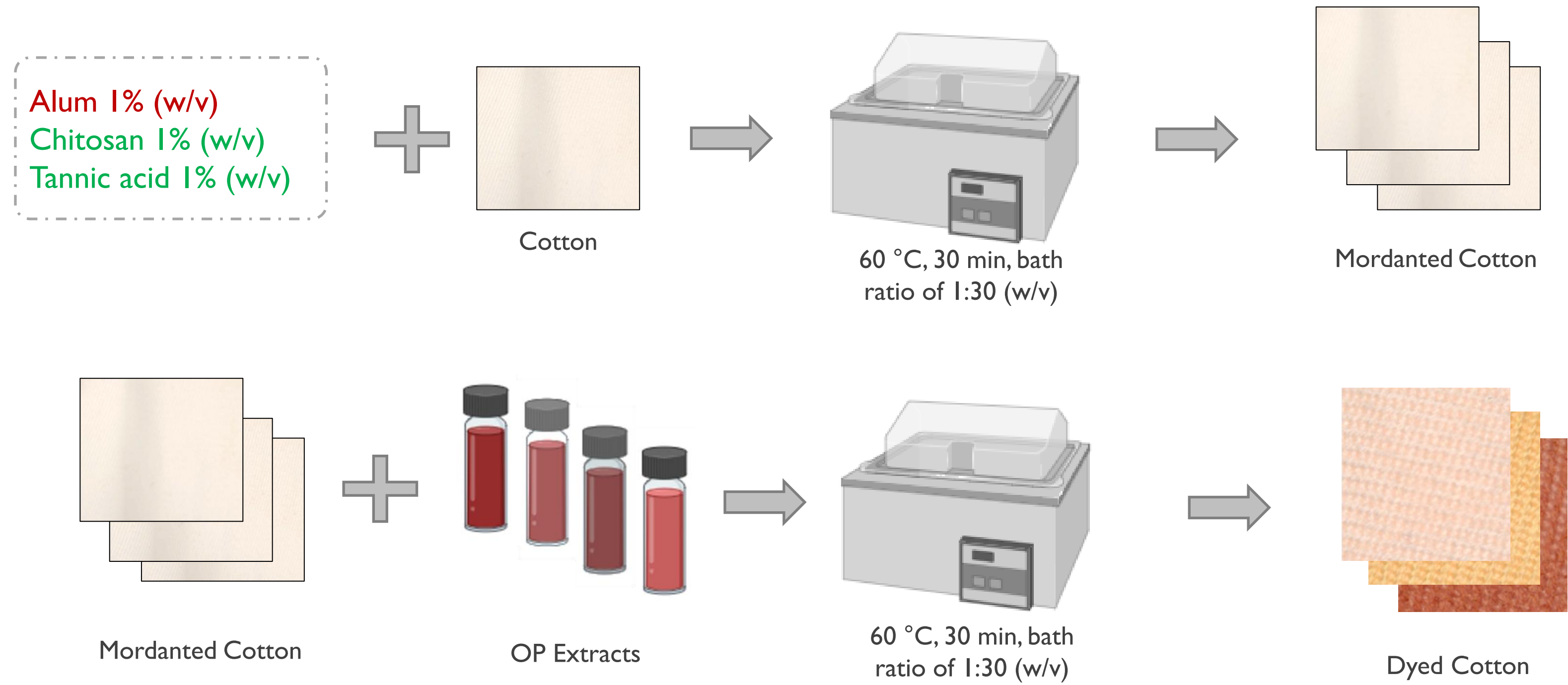


Growth inhibition (%) of *E. coli* (blue) and *S. aureus* (red) in each textile sample. Each set of letters represents a sample:

- T** – fabric treated with Na_2CO_3 ;
- A** – fabric treated with Na_2CO_3 + curcumin + rinsed with water;
- D** – fabric treated with Na_2CO_3 + curcumin + washed with detergent;
- Q** – fabric treated with Na_2CO_3 + chitosan;
- QA** – fabric treated with Na_2CO_3 + chitosan + curcumin + rinsed with water;
- QD** – fabric treated with Na_2CO_3 + chitosan + curcumin + washed with detergent;

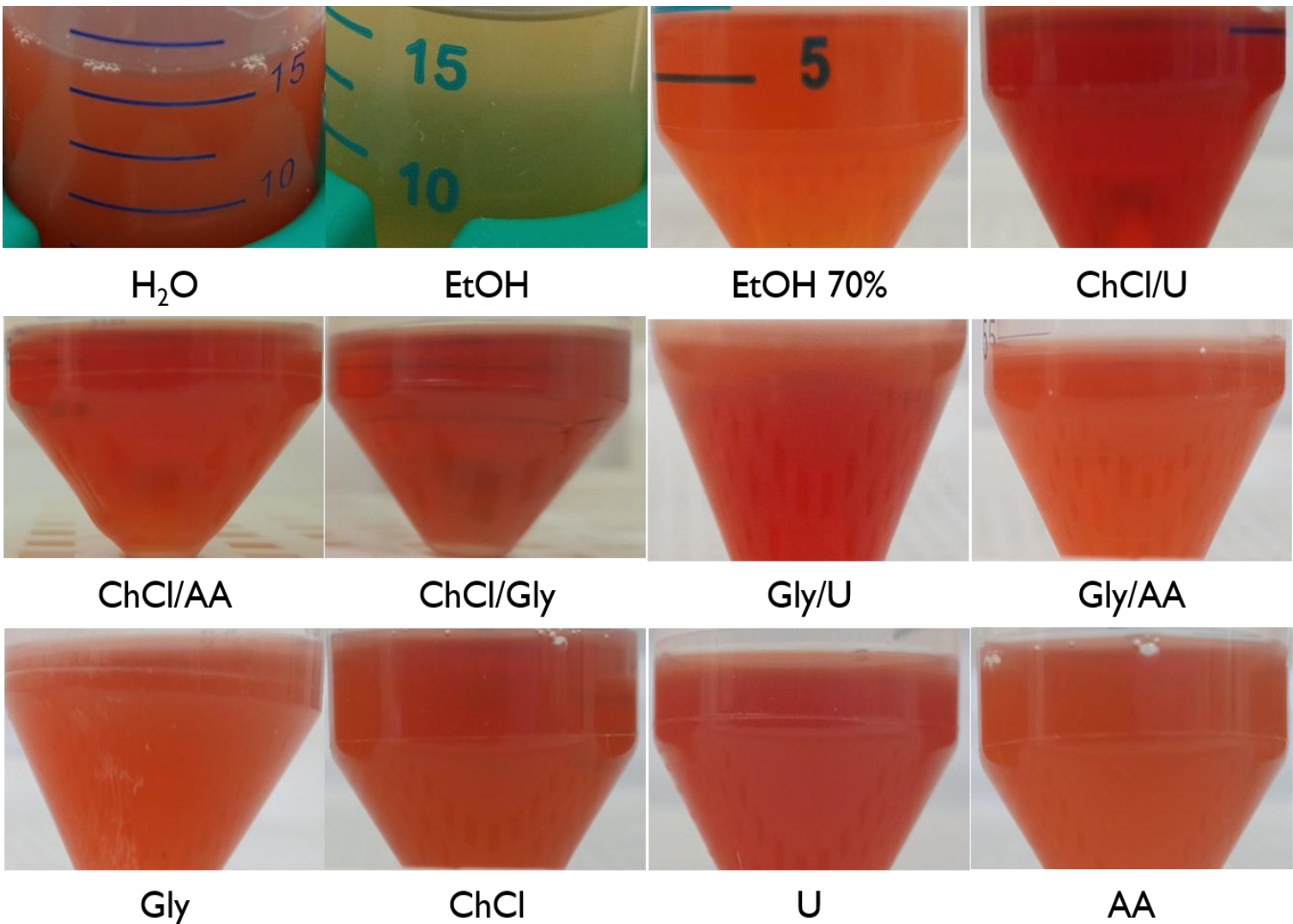
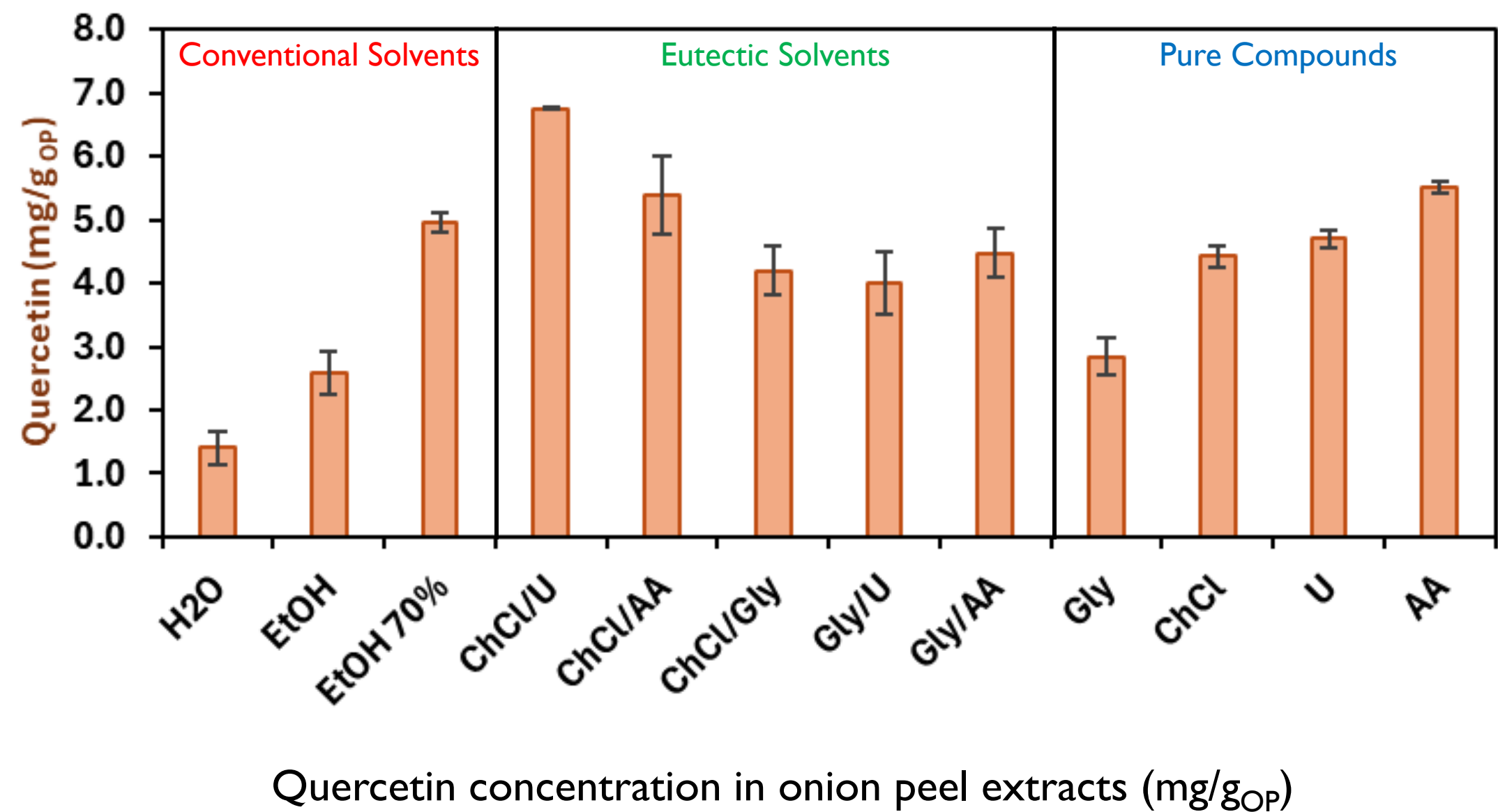
- Z** – fabric treated with Na_2CO_3 + zinc;
- ZA** – fabric treated with Na_2CO_3 + zinc + curcumin + rinsed with water;
- ZD** – fabric treated with Na_2CO_3 + zinc + curcumin + washed with detergent;
- C** – fabric treated with Na_2CO_3 + copper;
- CA** – fabric treated with Na_2CO_3 + copper + curcumin + rinsed with water;
- CD** – fabric treated with Na_2CO_3 + copper + curcumin + washed with detergent

Cotton Mordanting and Dyeing



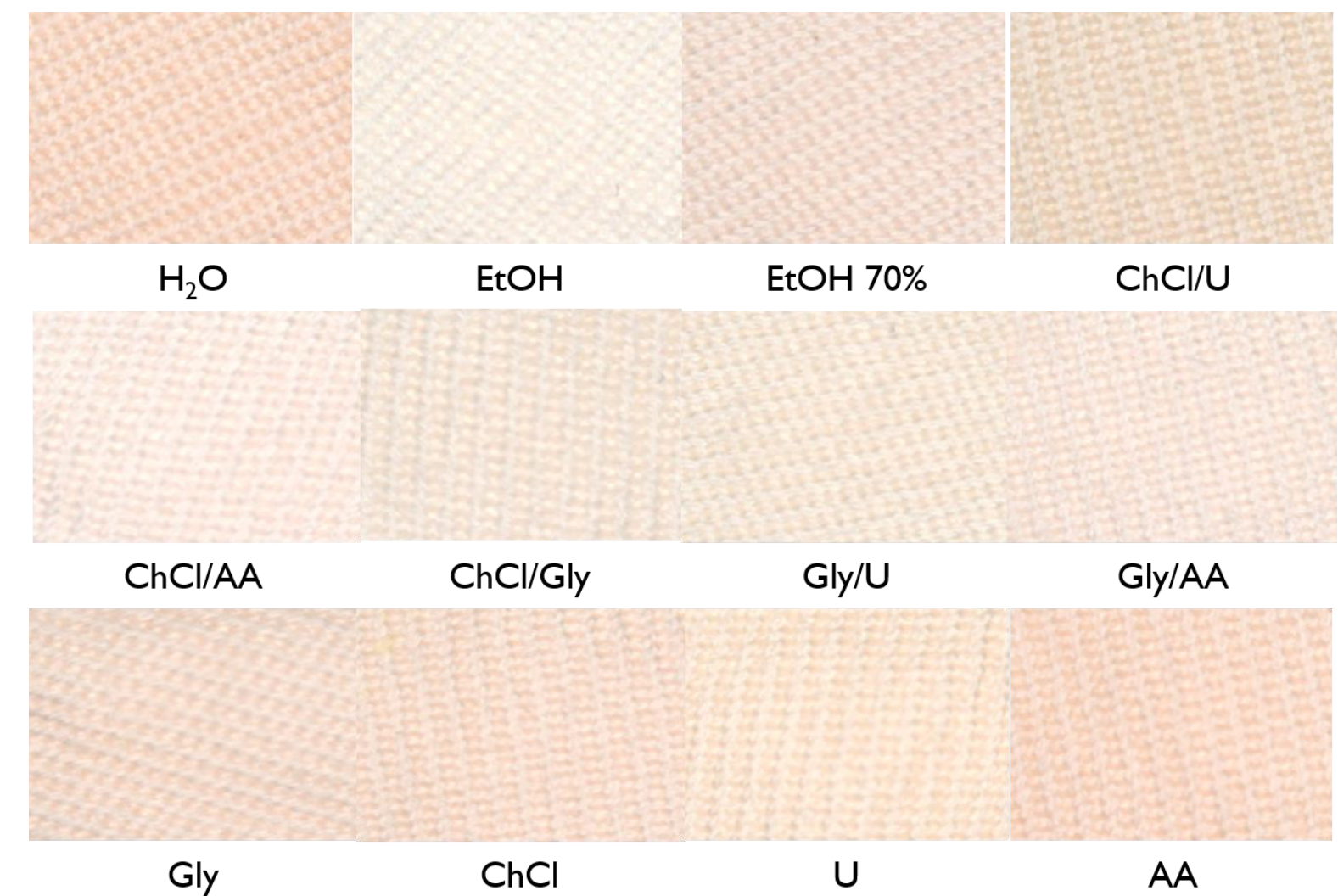
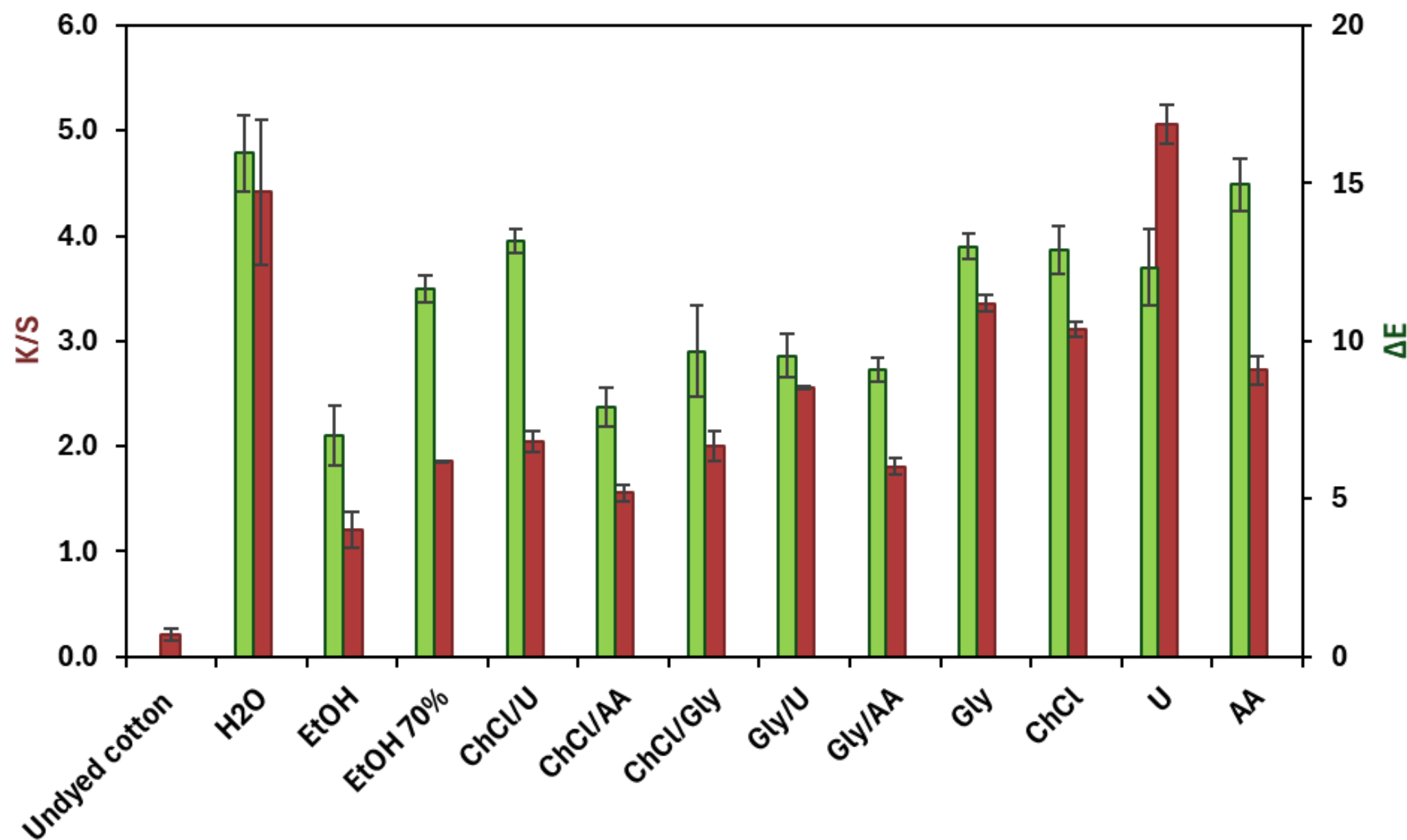
Textile characterisation: Colour Strength (K/S), Colour Variation (ΔE), and Ultraviolet Protection Factor (UPF)

Solvents Screening for Extraction



Eutectic solvents (ES) achieved up to **4.8-fold higher quercetin extraction** from onion peels compared to conventional solvents.

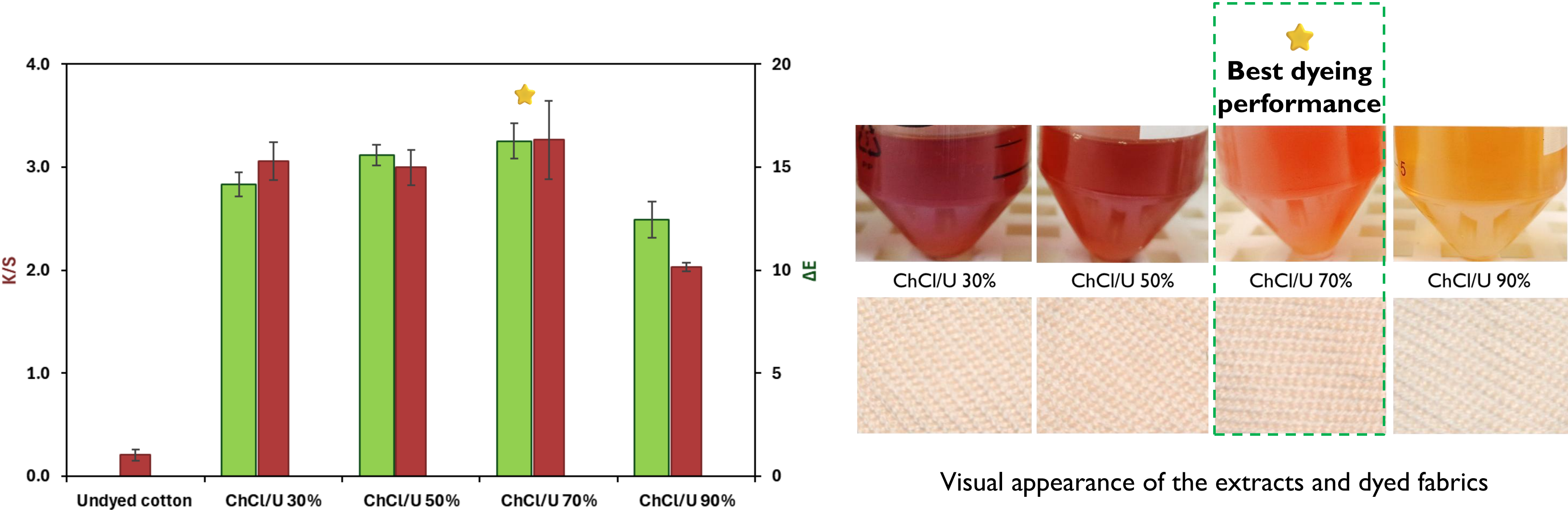
Solvents Screening for Dyeing



Visual appearance of dyed fabrics

Eutectic solvents exhibited higher extraction efficiencies for quercetin, whereas aqueous systems yielded superior dye uptake and color strength on cotton fabrics.

Influence of Water on ChCl/U (1:2) Dyeing Performance of Cotton

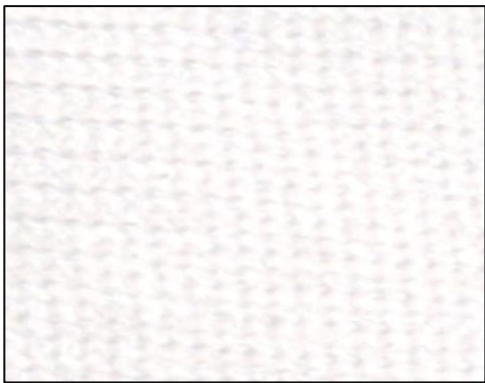


The water content of ChCl/U extracts strongly influenced dyeing performance, with intermediate dilutions (50–70% (w/w) water) providing the highest colour strength and colour variation.

Influence of Mordant Type on Fabric Appearance and Functionality



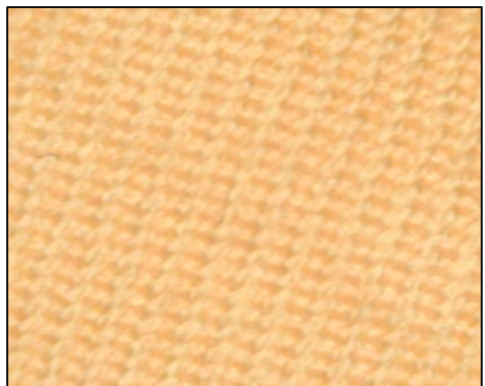
Alum



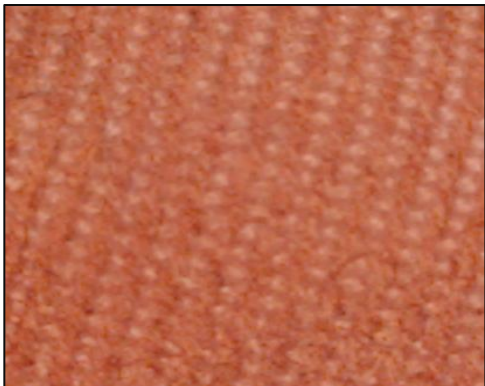
Chitosan



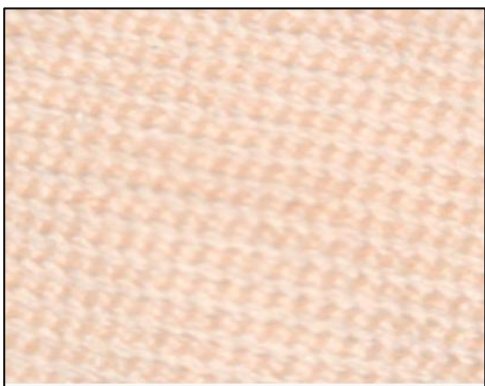
Tannic Acid



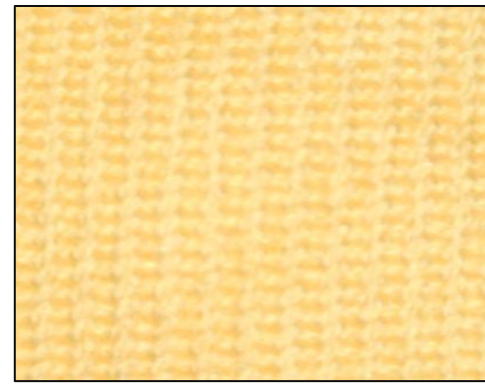
H₂O Alum



H₂O Chitosan



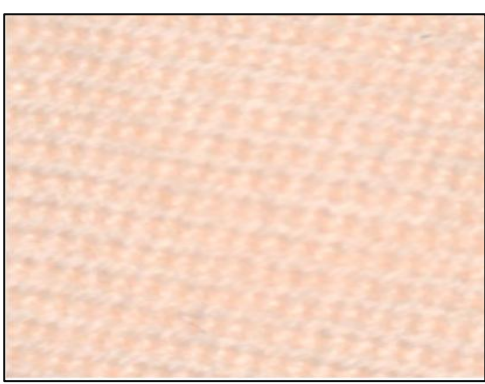
H₂O Tannic Acid



ChCl/U 70 Alum



ChCl/U 70 Chitosan



ChCl/U 70 Tannic Acid

UV Protection Performance

- Tannic Acid: UPF = 16.4 ± 1.3
- H₂O + Chitosan: UPF = 21.7 ± 4.5
- H₂O + Tannic Acid: UPF = 21.9 ± 2.1
- ChCl/U 70 + Tannic Acid: UPF = 13.6 ± 1.1

Tannic acid enhanced the UV protection of cotton fabrics even without dyeing (UPF = 16.4).

Good UV protection (UPF > 21) was achieved with H₂O extracts combined with chitosan or tannic acid.



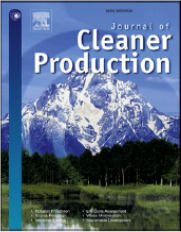
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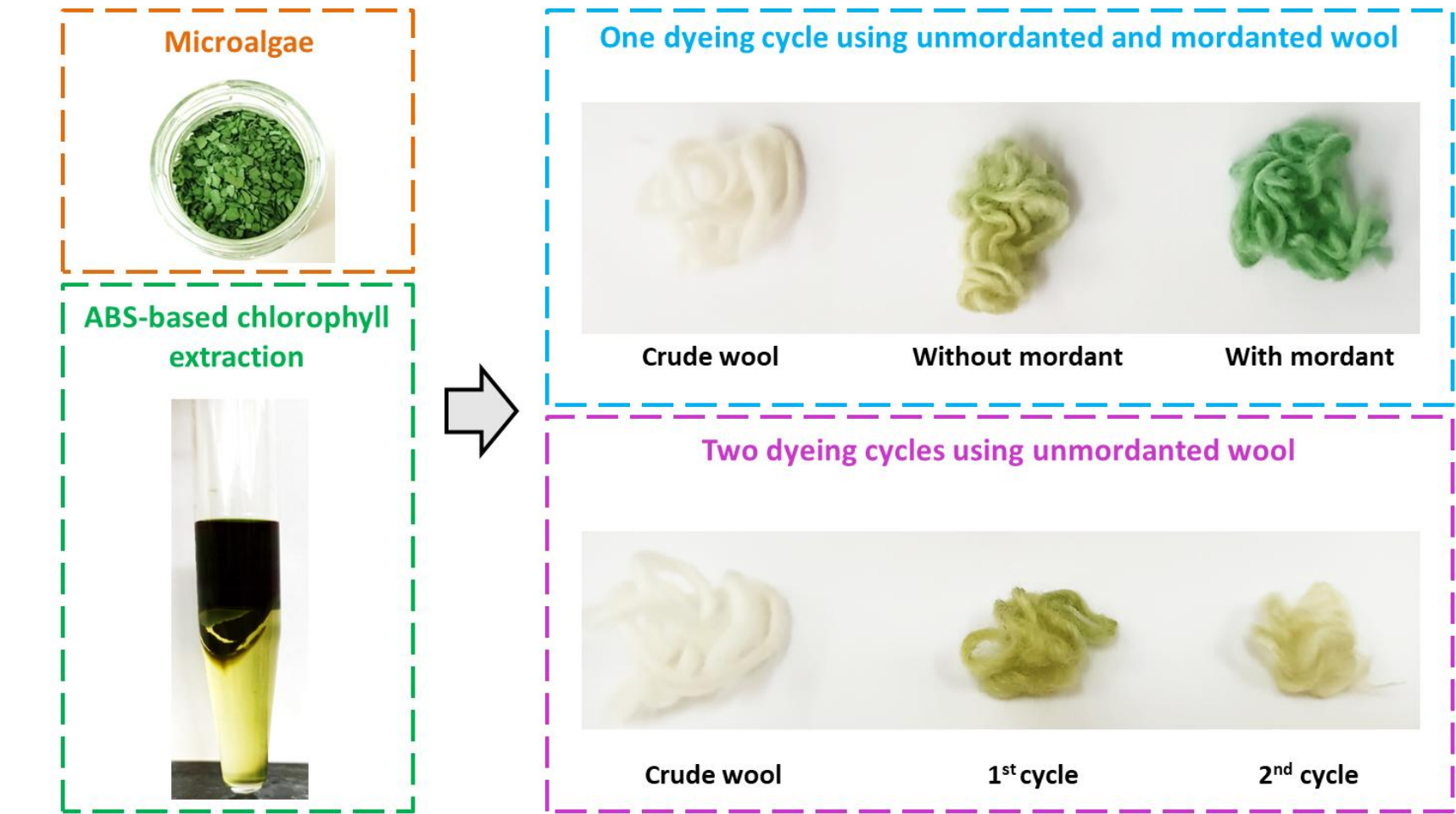
journal homepage: www.elsevier.com/locate/jclepro



Sustainable extraction and utilization of chlorophyll from microalgae for eco-friendly wool dyeing

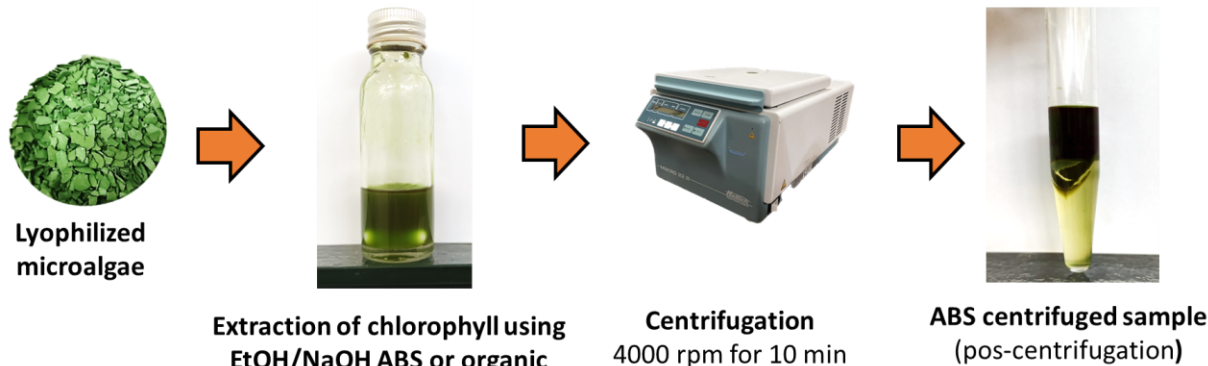
Alexandre M.S. Jorge, Pedro R.M. Pedroso, Jorge F.B. Pereira*

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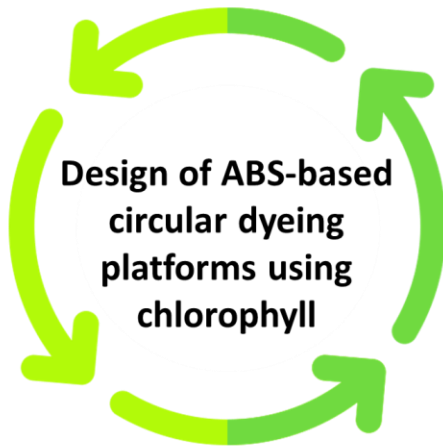
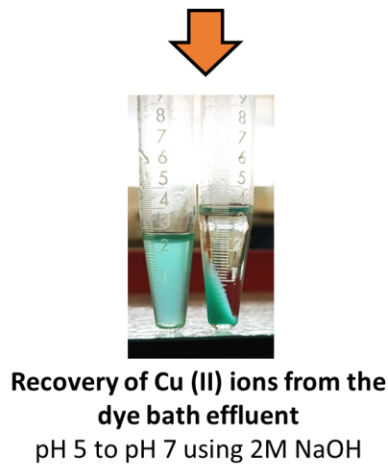
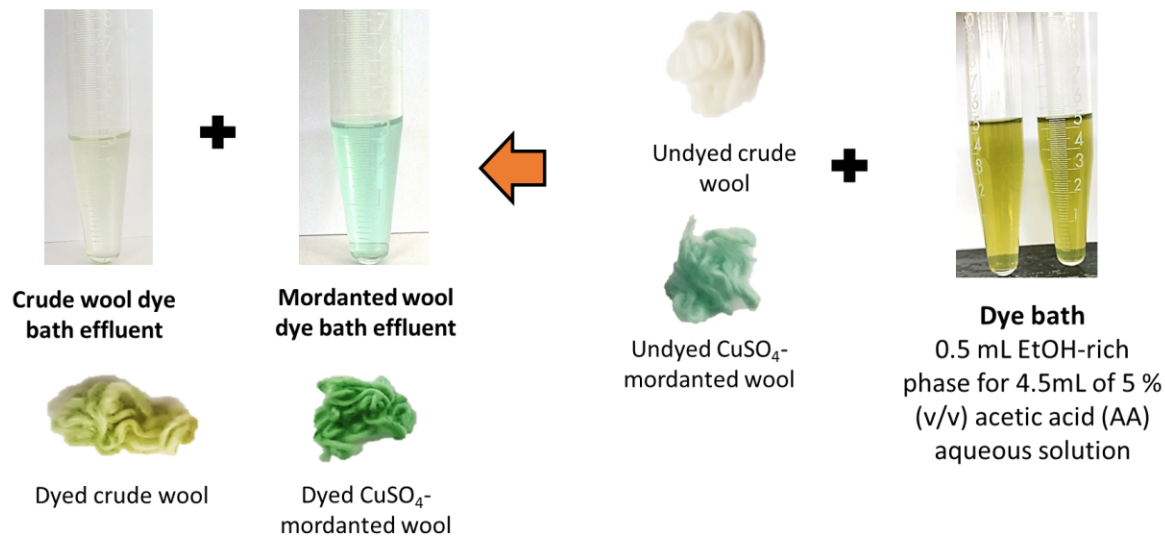


MATERIALS AND METHODS

Extraction cycle: 50 °C at 700 rpm for 1h, using 1:100 solid-to-solvent/ABS ratio

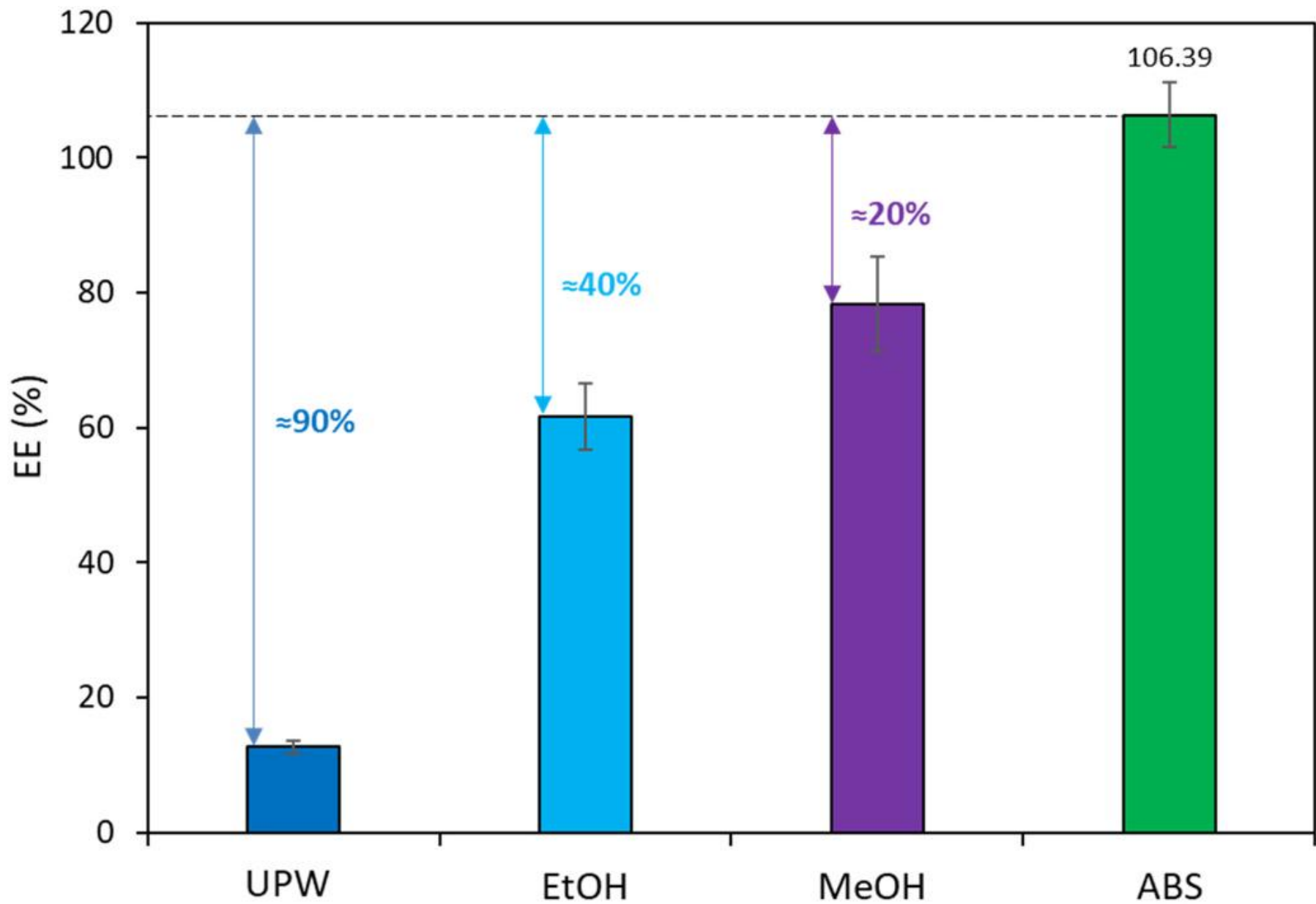


Dyeing cycle: 50 °C for 1h, using 30:1 liquor-to-fabric ratio

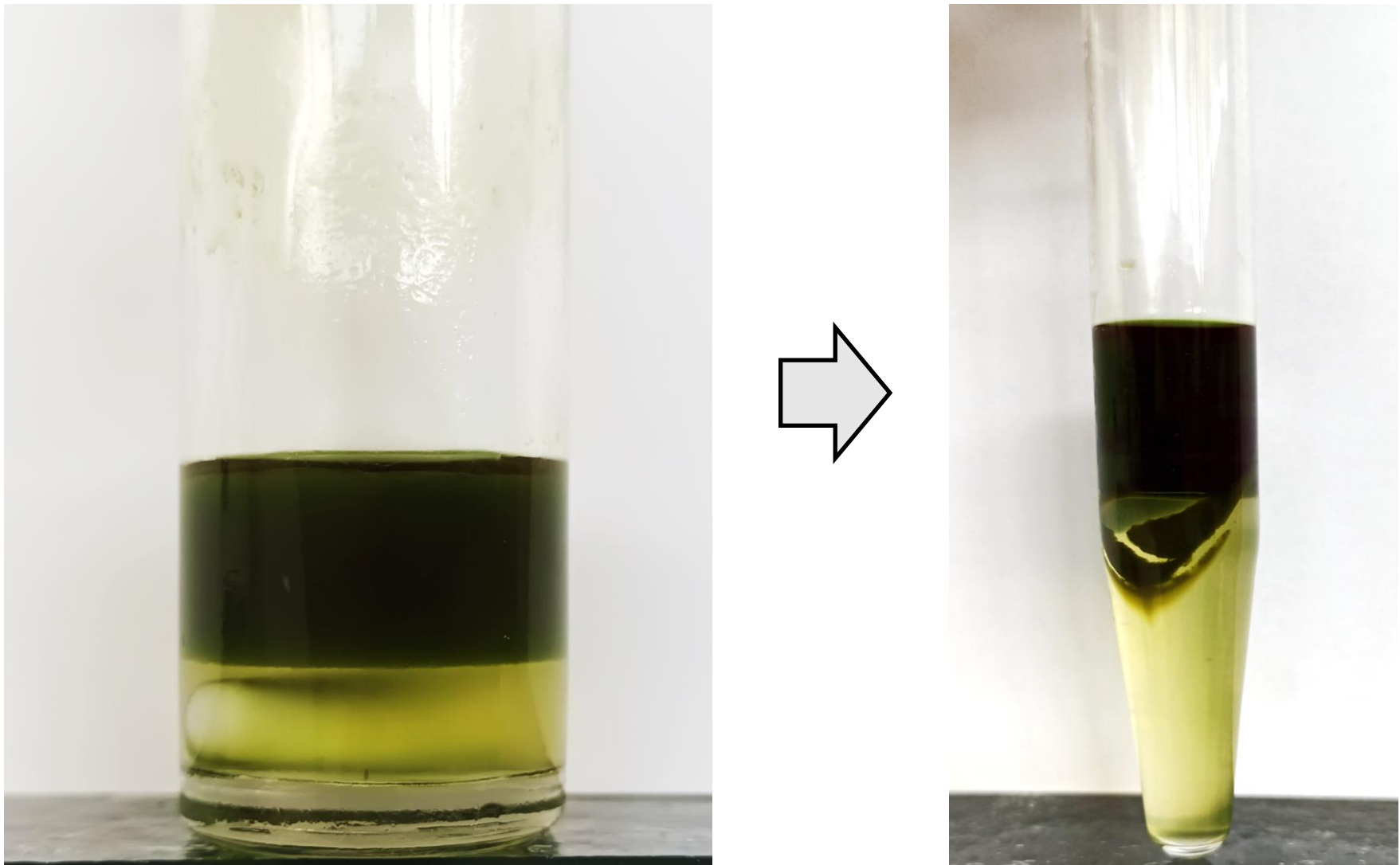


Research Highlights | Chlorophyll extraction and wool dyeing

Chlorophyll extraction efficiency (EE) – Conventional SLE vs SLLE using EtOH/NaOH ABS



Chlorophyll extraction efficiency (EE, %) using conventional solvents in the SLE processes comprised 100% EtOH, 100% MeOH, or 100% UPW, and using the selected EtOH/NaOH ABS in the SLLE process. The chlorophyll content attained for each extraction using UPW, EtOH, MeOH, or ABS were 1.00, 4.85, 6.16 and 8.37 mg of chlorophyll/gdry biomass, respectively



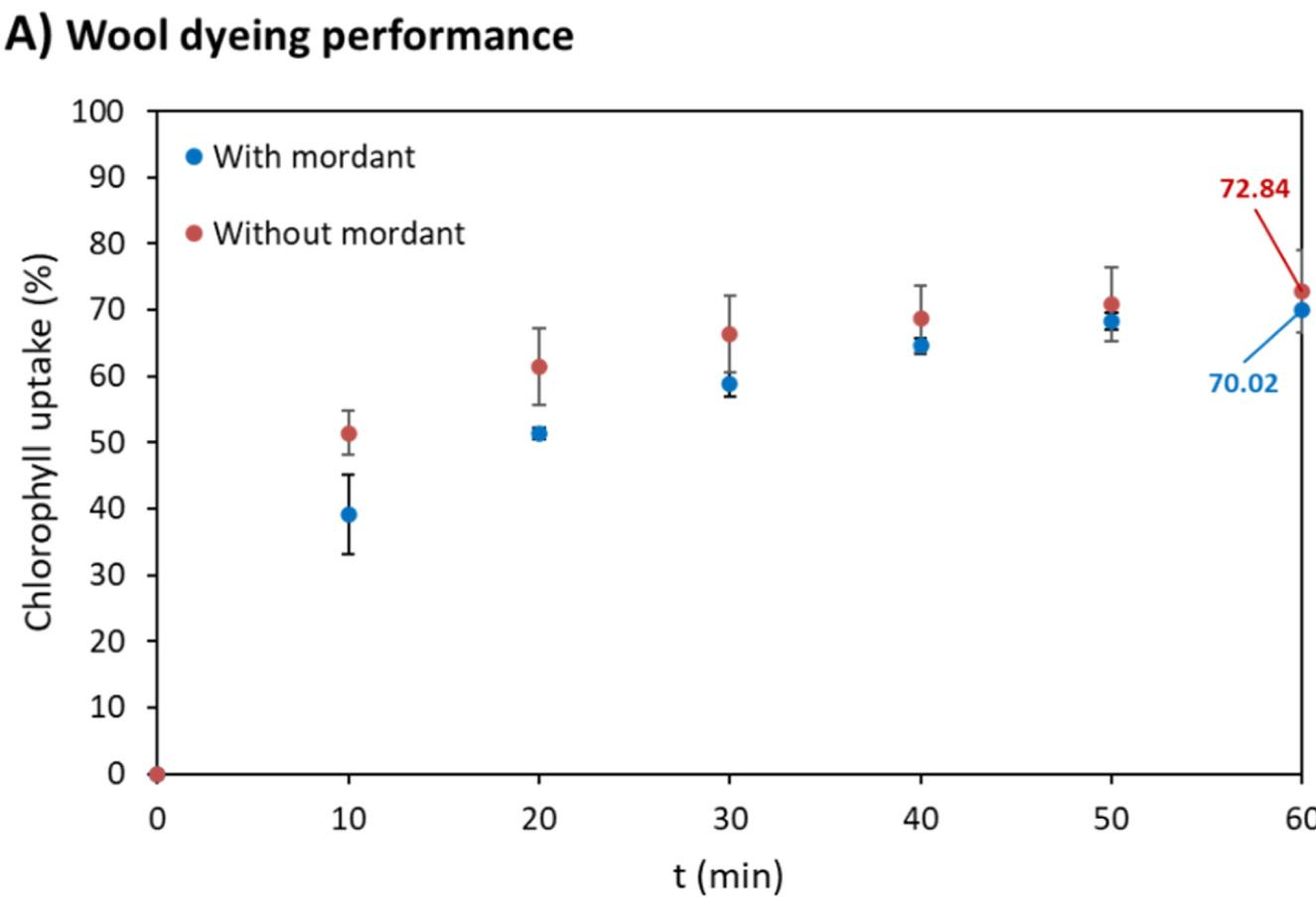
❑ EtOH/NaOH ABS outperforms conventional LLE systems



❑ Complete extraction of chlorophyll using 5× less solvent

Research Highlights | Chlorophyll extraction and wool dyeing

Wool dyeing performance – With or without mordant?

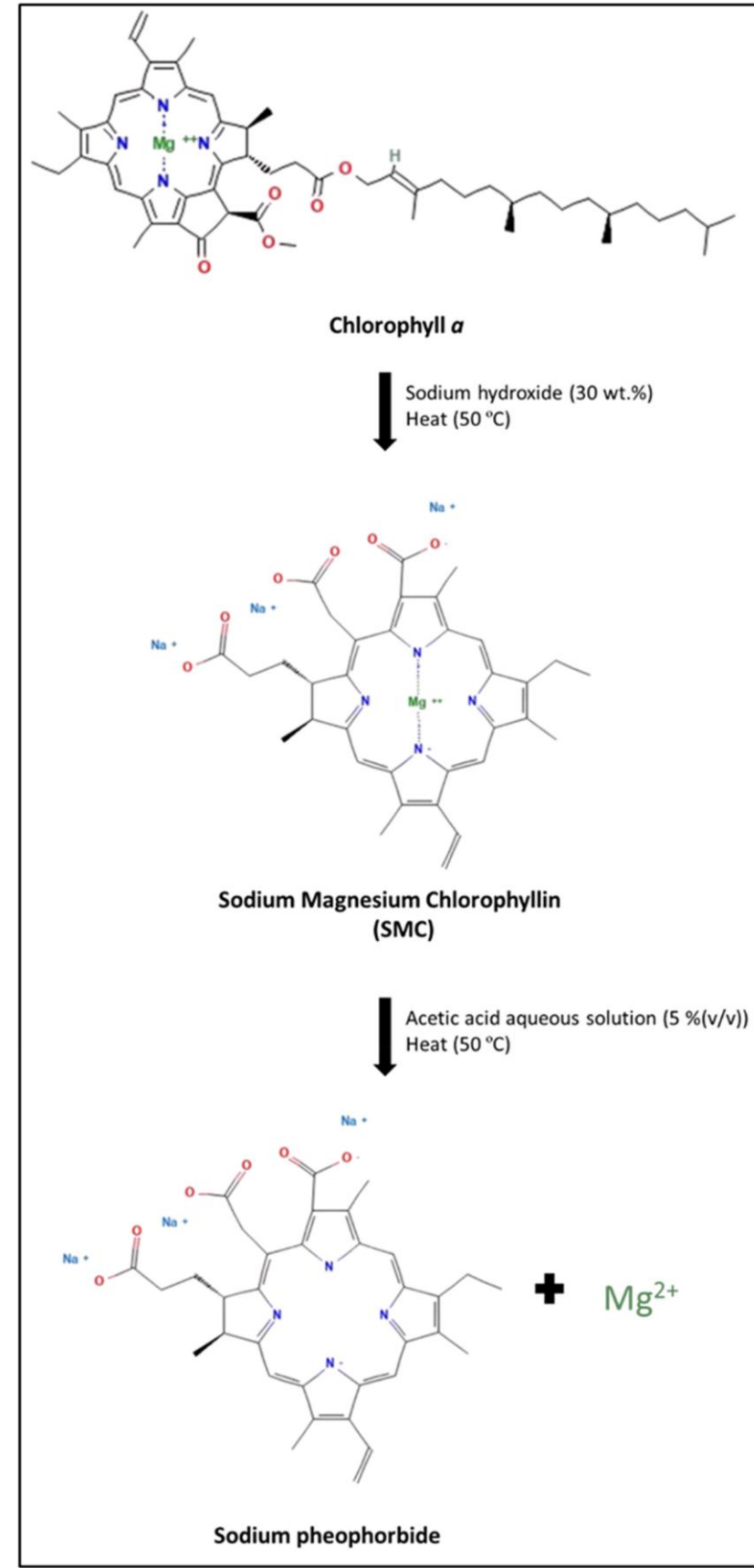


➡ Similar chlorophyll uptakes, with higher uptakes values without mordant!!!

Wool fibers absorbed ~70% of extracted chlorophyll, with unmordanted wool showing possible "self-mordanting" due to Mg^{2+} ions.

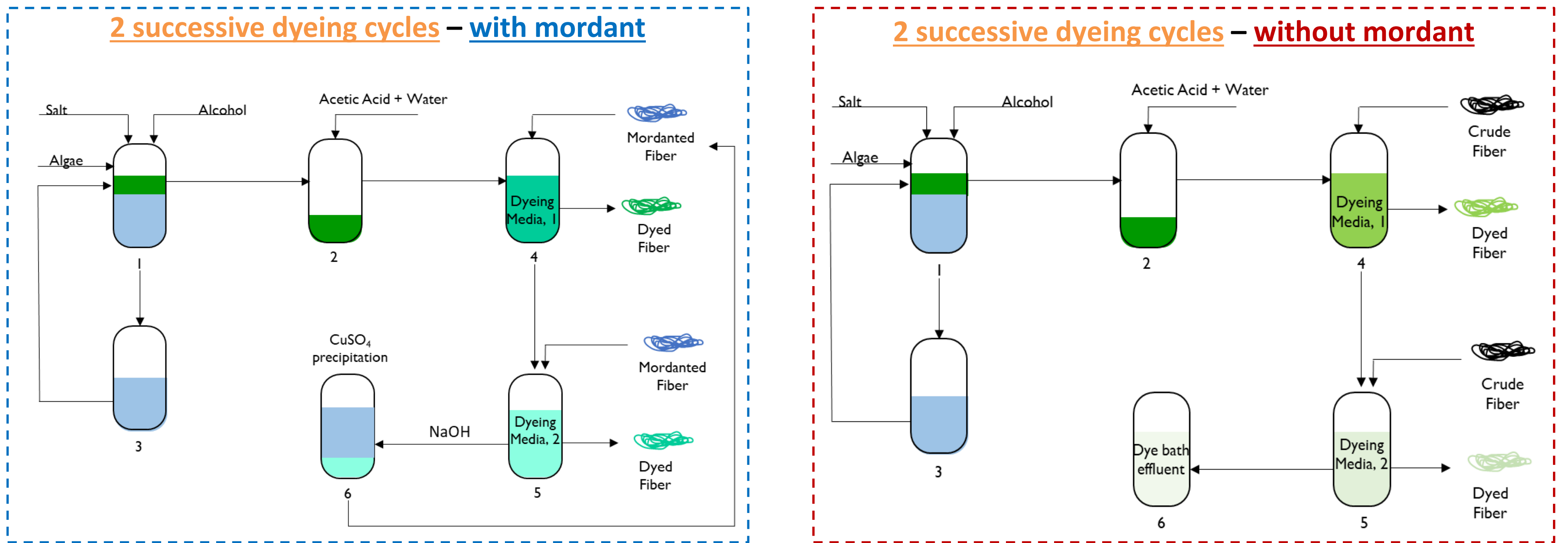


Fig.1 Chlorophyll uptake (%) of the wool fibers with and without $CuSO_4$ as mordant (A) and the visual aspect of the dyed wools (B) after one dyeing cycle (i.e., 60 min at 50 °C). The L^* , a^* and b^* colorimetric parameters of each type of wool fiber are presented at their left superior side. Undyed mordanted fibers presented L^* , a^* and b^* values of 73.71, 17.78 and 6.18, respectively.



Research Highlights | Chlorophyll extraction and wool dyeing

Circular dyeing process **with** and **without mordant**

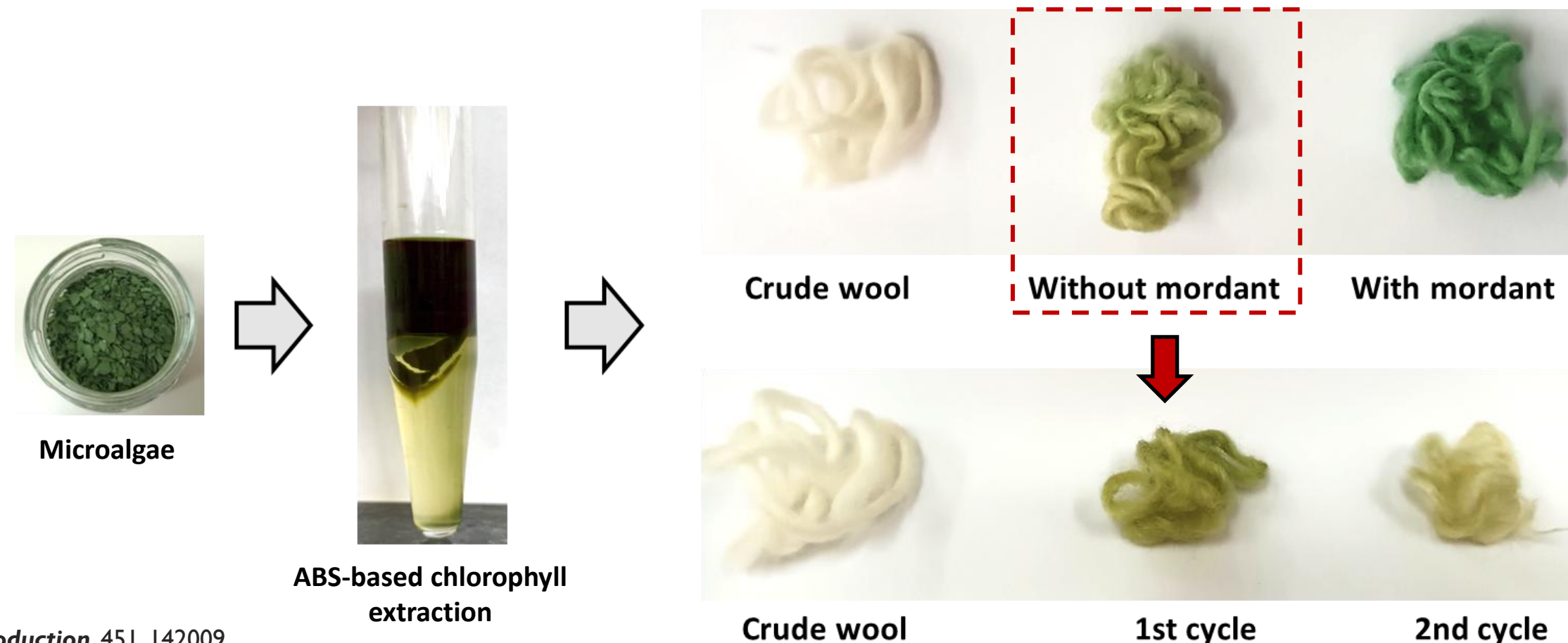


- ❑ The use of **2 successive dyeing cycles** allows:
 - ❑ Circular dyeing process using chlorophyll (natural pigment) with high chlorophyll uptakes
 - ❑ Four different colors of wool fibers are obtained (2 with mordant and 2 without mordant)
 - ❑ Dye bath effluents present low concentrations of easily treated chemicals/compounds

Research Highlights | Chlorophyll extraction and wool dyeing

Summary

- ❑ ABS outperformed conventional SLE processes, **in a single-step extraction**
- ❑ NaOH-rich phase could be reused in consecutive extraction cycles without any lost in EE
- ❑ High wool dyeing performances **with** and **without mordant**
- ❑ 98% of the CuSO_4 in effluents can be recovered and reused in new mordanting/dyeing processes
- ❑ 2 successive dyeing cycles allowed 87% of chlorophyll uptake, without mordant
- ❑ Two circular, dyeing platforms (**with** and **without mordant**) are proposed using chlorophyll extracted using EtOH/NaOH ABS
- ❑ Easily treated dyeing effluents are obtained at the end of both dyeing processes



Research Highlights | Phycocyanin-based wool dyeing

Experimental



Pre-Mordanting

To increase dye-fiber affinity.



Enzymatic Fixation

To target stable covalent bonding.



Chemical Crosslinking

To promote stronger dye-fiber interactions.

1. Mordanting Process



Untreated wool



Mordant solution
(60 °C for 60 min)

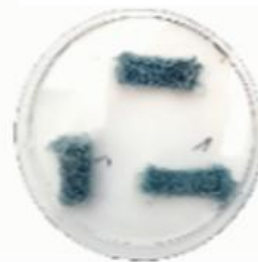


Rinse with water
(remove excess mordant)

2. Dyeing Process



Dye bath
(1 g/L of phycocyanin,
60 °C for 60 min)

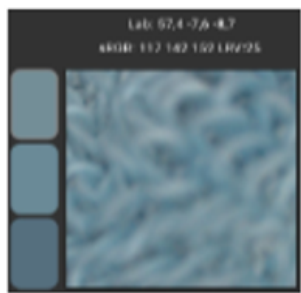


Rinse and dry
(30 °C for 120 min)

3. Data collection

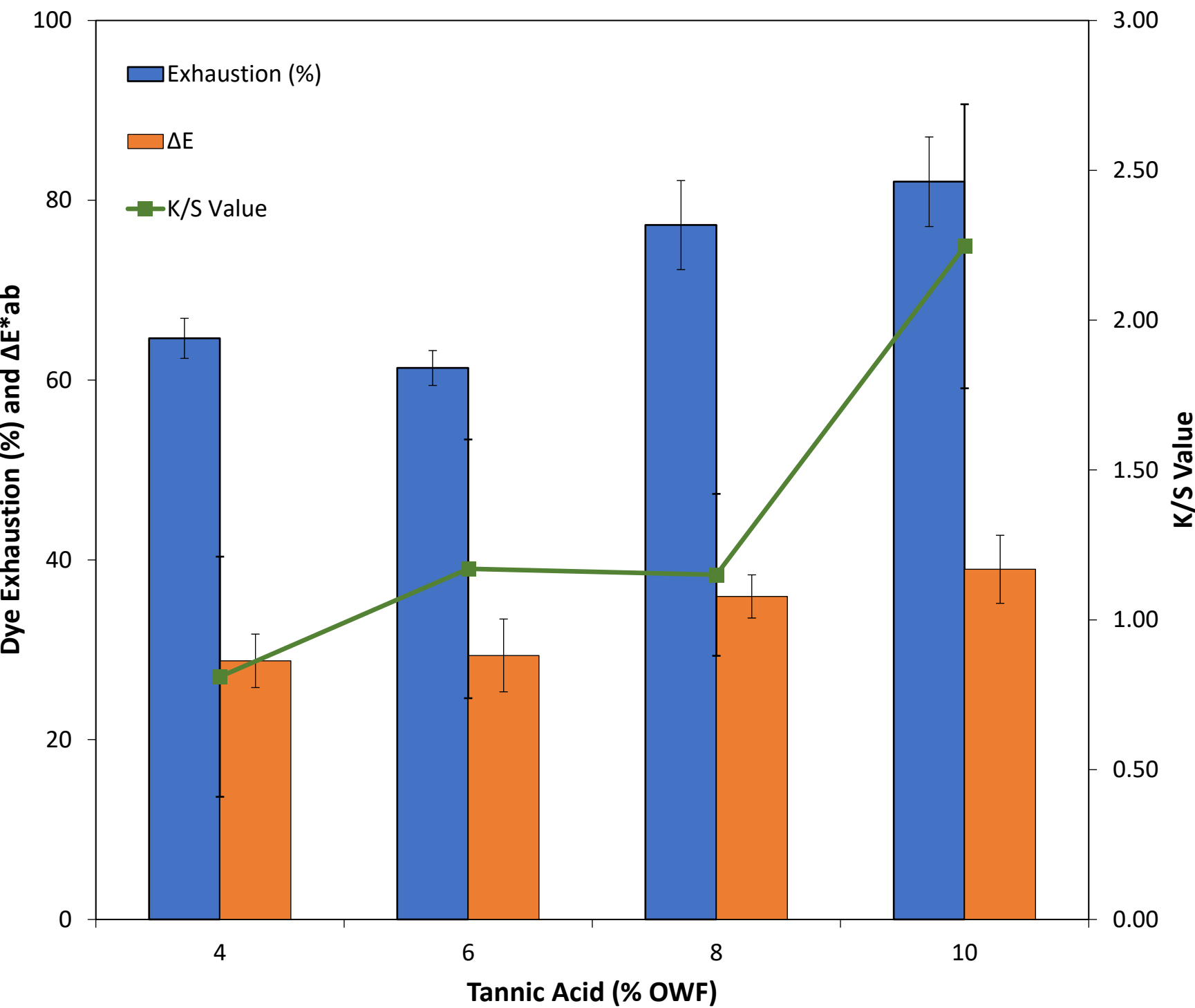


Measure dye bath
exhaustion at 615 nm



Collect colorimetric
data

Fiber Activation – Tannic Acid Pre-mordanting



Optimal Concentration: 10% OWF

EXHAUSTION
83.80%
K/S VALUE
2.25

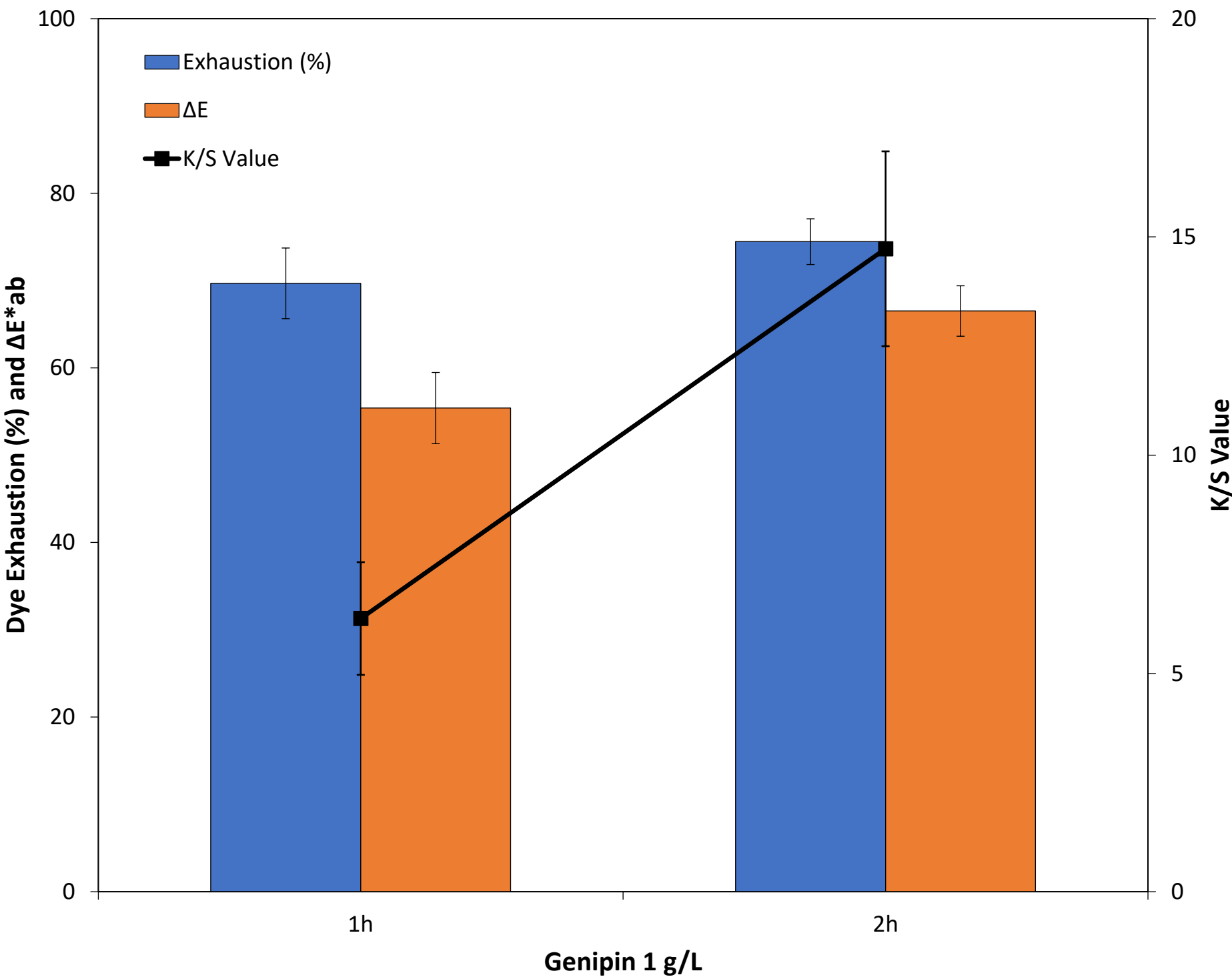
- ✓ Peak Fixation: 19% increase in exhaustion from 4% to 10% OWF.
- ✓ Visual Impact: ΔE reached 38.95 at 10% OWF of Tannic Acid.

	Untreated wool	Wool + Phycocyanin	Wool + Phycocyanin + Tannic acid
ΔE	-	13.9 ± 1.51	38.9 ± 3.78
K/S	0.07 ± 0.00	0.20 ± 0.02	2.03 ± 0.77
Dye Exhaustion	-	55.95 ± 1.72	83.79 ± 5.31
			

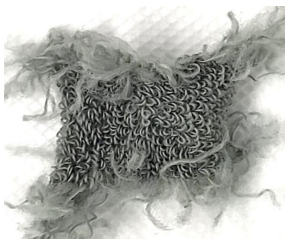
Current Limitation: Color Uniformity

Research Highlights | Phycocyanin-based wool dyeing

Chemical crosslinking - Genipin



- ❑ Genipin treatment resulted in the **highest ΔE and K/S values** achieved in this work
- ❑ Similar exhaustion values suggest color enhancement was mainly caused by genipin
- ❑ Genipin contributed simultaneously to fixation and color development

	Untreated wool	Wool + Genipin	Wool + Phycocyanin + Genipin
ΔE	-	45.5 ± 3.47	55.4 ± 4.07
K/S	0.07 ± 0.00	3.25 ± 0.93	6.26 ± 1.29
Dye Exhaustion	-	-	69.68 ± 4.04
			

Research Highlights | Phycocyanin-based wool dyeing

Main Conclusions & Findings



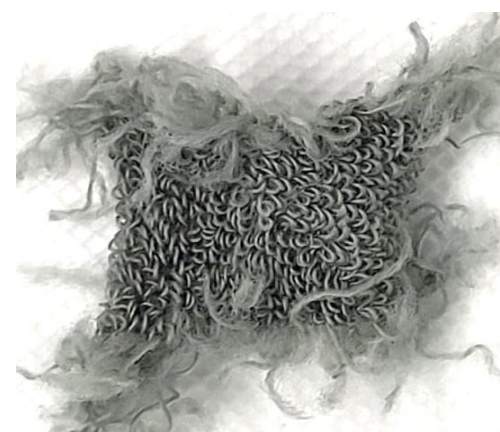
Untreated wool



Wool + Phycocyanin



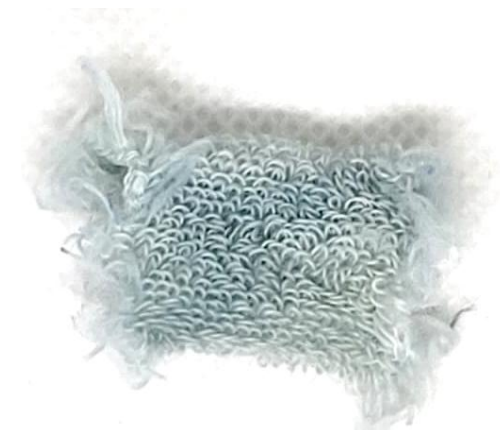
Wool + Transglutaminase



Wool + Genipin



Wool + Phycocyanin
+ Tannic acid



Wool + Dye bath +
Transglutaminase

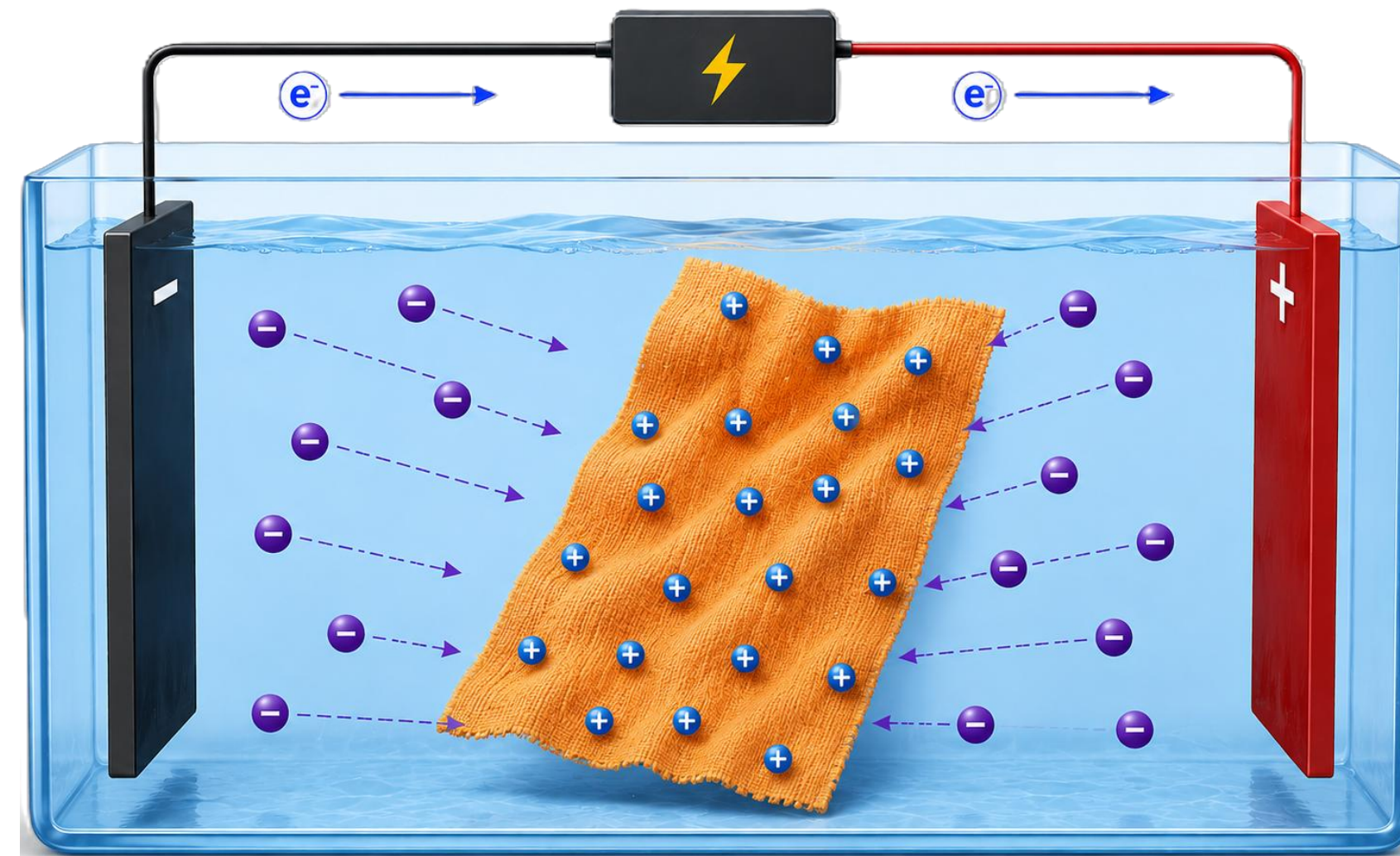


Wool + Dye bath + Genipin

- ❑ Tannic acid pre-mordanting significantly improved phycocyanin dye incorporation and color development in wool fibers.
- ❑ Transglutaminase and genipin demonstrated potential as bio-based fixation strategies through crosslinking mechanisms.
- ❑ Genipin treatment enhanced color intensity through both dye fixation and intrinsic chromophore formation.

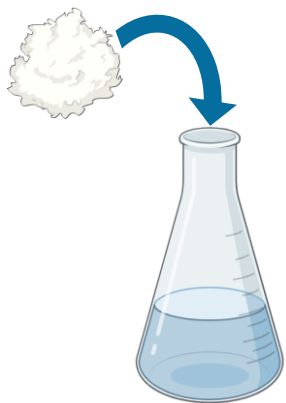


Dyeing strategy combining electric field assistance with cationised cotton fibres.



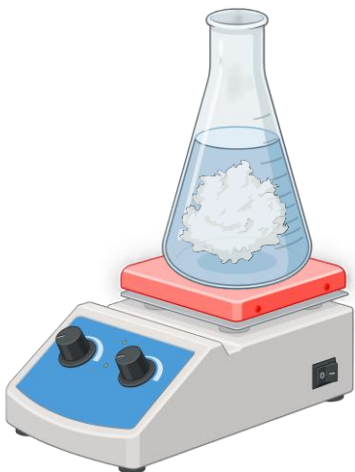
- Efficient salt-free dyeing with high dye uptake.
- Improving sustainability and resource efficiency.

Cotton cationisation process

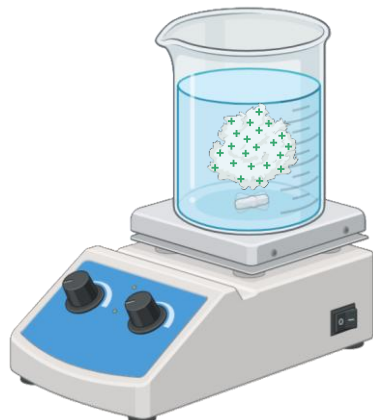


Cationisation solution

- EPTAC (10 g/L)
- NaOH (30 g/L)

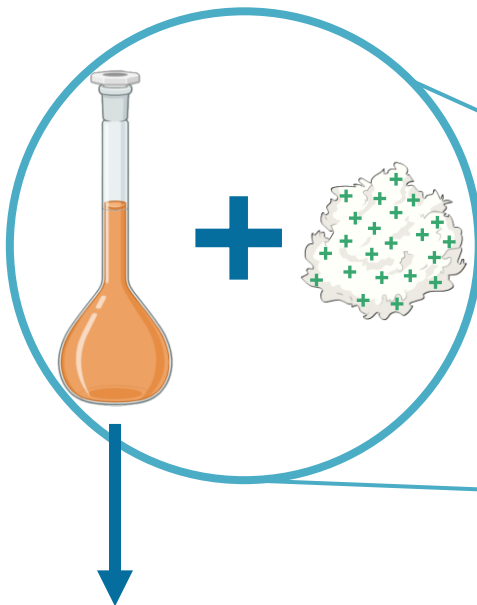


Cationisation reaction
90°C at 200 rpm for 20 minutes

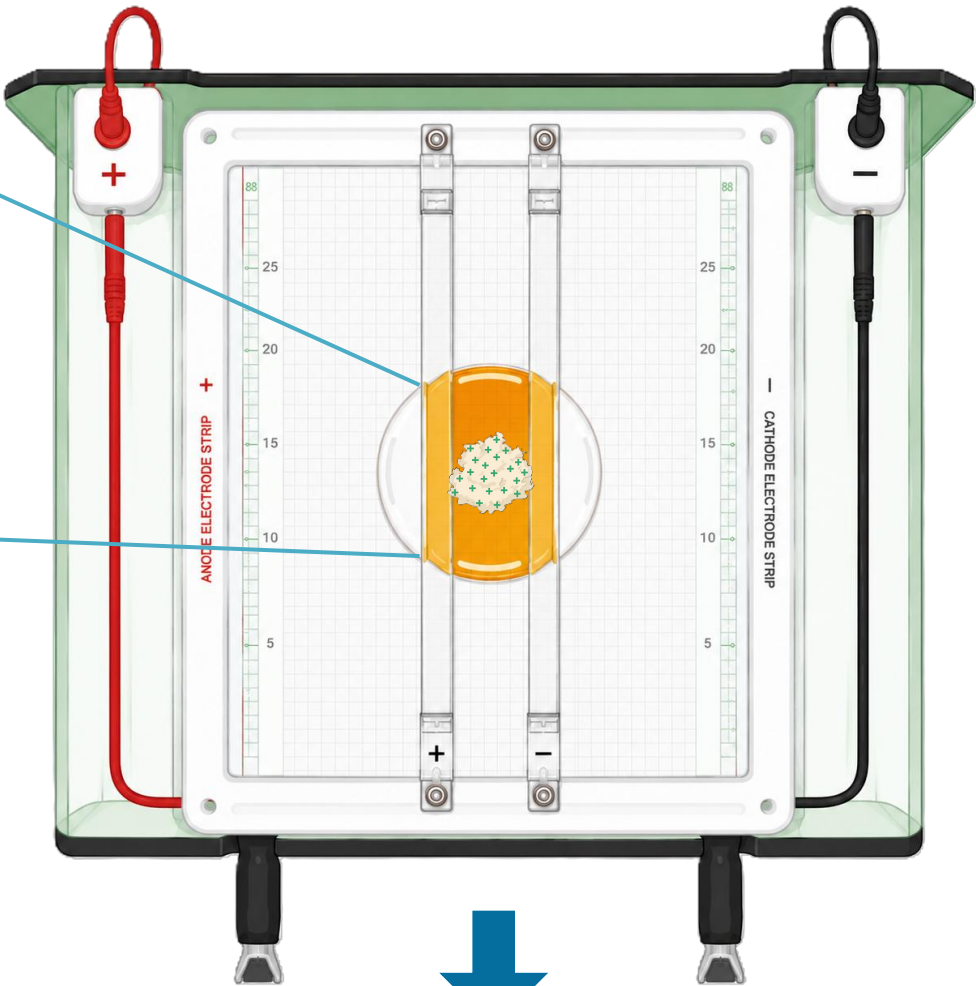


Washing
Distilled water at 200 rpm
until pH=7

Electric field-assisted dyeing

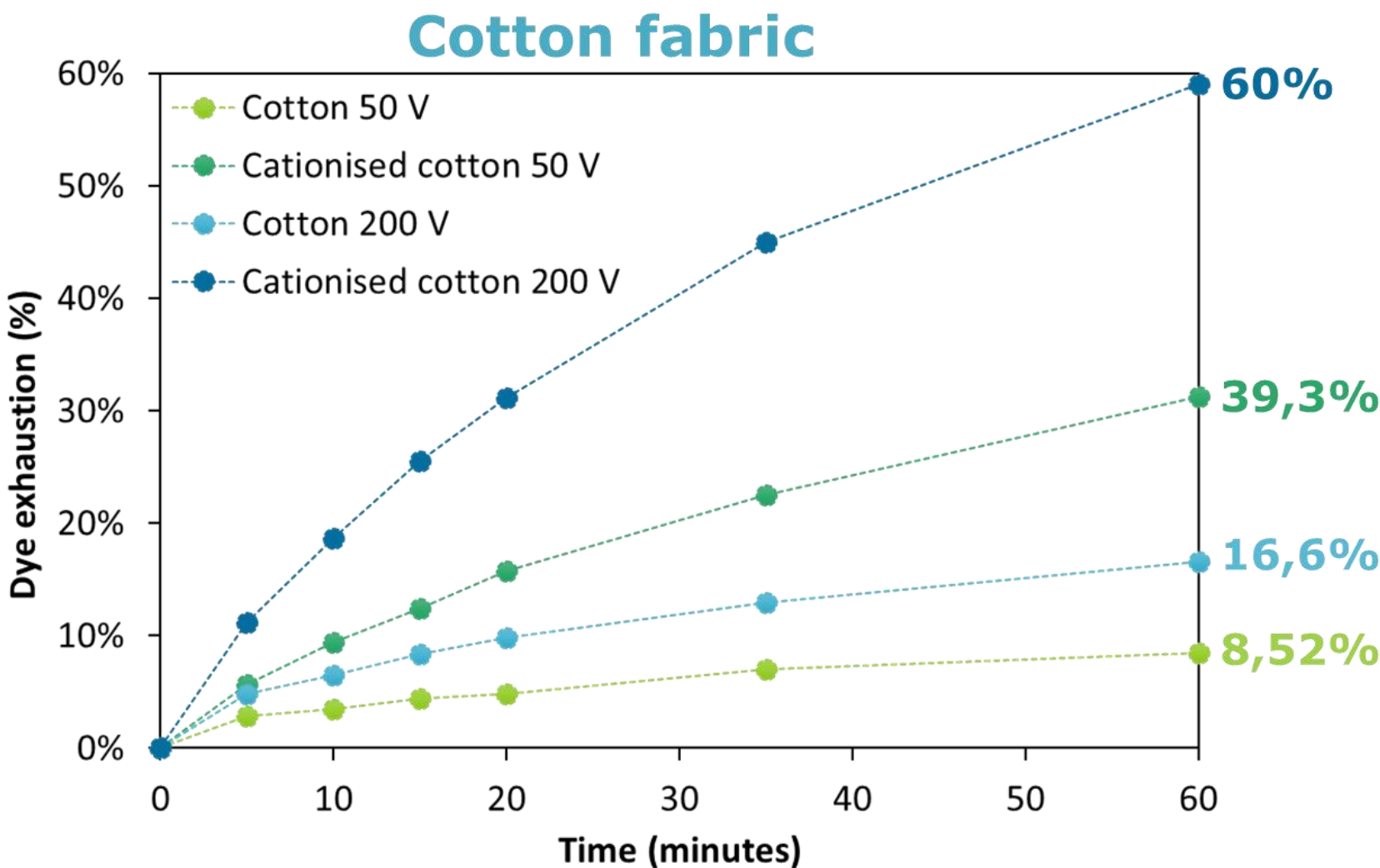
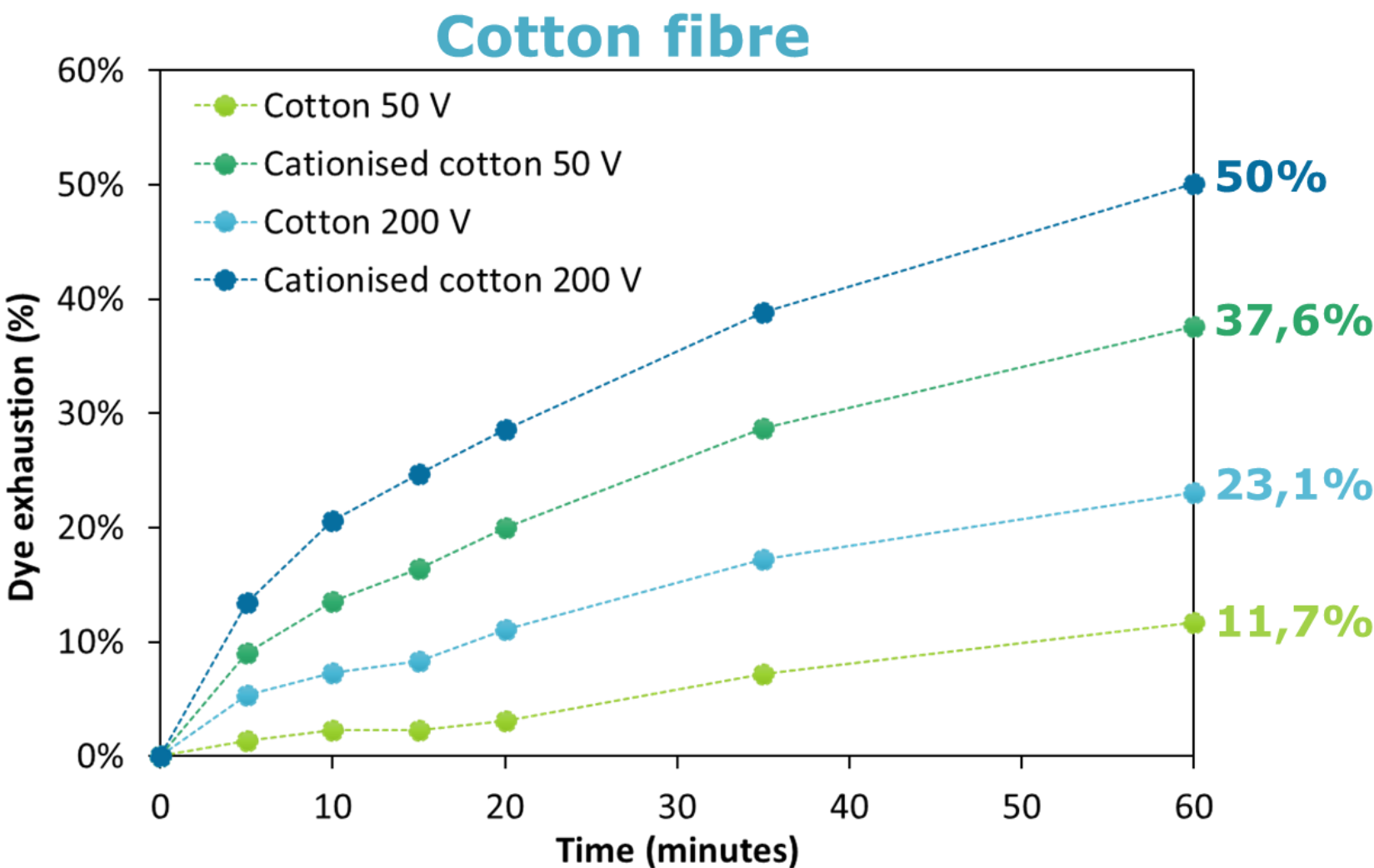


Methyl orange
was used as a
model dye.



Dyeing conditions:
Methyl Orange solution of 20 ppm
Liquor-to-fiber/fabric ratio of 1:200
Dyeing for 60 minutes, at 50 V and 200 V

Dye exhaustion kinetics



- Higher dye exhaustion for cationised cotton.
- Higher voltage enhanced dye uptake in cationised cotton.
- Cationised cotton fabric promoted better dye uptake than cationised fibre.
- Combined treatment achieved salt-free dyeing.

Cotton fibre and fabric samples

Cotton Fibre		Cotton Fabric	
Untreated	Cationised	Untreated	Cationised
50 V		50 V	
			
200 V		200 V	
			



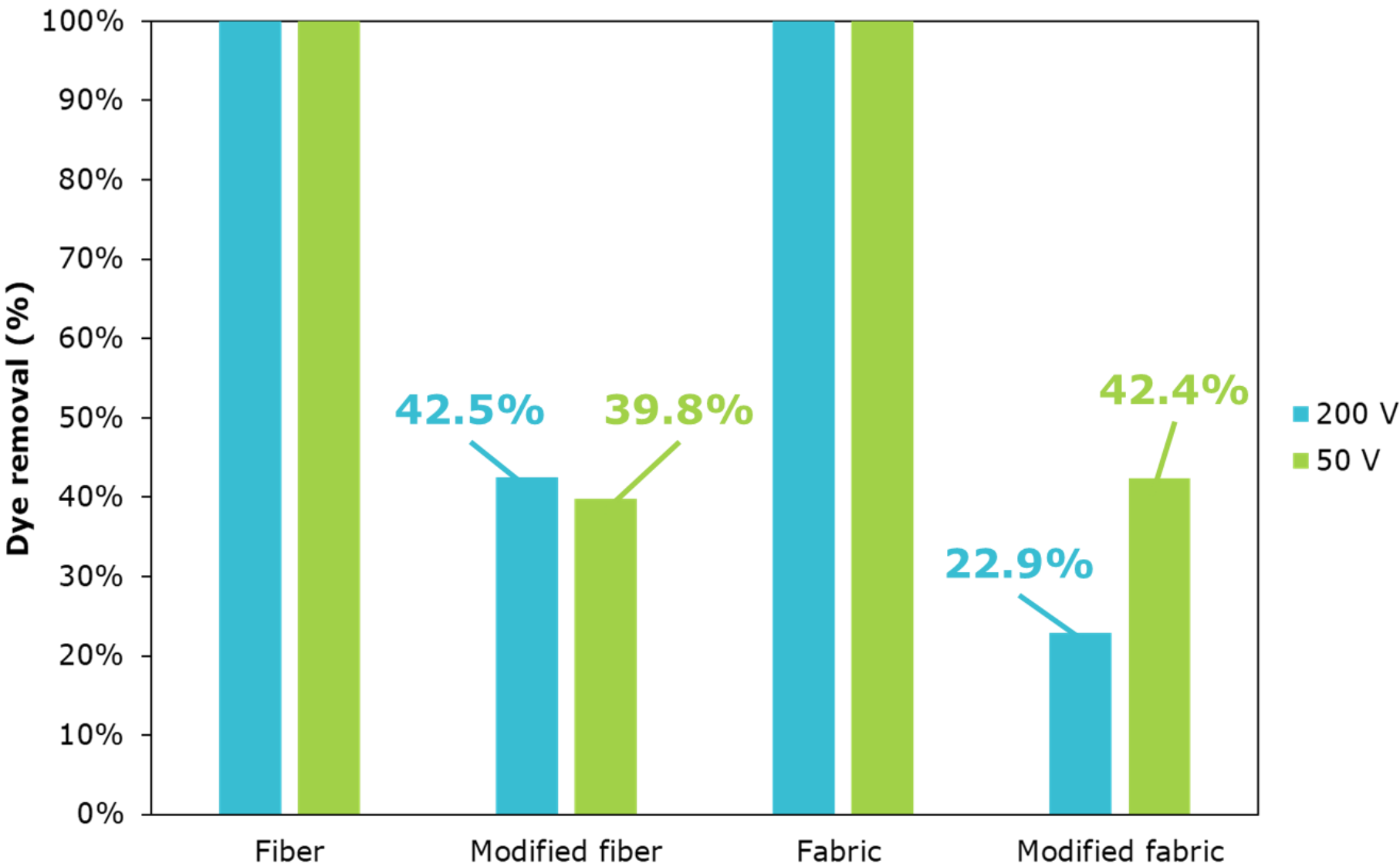
- Cationised samples exhibit similar colour intensity.
- More vibrant and homogeneous coloration at higher voltage.



- ✓ Cationisation enhances dye uptake.
- ✓ Electric field improves dye distribution and levelness.

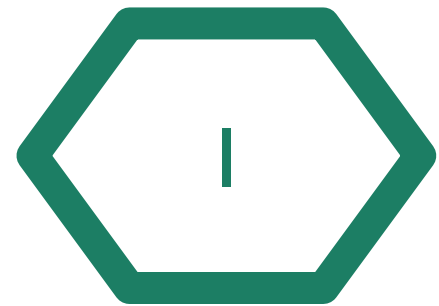
Cotton fibre and fabric samples – washing fastness

Cotton Fibre		Cotton Fabric	
Untreated	Cationised	Untreated	Cationised
50 V		50 V	
			
200 V		200 V	
			



- Untreated cotton samples show complete dye leaching for all samples.
- Cationised cotton samples still show robust and homogeneous coloration.
- Higher voltage allowed less dye leaching for cationised cotton fabric.

Summary



Different fibers have different chemical composition, dye affinity and dyeing performance



Understanding dye-fibre interactions is crucial for an efficient and durable textile product



DyeLoop™ is a key example of how this understanding can improve the sustainability and circularity of the textile industries



Integration between innovative dyeing strategies and deeper understanding of fibre-natural dye interactions is crucial for promoting more sustainable textile products



Process intensification and modification can significantly enhance the sustainability and competitiveness of textile industries

The acknowledgements

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Thank you for your attention!!!!