Undyeing jeans with Acetone (Project: Jeans to Sweater)

Ministerie van Economische Zaken en Klimaat

MNEXT MATERIALS & ENERGY X TRANSITION

Goal

The fashion industry heavily strains natural resources; for example, producing one cotton T-shirt uses around 2,700 liters of water(1). By developing more effective and sustainable dye removal methods, this work aims to improve fabric recyclability and reduce environmental burdens associated with textile waste and production.

To support this textile recycling, this project compares extraction techniques such as heated solvent extraction, ASE, and supercritical CO_2 to remove indigo dye from denim.

Methods

1. Solvent Screening (Small-Scale):
Test various solvents (e.g., acetone, ethanol) in small vials with denim pieces on a heating plate to assess extraction potential.

2. Best Solvent Selection:
Analyze extracted samples using UV-Vis spectroscopy to compare dye removal efficiency and identify the most effective solvent.

3. Calibration Curve Creation:
Prepare a concentration series of indigo in the selected solvent and generate a calibration curve using UV-Vis to quantify extraction results.

4. Accelerated Solvent Extraction (ASE):
Use ASE to test solvent performance under high temperature and pressure with layered denim and diatomaceous earth in pressurized cells.

5. Supercritical CO_2 Extraction:
Employ supercritical CO_2 with ethanol as co-solvent under controlled temperature and pressure to evaluate indigo extraction on denim.

6. Color Measurement with LAB Meter:
Measure color strength (ΔE) on denim before and after treatment to quantify dye removal in CIELAB color space.

Background

Indigo (Dye)

- Indigo has two carbonyl groups (-C=O) and two secondary amines (-NH)(figure 1), which typically gives polarity to the molecule(2).
- Mostly non-polar due to electron delocalization, despite having polar groups(2).
- It forms only weak bonds with the polar cotton fibers.

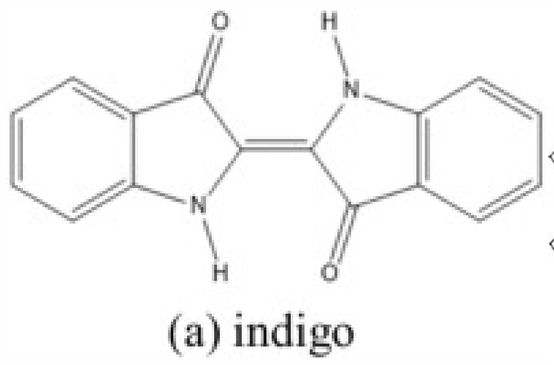


Figure 1: Molecular structure of indigo

Cotton

- Made of cellulose; highly polar with many hydroxyl (-OH) groups(figure 2)(2).
- Hydrophilic and swells ~11% in water.(3)
- Strong hydrogen bonding affecting solvent interaction.

.

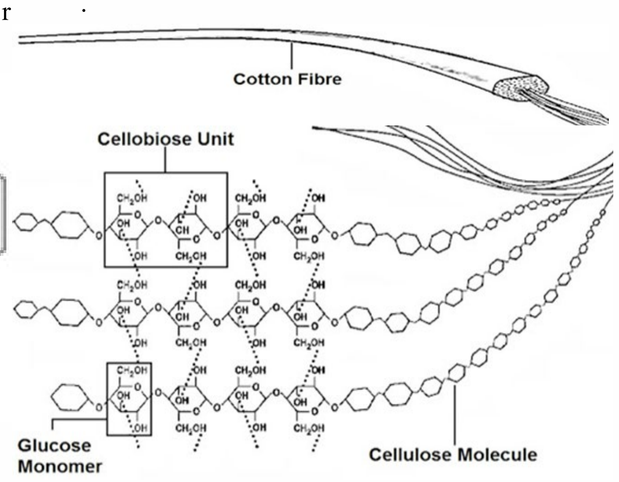


Figure 2: Cellulose-Monomer for Cotton Fiber

.

Solvents

Deep Eutectic Solvent (DES)

- Mix of thymol + decanoic acid creates hydrophobic DES.
- Forms hydrogen bond networks that enhance non-polar indigo solubility(10).

Ethanol

- Moderately polar (~6/10); interacts with cotton but limited for indigo(4)(5).

Acetone

- Polar aprotic (~5/10); better at dissolving indigo, minimal cotton interaction(6)(7).

Tert-Butyl Alcohol

- Slightly polar (~4/10); aids indigo solubility and cotton penetration(8).

Extraction Techniques

Supercritical CO_2 Extraction

- CO_2 above critical temp/pressure behaves like both gas and liquid.
- Efficient for extracting non-polar dyes like indigo.
- Co-solvents (e.g., ethanol) help extract more polar dyes.

(11)

Accelerated Solvent Extraction (ASE)

- Applies high temperature and pressure to force solvent through fabric.
- Increases dye solubility and extraction rate.
- Efficient for both polar and non-polar dyes; uses less solvent than traditional methods.

(9)

Jeroen Valkenburg
Project/Research Group: Biobased Building Blocks and Products
Contact information: jeroenvalk2702@hotmail.com
Date: 12 June 2025

Results

Acetone was found to be the most effective solvent for indigo extraction, outperforming all other tested solvents, including ethanol, a tert-butyl alcohol mixture, and DES.

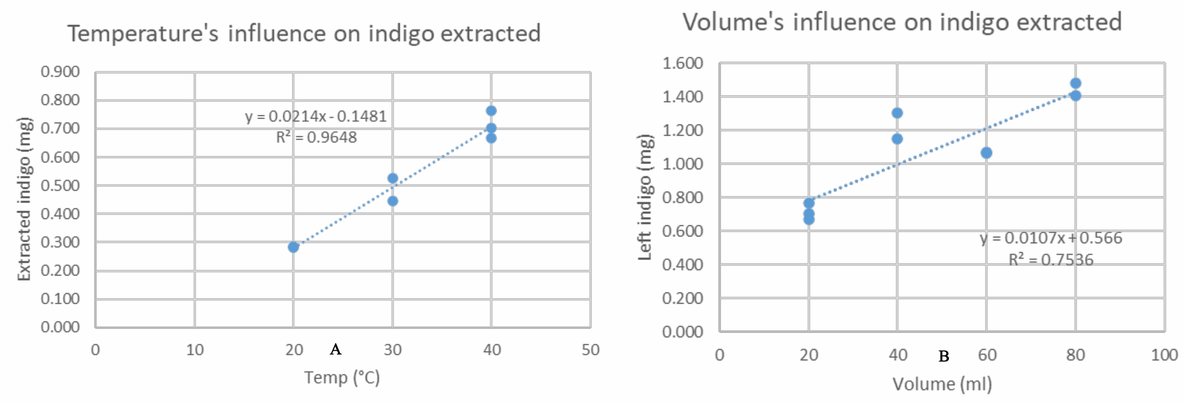


Figure 3: (A) Temperatures influence on the amount of indigo left in a jean, (B) Volume's influence on the mount of indigo left

Figure 3 consists of two graphs showing the influence of temperature (A) and solvent volume (B) on the amount of indigo extracted from denim using acetone. In graph A, the trendline equation is $y = 0.0214x - 0.1481$ with an R^2 value of 0.965, indicating a strong positive correlation between temperature and extraction efficiency. Graph B shows the effect of volume with a trendline of $y = 0.0107x + 0.566$ and an R^2 value of 0.754, suggesting a moderate positive relationship. Together, these results highlight how both factors contribute to indigo removal.

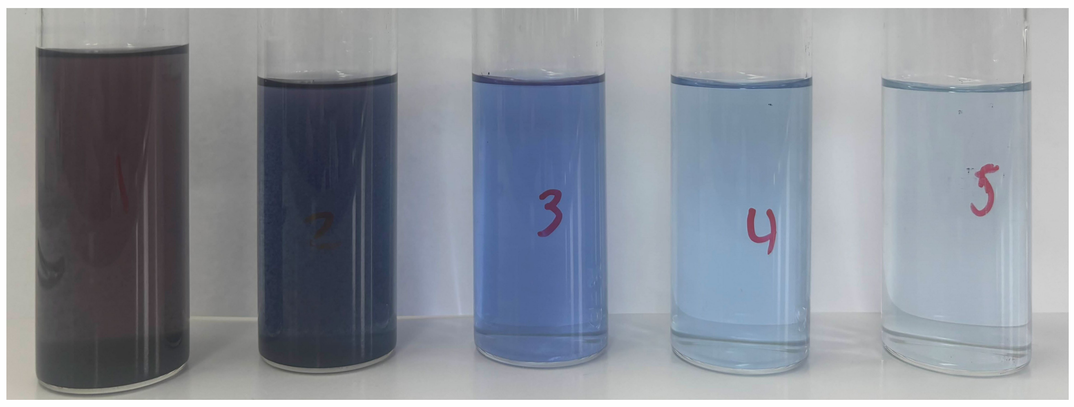
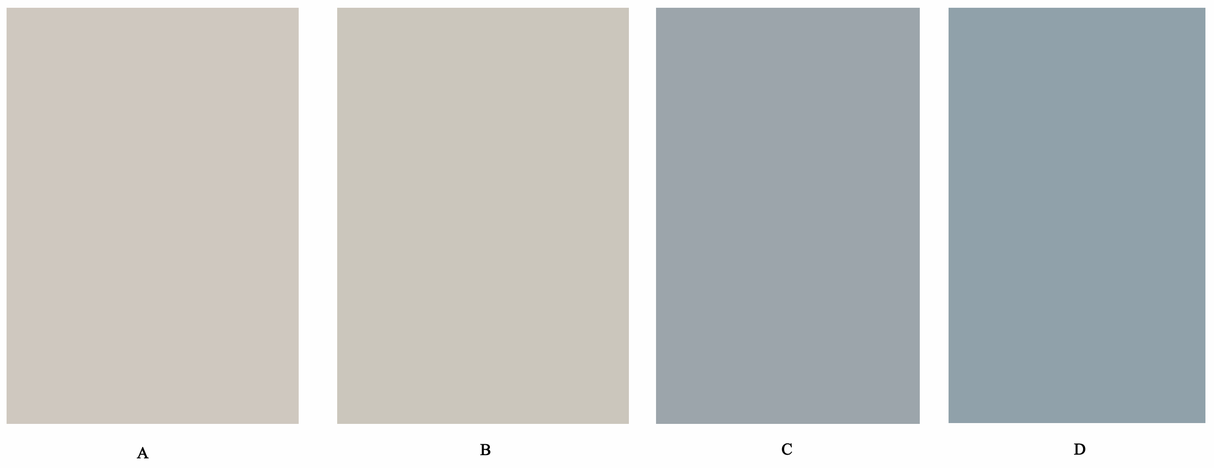


Figure 4: Indigo-rich acetone solutions from sequential ASE rinses (approx. 30 mL per bottle).

An ASE system was used to test acetone under intense conditions with a 5 g denim sample subjected to five rinses, with data shown in Figure 4. After four rinses totaling 106.5 mL, 3.393 mg of indigo was removed, resulting in near-complete decoloration and a lab measurement of ΔE 16.18. The sample, including a duplicate of the experiment, can be seen below.



Samples A & B: Denim pieces treated five times with acetone under ASE conditions (120 °C, 110 bar, 1 hr).
Samples C & D represent before of sample A and B.

The images above display denim samples before and after. Samples C & D show the original, untreated denim, while Samples A & B depict the same fabric after undergoing five rinses at 120 °C and 110 bar for one hour each. The visible contrast highlights the effectiveness of the ASE method in removing indigo dye.

Conclusion

Acetone proved the most effective solvent for indigo extraction, removing up to 3.4 mg from 5 g denim with ΔE values over 16 (see samples A & B). Its strong performance and recovery potential make it a top candidate for sustainable dye removal.

References

1. European Parliament. (2024, Mar 21). The impact of textile production and waste on the environment (infographics).
2. Nazim Sarwar. (2025, Jan 19). The Chemistry behind Indigo Back Staining !
3. Shivam Salokhe, , Mohammad Rahmati, Ryan Masoodi & Jane Entwistle. (2023). The effect of fibre swelling on fluid flow in cotton fabrics: An experimental study. Sciopen.
4. Ethanol . Pub Chem. https://pubchem.ncbi.nlm.nih.gov/compound/Ethanol
5. Ethanol Dielectric Constant . Springer Materials. https://materials.springer.com/thermophysical/docs/dec_c11
6. Acetone . Pub Chem. https://pubchem.ncbi.nlm.nih.gov/compound/Acetone
7. Acetone Dielectric Constant . Springer Materials. https://materials.springer.com/thermophysical/docs/dec_c4
8. Tert-Butyl Alcohol . Pub Chem. https://pubchem.ncbi.nlm.nih.gov/compound/Tert-Butyl-Alcohol
9. Dr.Anupam Sharma, Dr. R.P.Mathews, & Dr.Manoj M.C.Accelerated Solvent Extractor (ASE). https://www.bsip.res.in/old/pdf/SAIF/1.%20ASE%20-Anupam%20Sharma.pdf
10. Aoonboontum, P., Thong-on, P., Wisidsri, N., Thugmangmee, S., Rungsang, T., Khorana, N., & Srivilai, J. (2025). Green extraction of natural indigoid from Baphicacanthus cusia (Nees) Bremek using hydrophilic and hydrophobic deep eutectic solvent technology. Next Sustainability, 5, 100090. 10.1016/j.nxsust.2024.100090
11. Clifford, A. A., & Williams, J. R. (2000). Introduction to Supercritical Fluids and Their Applications. In J. R. Williams, & A. A. Clifford (Eds.), Supercritical Fluid Methods and Protocols (pp. 1–16). Humana Press. 10.1385/1-59259-030-6:1