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HYDROLYSIS OF ZEOLITE-TANNED LEATHER SHAVINGS

VS

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What is a Zeolite?

- Zeolite = family of crystalline microporous minerals
- Mainly Aluminium, Silicon, Oxygen

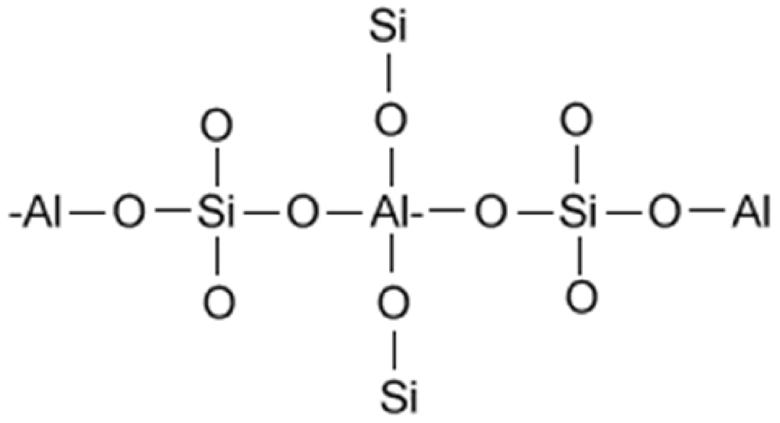

Figure 1: Basic Zeolite Structure

What is a Zeolite?

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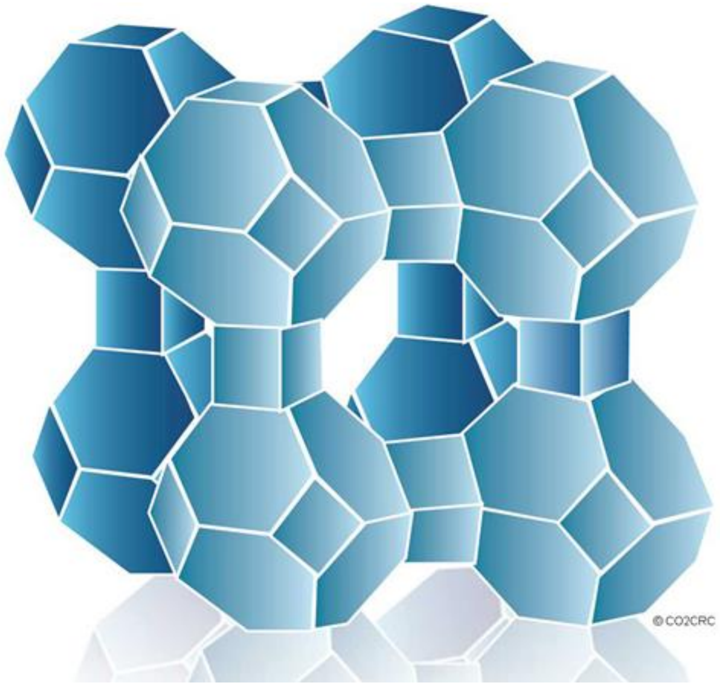
- Mainly Aluminium, Silicon, Oxygen
- Forms 3D aluminosilicate frameworks (cage-like structure)



What is a Zeolite?

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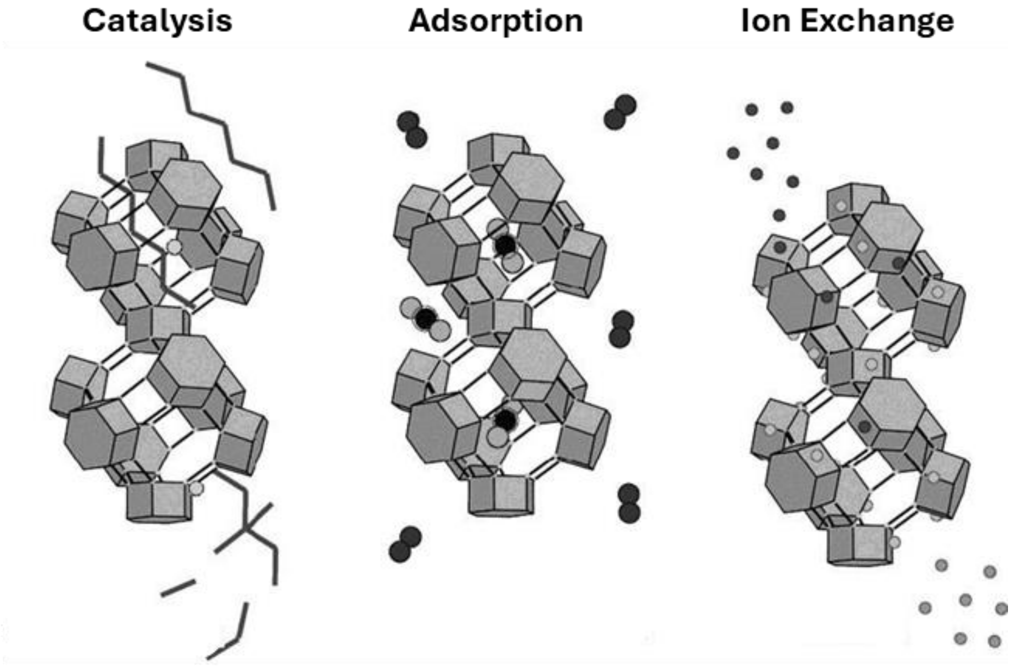
- Mainly Aluminium, Silicon, Oxygen
- Forms 3D aluminosilicate frameworks (cage-like structure)
- Porosity key to tanning interaction



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What is Zeolite Tanning?

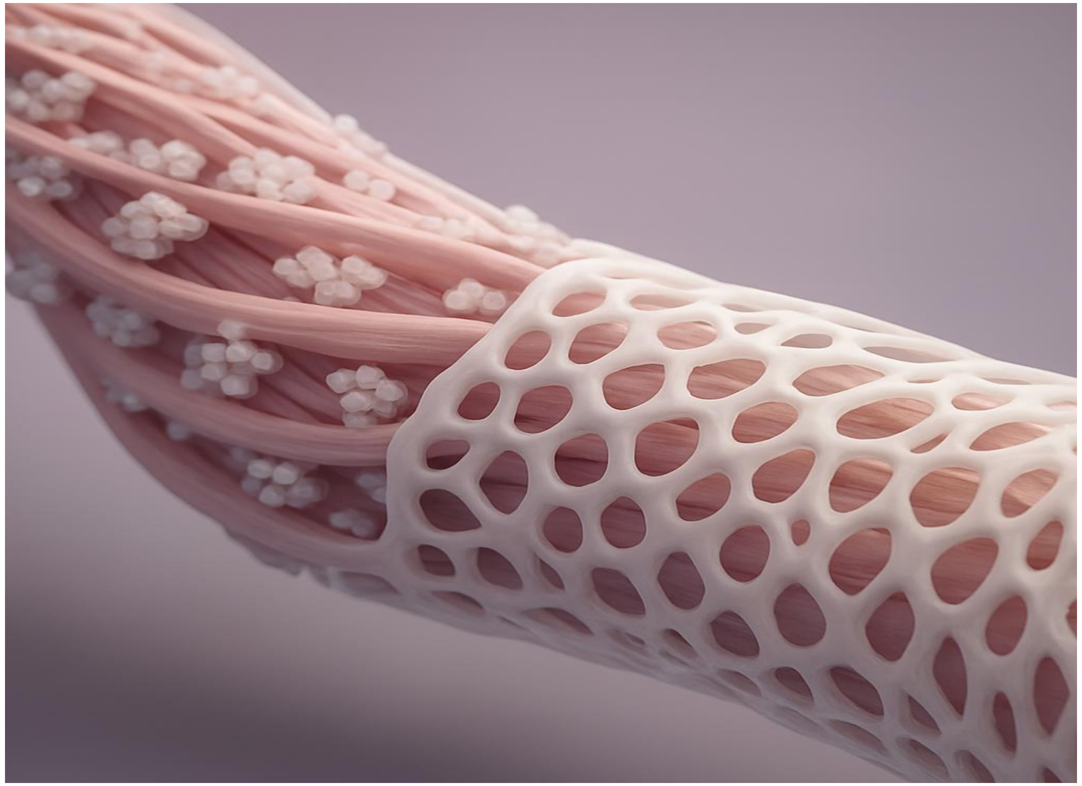
Chrome-free process

- Uses zeolite minerals instead of chromium salts.
- Non-toxic, environmentally benign, and chemically stable.
- Produces cleaner, heavy-metal-free effluent.

What is Zeolite Tanning?

How it works?

- Zeolite forms a 3D sheath around collagen fibres
- Sheath stabilises fibres via electrostatic interactions and some covalent bonds
- No chemical crosslinks like in chrome tanning.



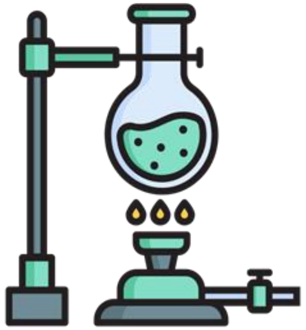
Research Objectives?

1. **Optimise alkaline hydrolysis** → refine the process to achieve complete breakdown of zeolite-tanned leather shavings.

2. **Quantify zeolite retention** → assess how much tanning agent remains in hydrolysed and unhydrolysed fractions.

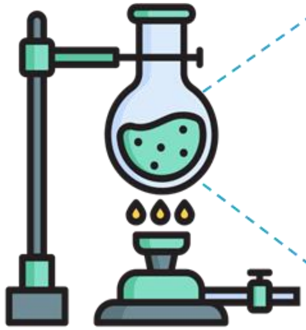
3. **Assess hydrolysate purity** → determine the proportions of collagen (organic) and residual minerals (inorganic) to evaluate suitability for industrial applications.

Methods at a glance?



Alkaline Hydrolysis
(0.1M NaOH at 90 ℃ for 24hrs)

Methods at a glance?



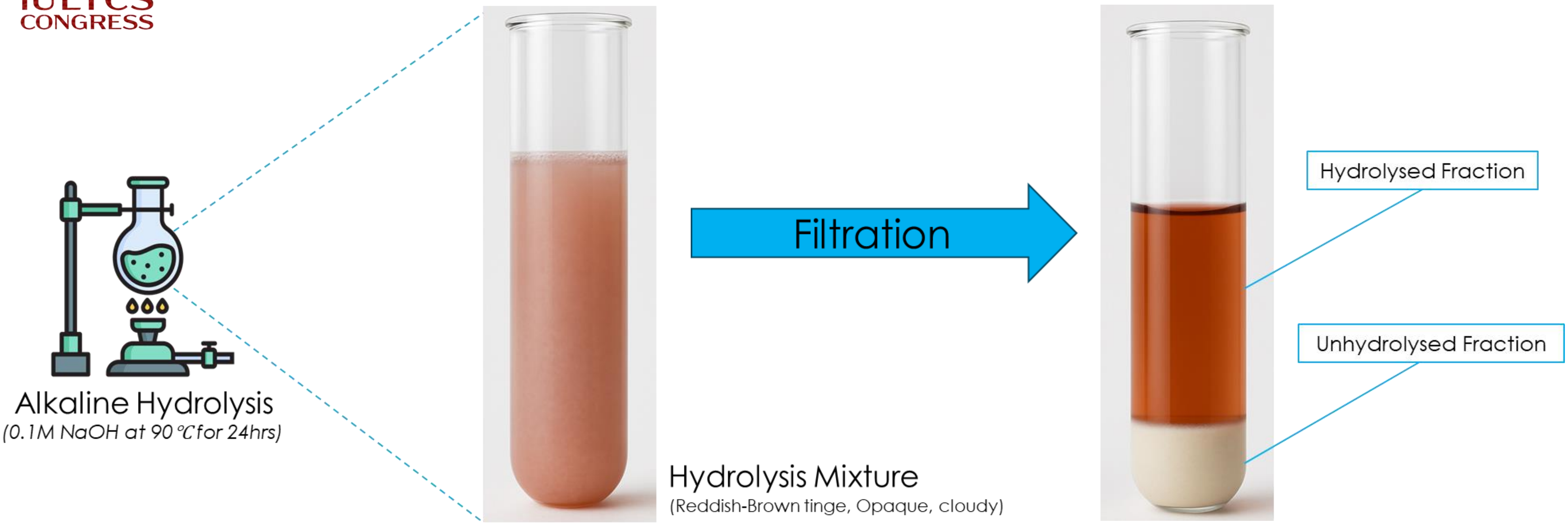
Alkaline Hydrolysis

(0.1M NaOH at 90 °C for 24hrs)

Hydrolysis Mixture

(Reddish-Brown tinge, Opaque, cloudy)

Methods at a glance?



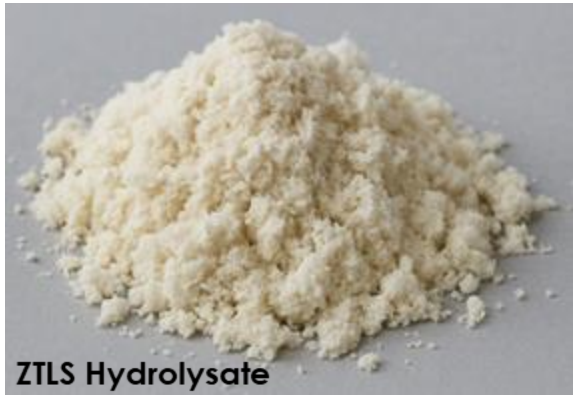
Methods at a glance?

Hydrolysed Fraction → Drying

Unhydrolysed Fraction → Drying

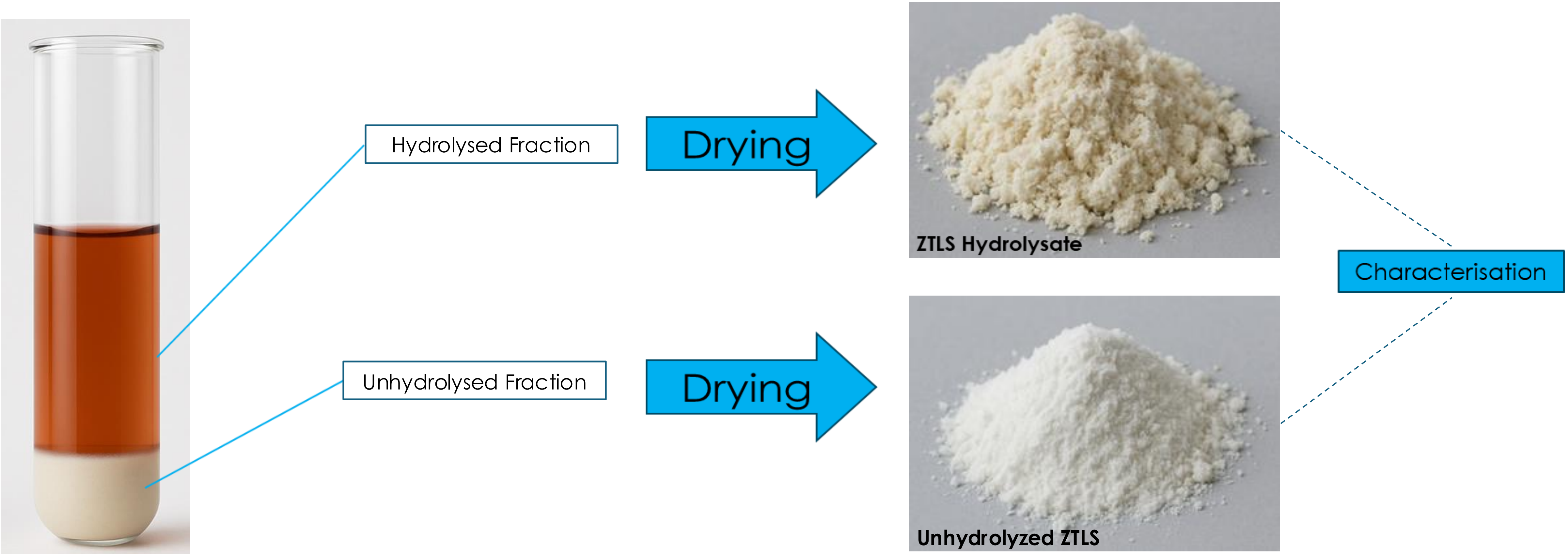
Methods at a glance?

Hydrolysed Fraction → Drying →



Unhydrolysed Fraction → Drying →

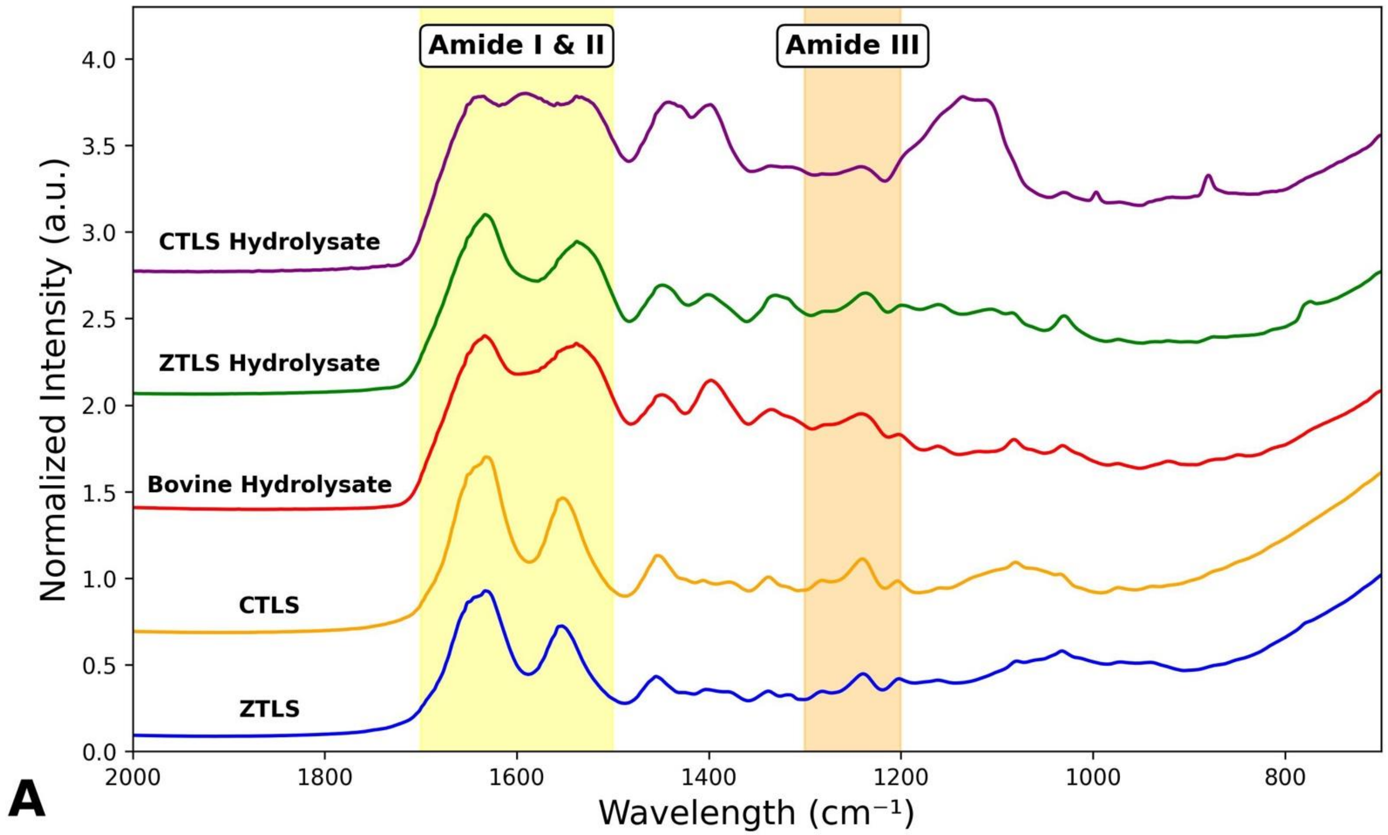
Methods at a glance?



Characterisation?

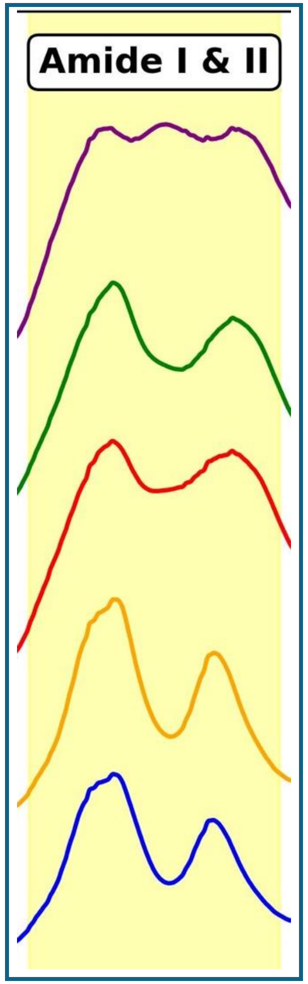
1. FTIR (Fourier-Transform Infrared Spectroscopy)
2. ICP-MS & AAS (Elemental Analysis)
3. SEC (Size-Exclusion Chromatography)
4. LC (Liquid Chromatography)

FTIR

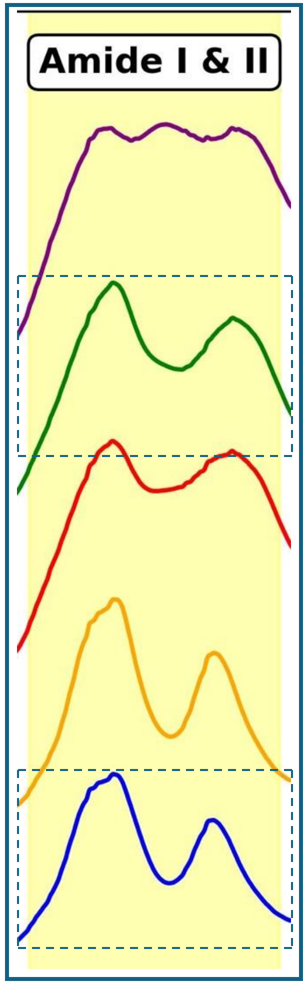


Amide I & II
Amide III
CTLS Hydrolysate
ZTLS Hydrolysate
Bovine Hydrolysate
CTLS
ZTLS
Normalized Intensity (a.u.)
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
2000
1800
1600
1400
1200
1000
800
Wavelength (cm⁻¹)
A

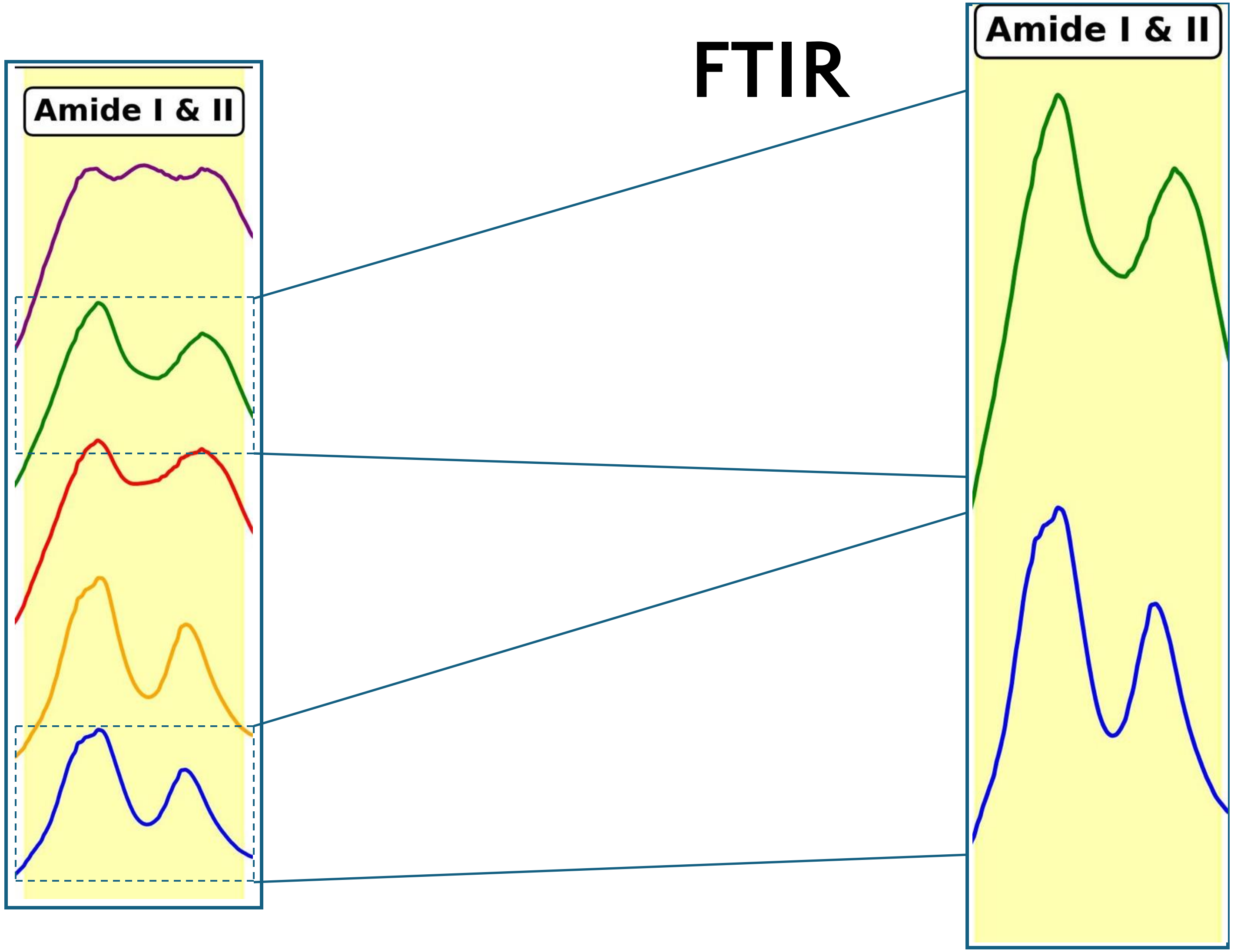
FTIR



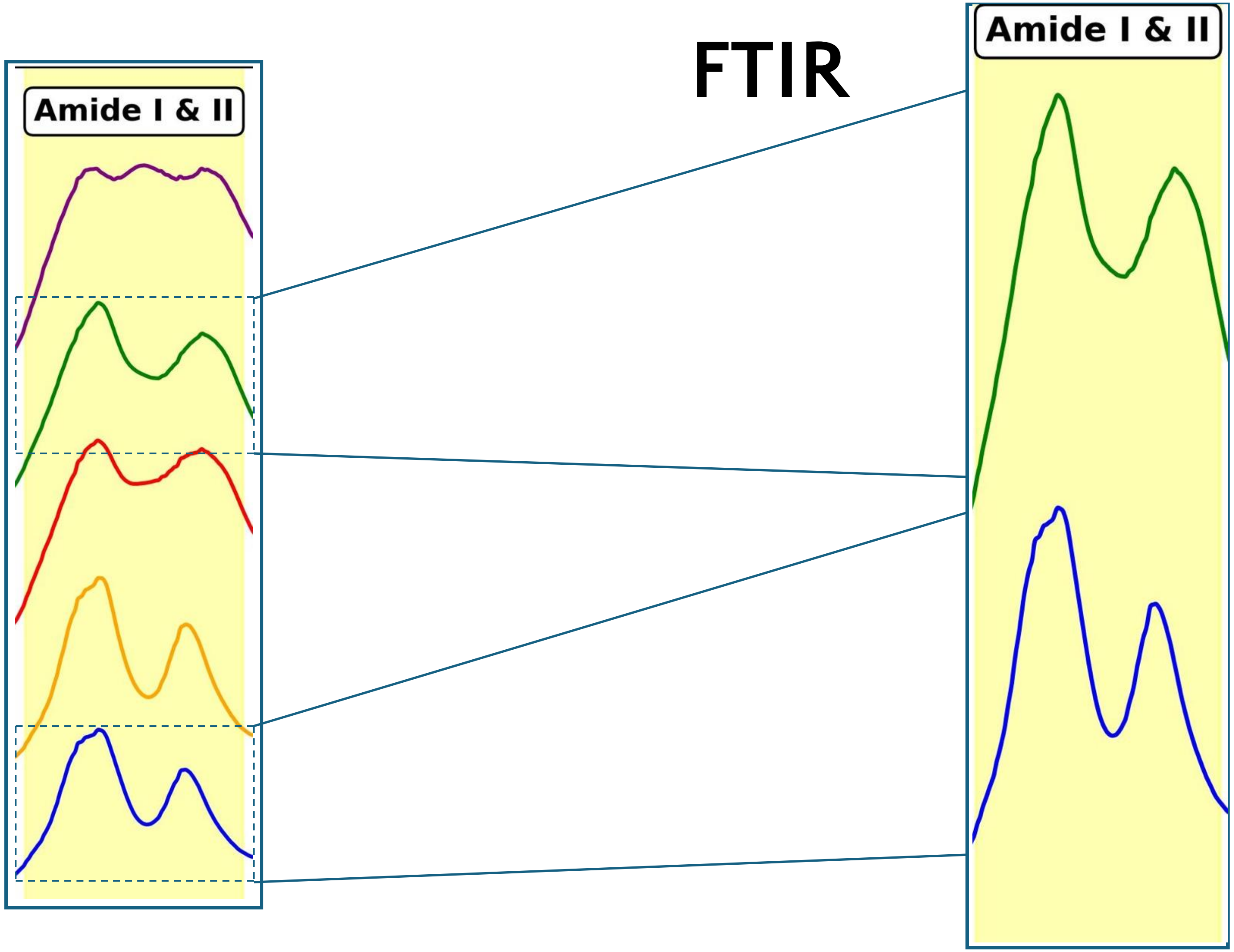
FTIR



FTIR



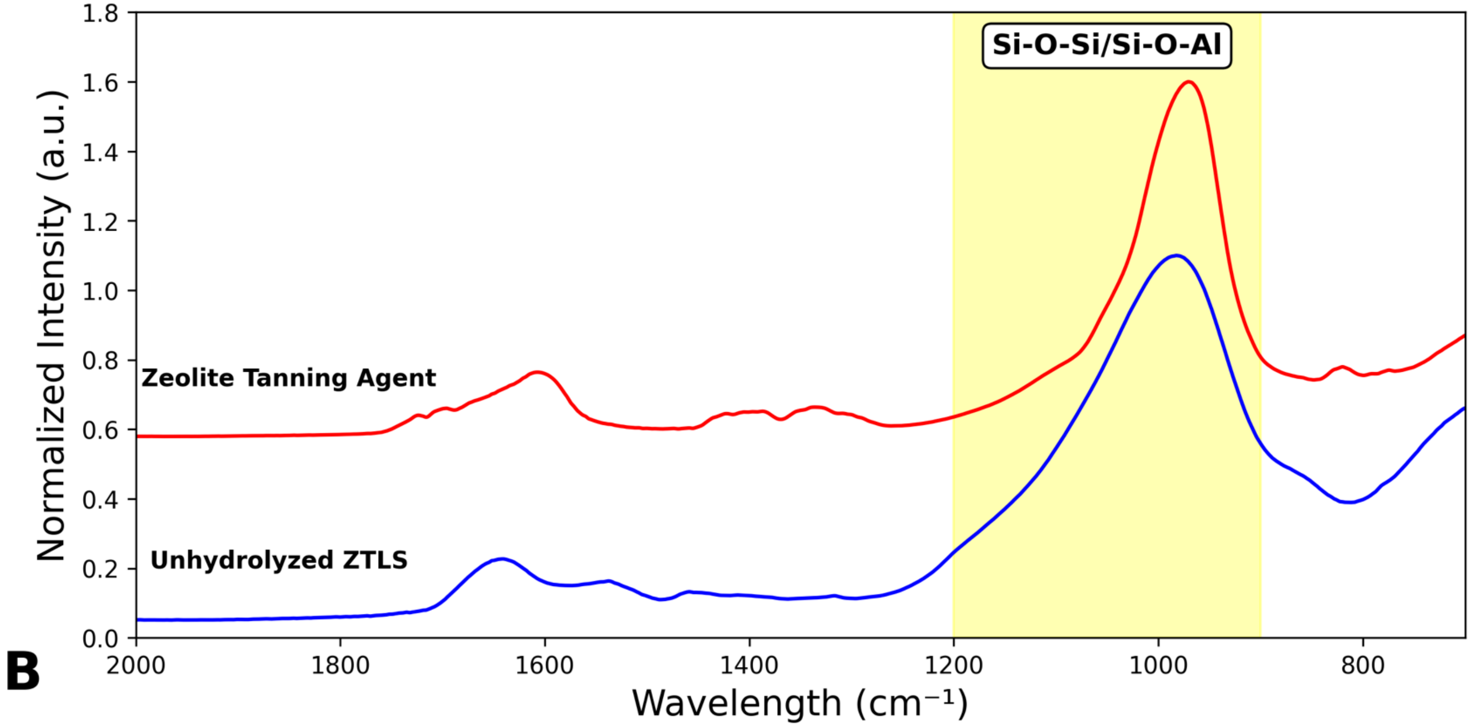
FTIR



Reduced Amide I & II peaks → collagen degraded in hydrolysis

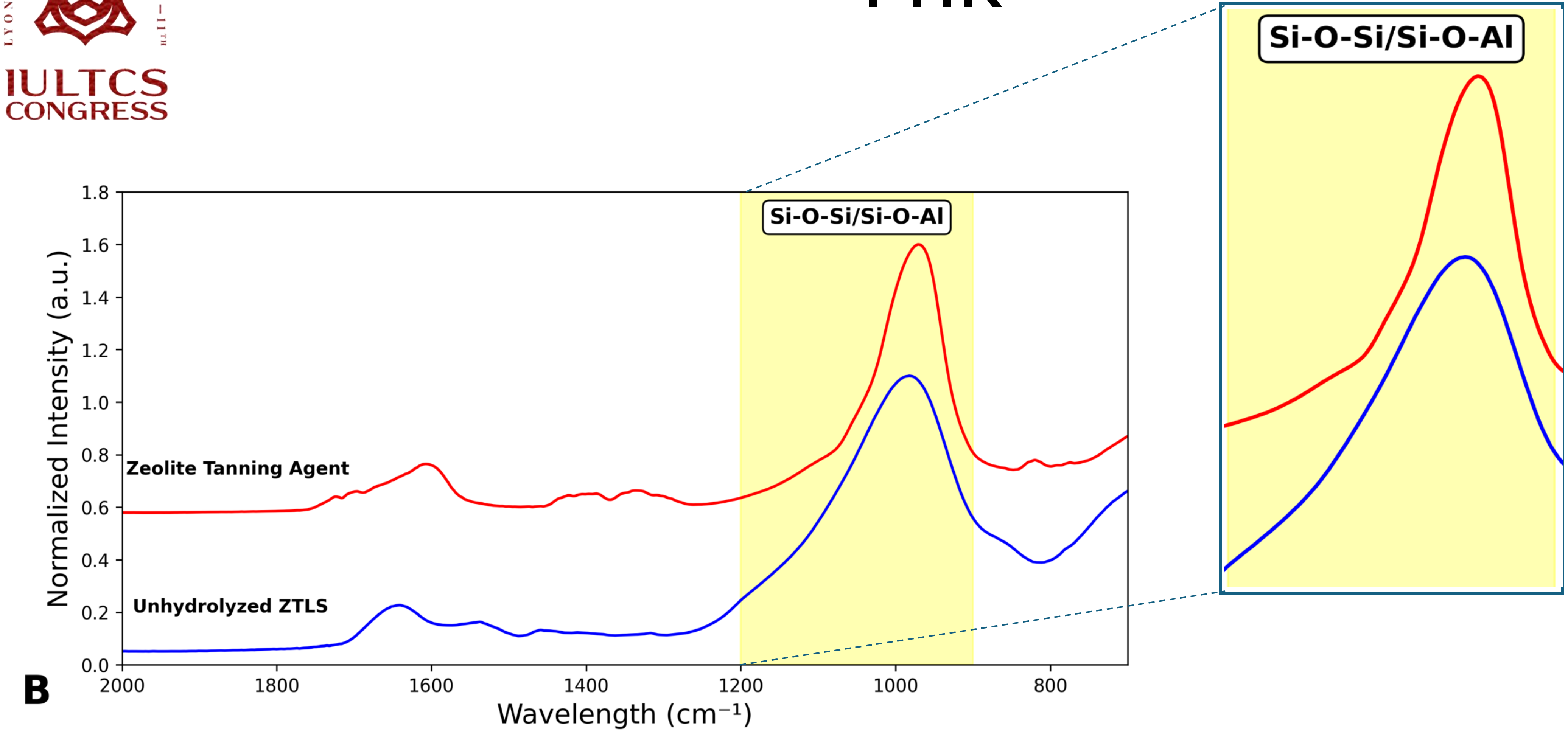
No clear zeolite peaks in hydrolysate → zeolite remained in unhydrolyzed residue

FTIR



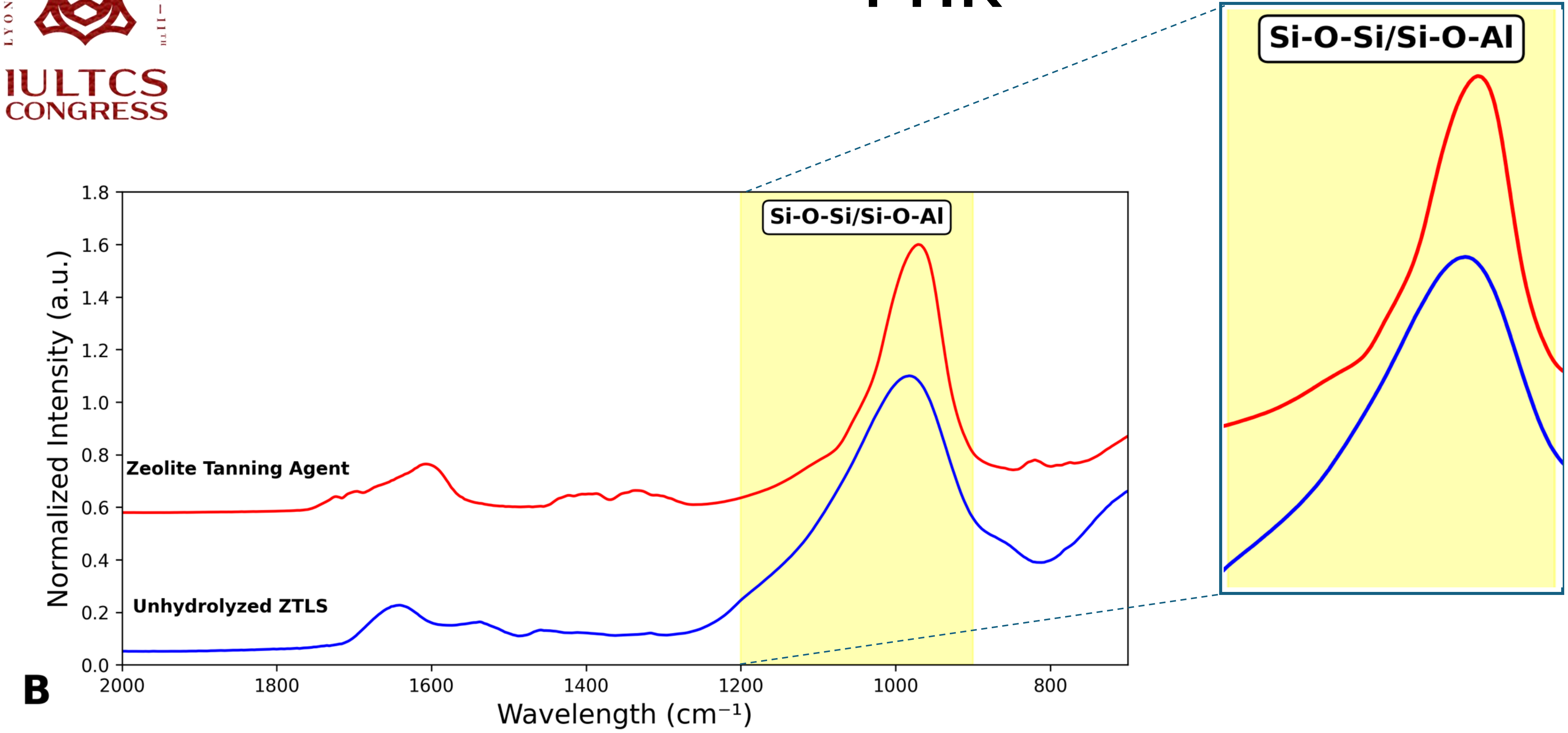
Si-O-Si/Si-O-Al
Zeolite Tanning Agent
Unhydrolyzed ZTLS
Normalized Intensity (a.u.)
Wavelength (cm⁻¹)
B

FTIR



Si-O-Si/Si-O-Al
Zeolite Tanning Agent
Unhydrolyzed ZTLS
Normalized Intensity (a.u.)
Wavelength (cm⁻¹)
B
1.8
1.6
1.4
1.2
1.0
0.8
0.6
0.4
0.2
0.0
2000
1800
1600
1400
1200
1000
800
Si-O-Si/Si-O-Al

FTIR



Strong Si–O, Si–O–Al peaks → zeolite structure

Spectrum matches pure zeolite → tanning agent retained

Minimal Amide I & II peaks → collagen largely hydrolysed

Elemental Analysis

Sample Reference	Al (III) (mg/kg)	Si (IV) (mg/kg)	Na (I) (mg/kg)
ZTLS Hydrolysate	3339	<10	51,983
ZTLS Unhydrolyzed	10,400	<10	5444
ZTLS	14,600	<10	1145

Elemental Analysis

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~20% Al (III)

~80% Al (III)

Elemental Analysis

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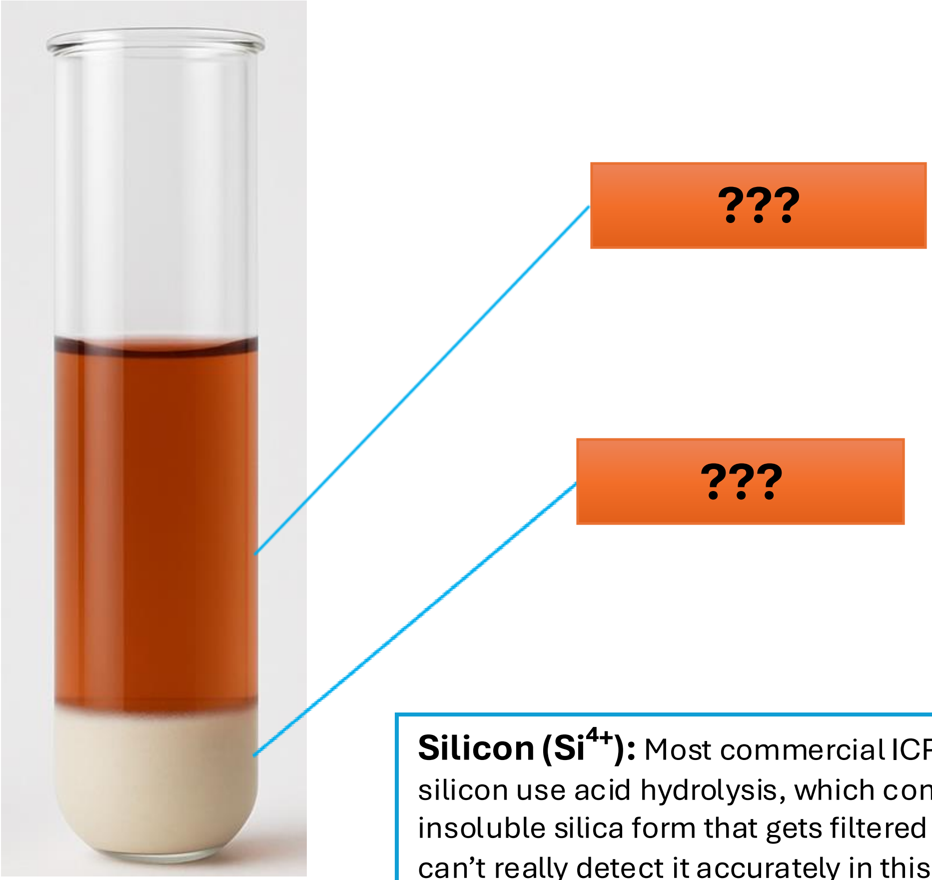


Silicon (Si^{4+}): Most commercial ICP methods for silicon use acid hydrolysis, which converts it to an insoluble silica form that gets filtered out — so you can't really detect it accurately in this case.

Elemental Analysis

Sample Reference	Al (III) (mg/kg)	Si (IV) (mg/kg)	Na (I) (mg/kg)
ZTLS Hydrolysate	3339	<10	**51,983**
ZTLS Unhydrolyzed	10,400	<10	**5444**
ZTLS	14,600	<10	**1145**

~4,540% Na (I)

~475% Na (I)

Sodium (Na):

- Extremely elevated in hydrolysate due to added NaOH during hydrolysis.
- Sodium ions don't bind strongly to collagen or zeolite.
- They remain dissolved and therefore accumulate in the hydrolysate.

SEC (Size-Exclusion Chromatography)

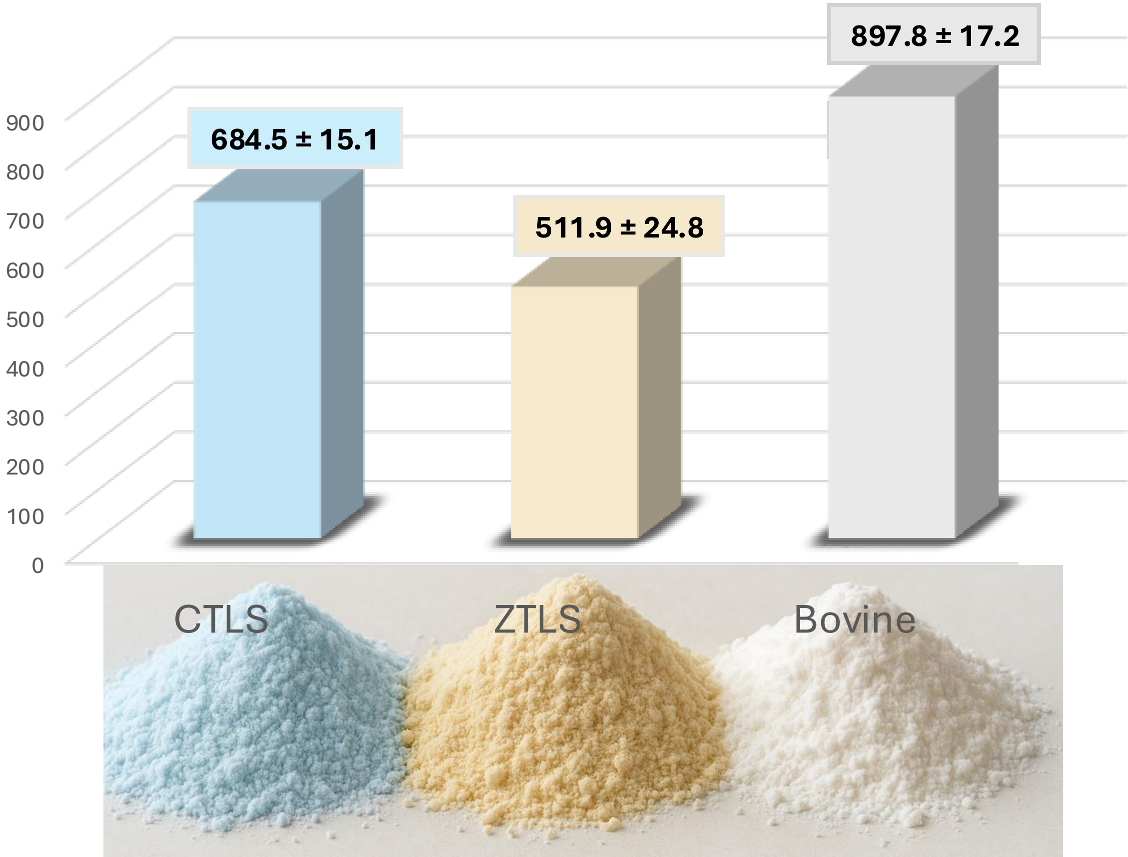
Sample	Molecular Weight Distribution (Da)
ZTLS Hydrolysate	500 – 296,000
CTLS Hydrolysate	200 – 38,600
Bovine Hydrolysate	100 – 35,900

ZTLS hydrolysate shows a much broader molecular weight distribution (~1–300 kDa) compared to bovine hydrolysate (~1–35 kDa).

FTIR confirms zeolite presence, and ICP shows ~20% of the zeolite remains in the hydrolysate.

This residual zeolite likely interacts with collagen, causing aggregation or other interactions, which contributes to the wider molecular weight distribution observed.

Amino Acid Analysis



Bovine hydrolysate → highest amino acid content.

ZTLS hydrolysate → lowest amino acid content.

Reason?

- ~20% Al solubilised → inorganic carryover lowers amino acid purity
- Lower starting protein in ZTLS vs bovine/CTLS
- Higher ash content: ZTLS ~17%, CTLS ~5%, bovine ~1–2%

Key Takeaways

• Alkaline hydrolysis effectively breaks down zeolite-tanned leather shavings.

• Most zeolite remains in the unhydrolyzed fraction, with a small portion bound to collagen in the hydrolysate, limiting total protein and amino acid yield.

• Zeolite–collagen interactions lead to aggregate formation, shown by the broader molecular weight distribution in the ZTLS profile.

• Lower starting protein content in ZTLS compared to bovine hide or CTLS further reduces amino acid yield.

• The hydrolysate is non-toxic and safe for potential industrial applications.

Thank you for your time and attention.

Acknowledgments

Sujay Prabakar, Shereen Ong, and Wouter Hendriksen

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