VISCOELASTIC CHARACTERIZATION OF LEATHERS PROPERTIES: A NOVEL NON DESTRUCTIVE METHOD USING MICRO-INDENTATION TECHNIQUE

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Viscoelastic characterization of leathers properties: a novel nondestructive method using micro-indentation technique

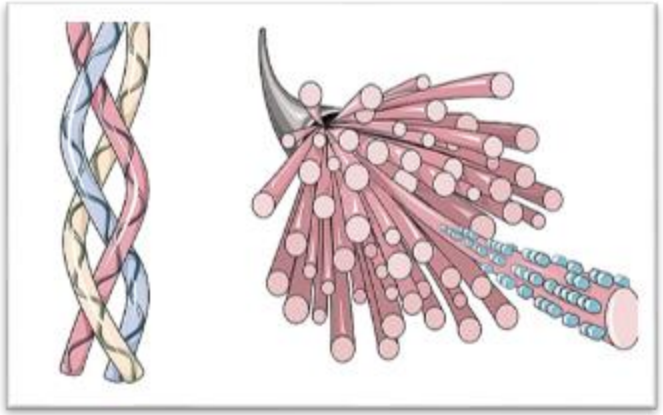
Scope of the Research

Material and Methods

Discussion of Results

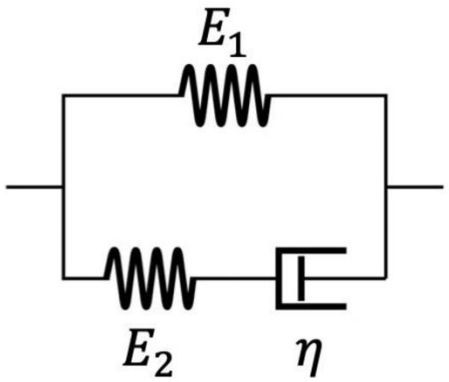
Scope of the Research

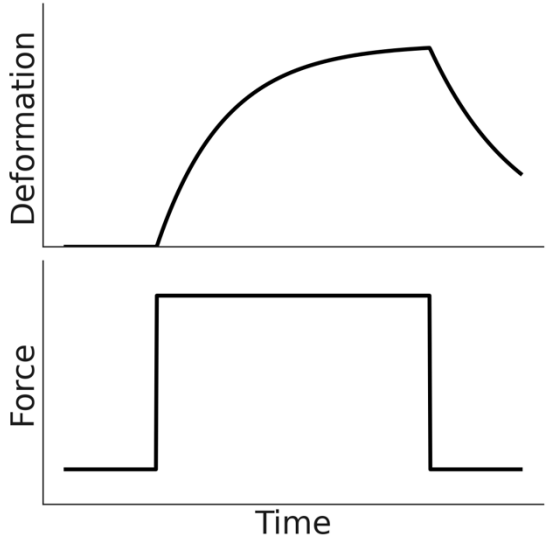
Leather exhibits a hierarchical **fibrous** structure of collagen, which directly governs its **time-dependent** mechanical response, making **viscoelasticity** an inherent consequence of its architecture



These properties are critical to the material's performance in a wide range of industrial applications, from fashion to the automotive sector.

Scope of the Research





A **Viscoelastic behavior** is a mechanical response of materials that combines both elastic (energy storage) and viscous (energy dissipation) properties, resulting in **a time-dependent** deformation after a force application.

Scope of the Research

Viscoelastic properties are analysed through Dynamic Mechanical Analysis (DMA), which defines:

- **E' (Storage Modulus)**, representing the elastic (energy-storing) component
- **E" (Loss Modulus)** representing the viscous (energy-dissipating) component
- **tan δ = E"/E' (Dumping factor)** that quantifies the damping ability of the material

DMA is a destructive instrumental analysis that requires a **highly sophisticated and expensive** apparatus involving a high energy consumption.

Scope of the Research

As an alternative, in the present study a novel **micro-indentation-based** methodology is proposed, using a testing device originally engineered in the Motorsport domain for the characterization of high-performance tires .

The technique involves the **controlled impact** of a semi-spherical **indenter** on the leather surface, followed by the analysis of the **resulting rebound curves**.

From the analysis of the **displacement vs time** curve, key **viscoelastic parameters** such as storage modulus and dumping factor are defined.

Scope of the Research

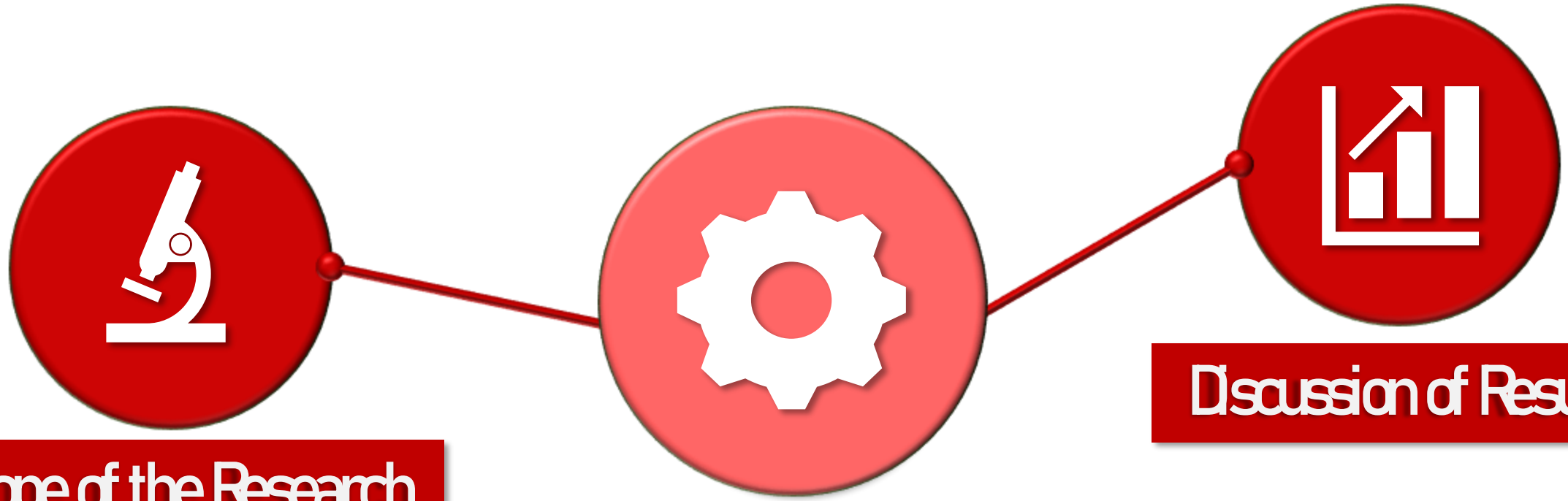
For the scope of this research :

- **First part :** the definition of a **test method** and the characterization of 4 different leather samples were carried out.

- **Second Part:** an example of application to identify viscoelastic changes in automotive leather after **mechanical and thermal treatments** is shown.

The aim is to develop a **non-destructive** diagnostic method to assess the **viscoelastic properties** or variations.

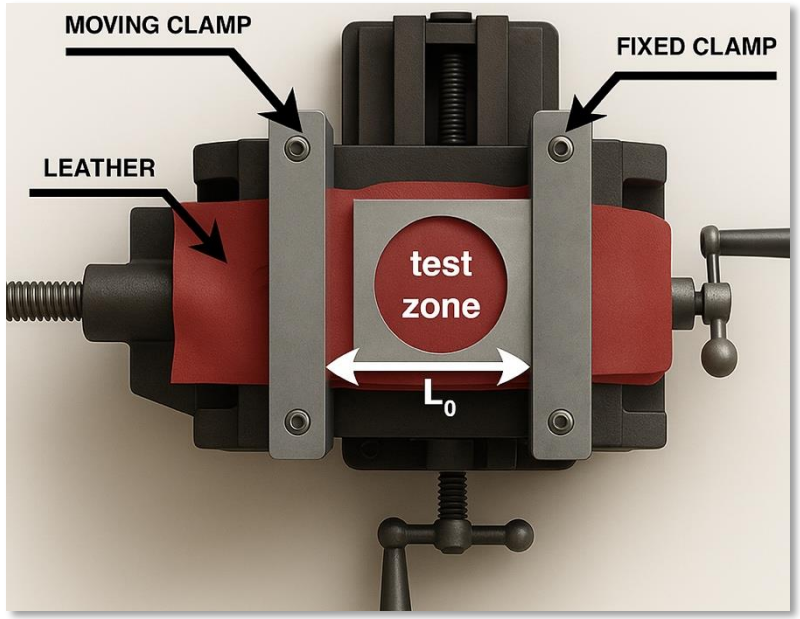
Viscoelastic characterization of leathers properties: a novel nondestructive method using micro-indentation technique



Material and Methods

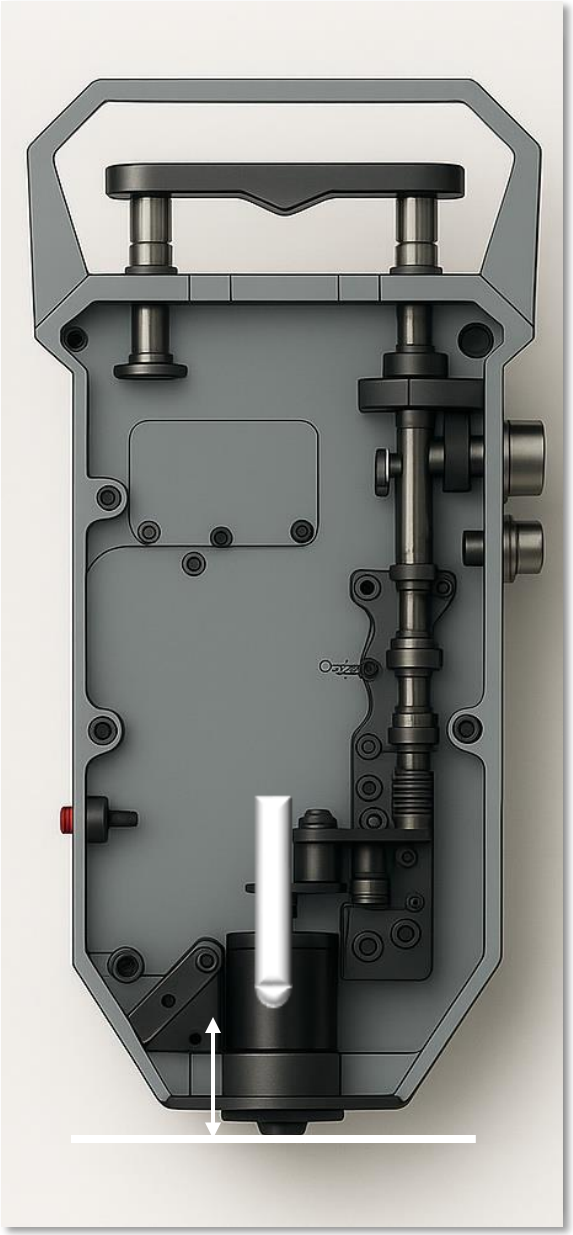
The device was originally designed for the characterization of elastomeric materials. Since leather is a flexible material, its response is strongly **influenced by the tension** in the area where the indenter impacts.

- To ensure a standardized extension in the indentation area a customized **specimen holder** was designed.
- To verify **the effects of distension**, Storage Modulus and Damping Factor were measured at different initial strain levels: **0 % (no strain), 3 %, 5 %, 7 % and 10 %**.



Material and Methods

The device used consists in a **steel rod with a semi-spherical** indenter that freely falls and **rebounds** on the surface.



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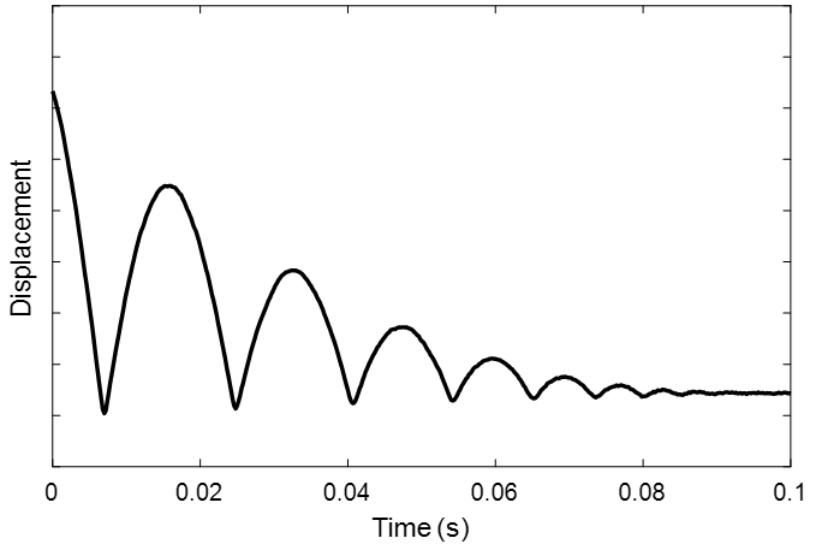
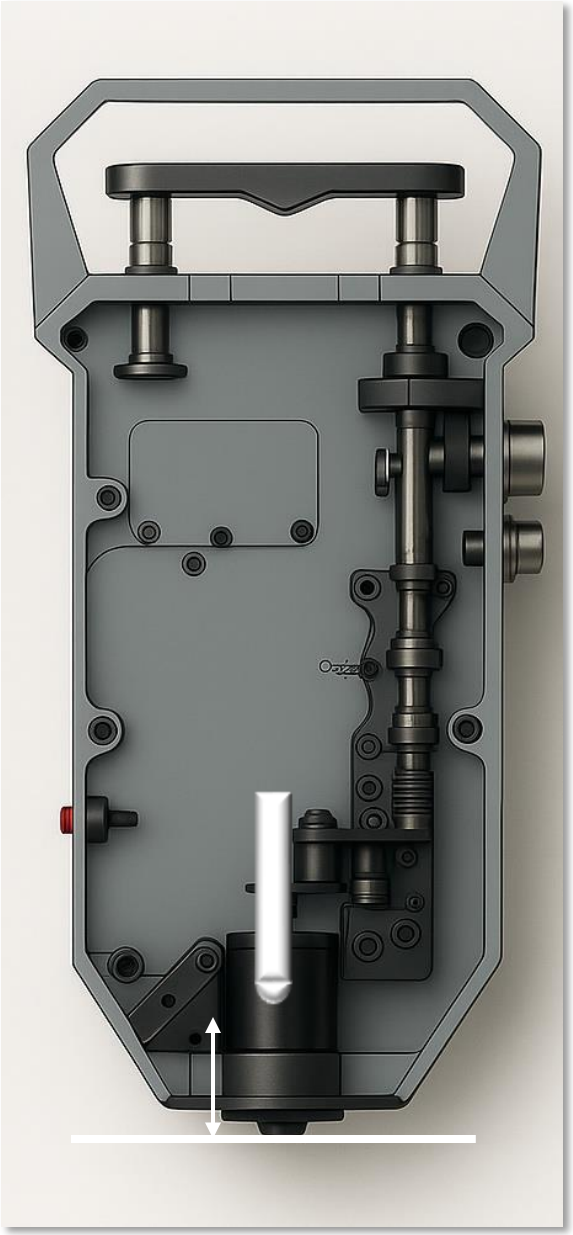
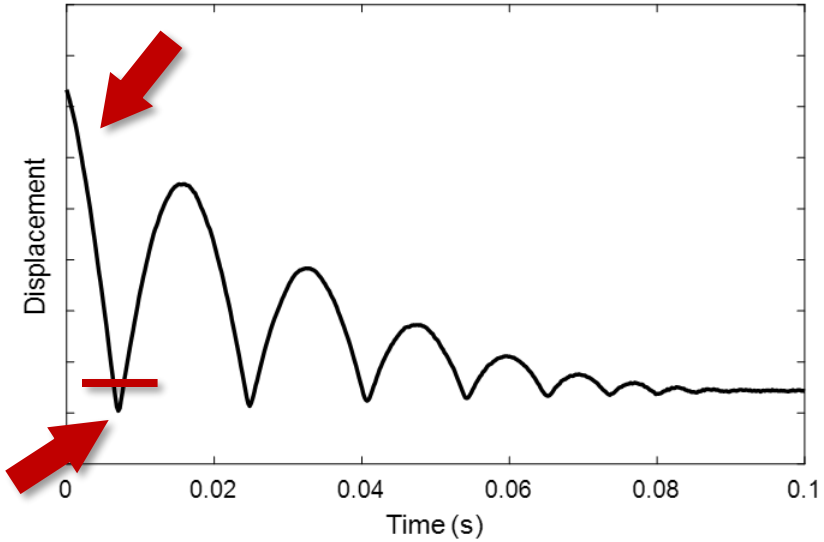


The resulting **displacement vs time** diagram is typical for each material and from its interpretation it is possible to qualify its mechanical response.

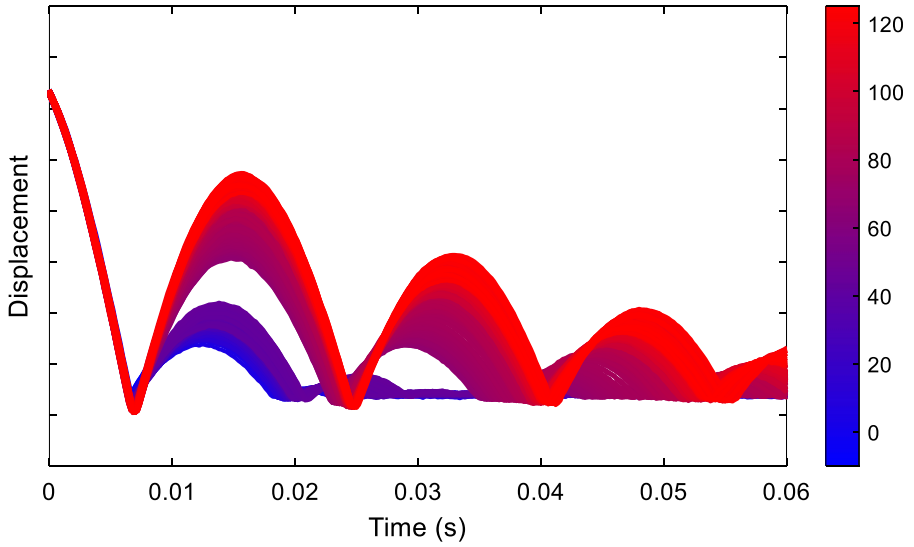


Material and Methods



The data elaboration software (VESevoLAB) allows the definition of the **displacement** curve of the rod and the **temperature** of the tested surface. A single curve shows:

1. The **falling phase** from the initial release height.
2. The **indentation** phase caused by the penetration of the indenter into the material.
3. The **transient** phase during which the rod shows a **damped oscillation.**



The **damped oscillation** is strongly related with the inverse of the **Storage Modulus (E').**

Material and Methods

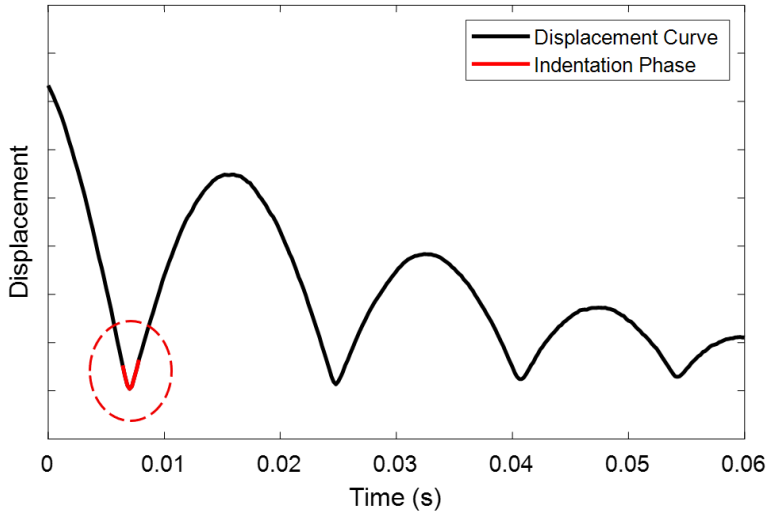
The **First indentation** allows to assess the **dissipative component**.

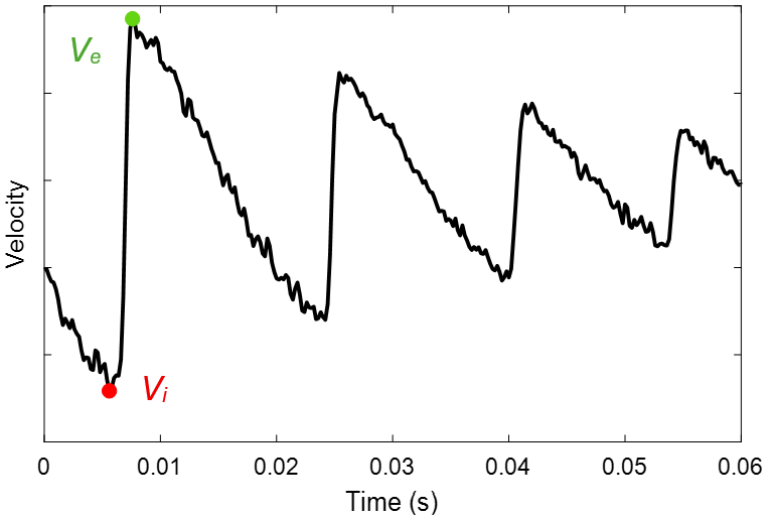
Deriving the displacement with respect to the time, the quadratic variation of the **impact velocity** (V_i, **red dot**) and the **rebound velocity** (V_e, **green dot**) is proportional to the kinetic energy dissipated in the material:

$$\Delta E_k = K\left(V_i^2 - V_e^2\right) \qquad \text{(Eq. 1)}$$

which was found to be proportional to the **Damping Factor (tan δ)**

The algorithm of the device's software **converts** geometrical information, displacements and velocities into the viscoelastic properties **E' and tan δ.**

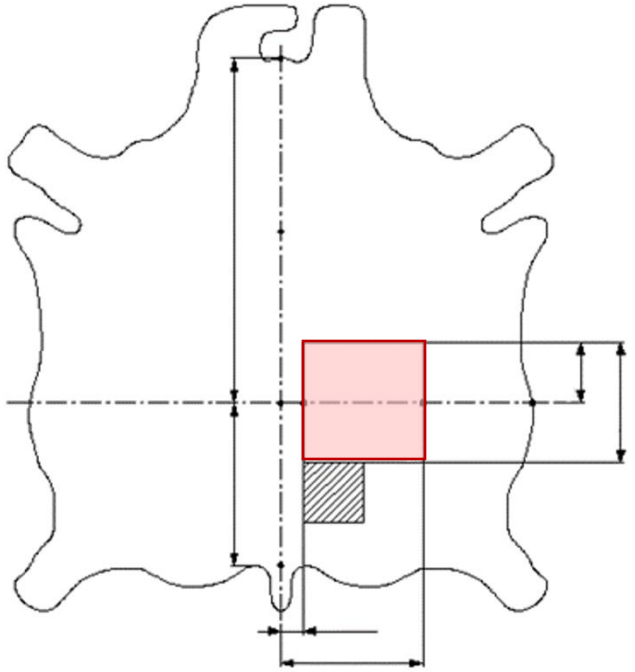




Material and Methods

Four different **sample** types were analysed using this technique:

- **Garment** chrome tanned calf in Crust (Sample 1)
- **Footwear** bovine chrome tanned leather (Sample 2)
- **Automotive** glutaraldehyde crust bovine leather (Sample 3)
- **Leather Goods** By-Cast PU-finished split leather (Sample 4)



In the **first step**, for the definition of the performances of the method specimens to be tested have been cut according to ISO 2418:2023 after conditioning for 24 hours at 23 °C and 50 % R.H. according to ISO 2419:2024

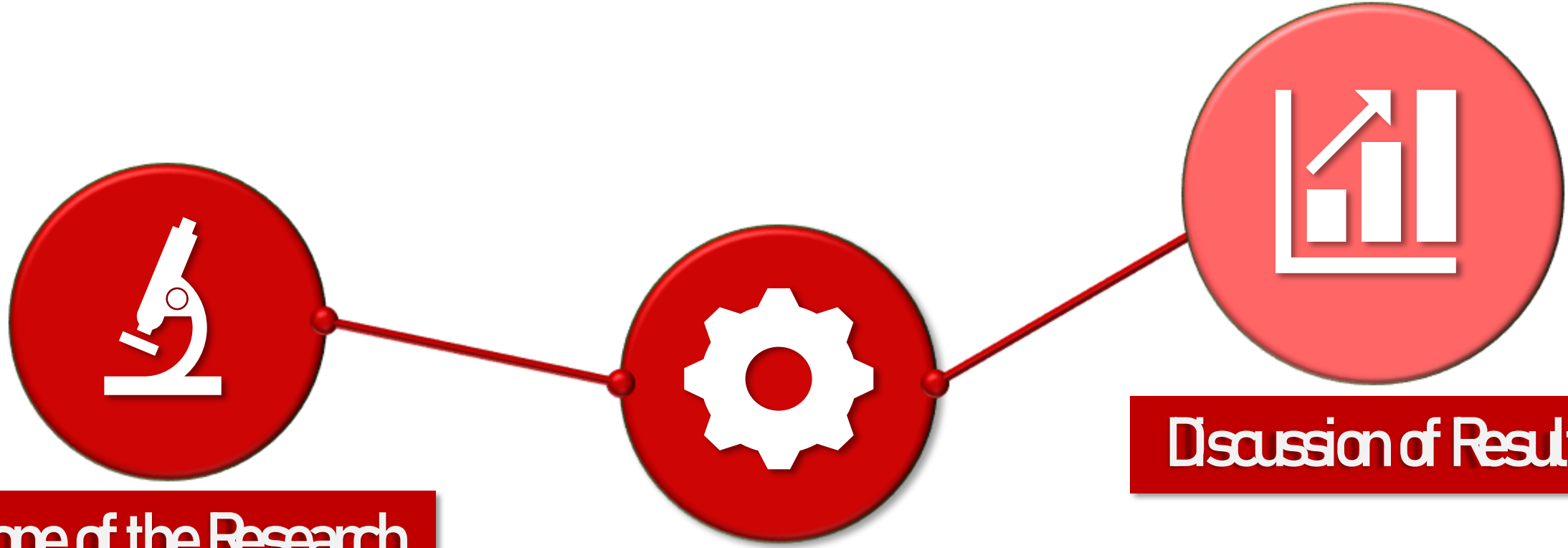
Material and Methods

As an application example, measurements were carried out on **automotive** crust leather after two distinct material conditioning treatments:

- **Thermal conditioning** at high-temperature to 120 °C for 4 hours,
- **Mechanical conditioning** at 10 % deformation applied for 1 hour under stress relaxation conditions to simulate a nailing process.

The tests aimed to evaluate changes in the Viscoelastic parameters resulting from **configurational modifications of the fibrous structure** induced by these thermal and mechanical stresses.

Viscoelastic characterization of leathers properties: a novel nondestructive method using micro-indentation technique



Scope of the Research

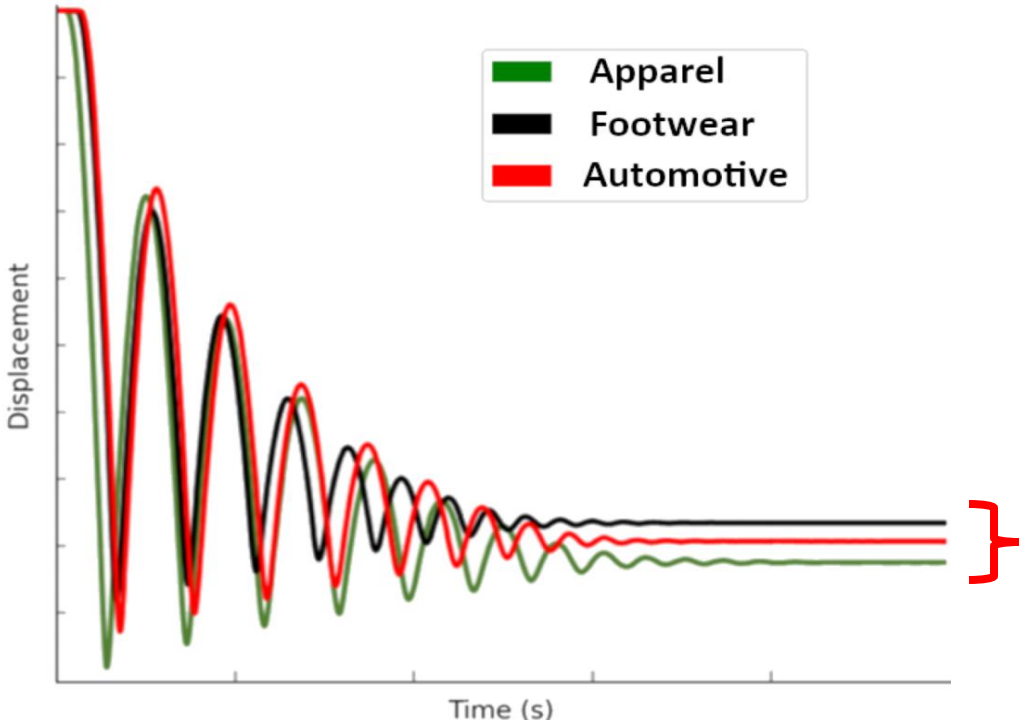
Material and Methods

Discussion of Results

Discussion of Results

The **rebound and damping profiles** of Samples 1, 2, and 3, (apparel, automotive, and footwear) show that each material type exhibits a **distinct** response profile in terms of:

- **Amplitude,**
- **Damping** behavior,
- **Phase shift** observed between the curves.



Additionally, the **residual height** after rebound provides indication about the material **softness.**

Discussion of Results

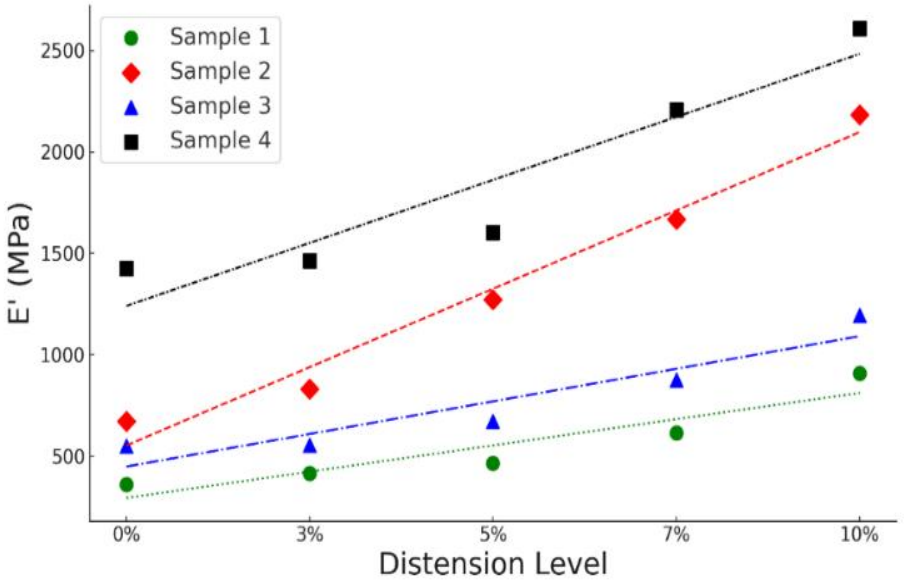
E' and tan δ at different initial strain levels (0 % to 10 %).

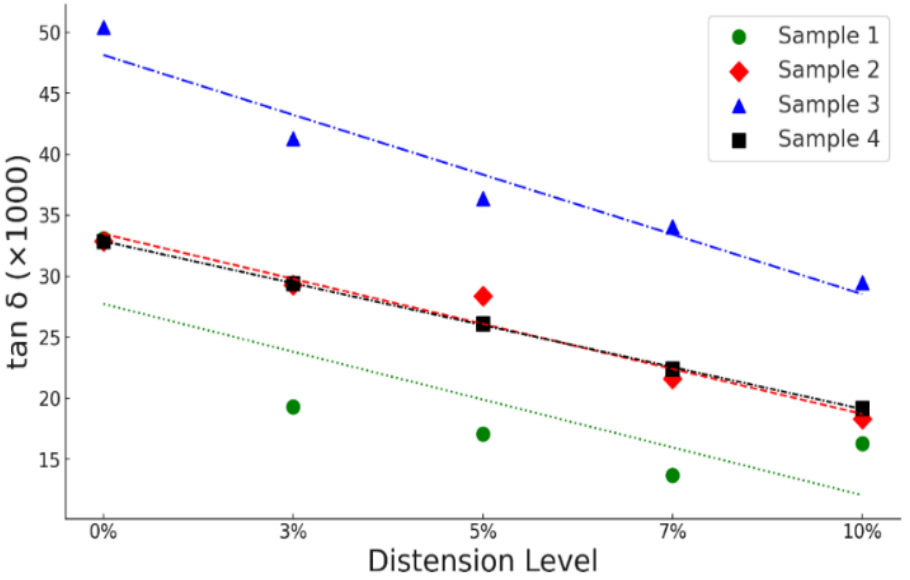
Increasing the initial strain leads to a:

- **increase** in the storage modulus (**E'**)
- **reduction** in the damping factor (**tan δ**)

Increasing the strain a progressive alignment of collagen fibers occurs. The ability to slide past each other diminishes, resulting in a stiffer structure with reduced internal friction.

Consequently, the material exhibits a **more elastic** and less **viscous response**.





Discussion of Results

The results of measurements for all samples at each strain level are shown in the following tables.

DISTENSION	0%		3%		5%		7%		10%	
SAMPLE N.	E'[kPa]	*St.Dev*	E'[kPa]	*St.Dev*	E'[kPa]	*St.Dev*	E'[kPa]	*St.Dev*	E'[kPa]	*St.Dev*
Sample 1	362	*36,0*	415	*37,1*	465	*29,4*	615	*46,1*	909	*45,6*
Sample 2	671	*19,8*	831	*33,0*	1273	*23,8*	1667	*13,9*	2184	*43,4*
Sample 3	551	*7,8*	556	*7,3*	673	*15,1*	877	*23,4*	1194	*25,7*
Sample 4	1427	*19,8*	1464	*33,0*	1603	*23,8*	2208	*13,9*	2608	*43,4*

Table 1 – Storage Modulus E' in kPa at different strain level

DISTENSION	0%		3%		5%		7%		10%	
SAMPLE N.	tan δ	*St.Dev*	tan δ	*St.Dev*	tan δ	*St.Dev*	tan δ	*St.Dev*	tan δ	*St.Dev*
Sample 1	28,1	*0,92*	25,3	*1,41*	24,7	*0,72*	23,0	*1,12*	20,2	*1,51*
Sample 2	33,1	*2,06*	19,3	*1,02*	17,1	*1,69*	13,7	*0,51*	16,3	*1,30*
Sample 3	32,9	*1,19*	29,3	*1,31*	28,4	*1,25*	21,6	*1,13*	18,3	*0,72*
Sample 4	50,4	*1,13*	41,3	*0,89*	36,4	*0,90*	34,1	*2,23*	29,5	*0,53*

Table 2 – Damping factor tan δ at different strain level

At **0 % strain** the data are consistent and repeatable.

The initial strain may be not strictly necessary for the viscoelastic characterization.

However, also a **strain of 3 %** was applied to ensure flat positioning of the leather samples.

Discussion of Results

Crust leather for automotive was subjected to two treatments:

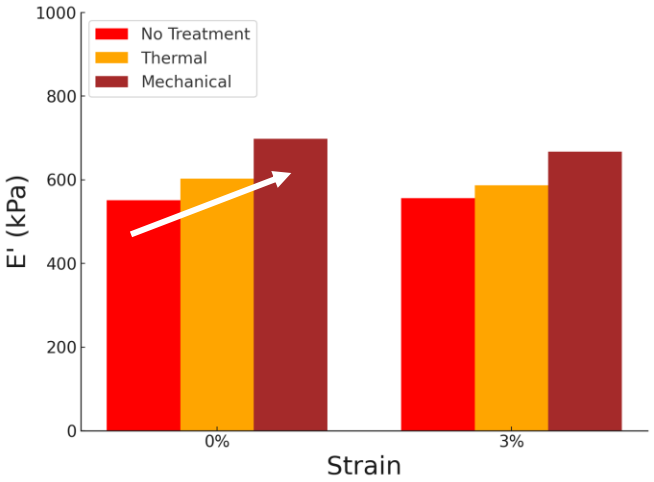
- **Mechanical treatment**: 10 % strain applied for 1 hour under stress-relaxation conditions to simulate the nailing process.
- **Thermal conditioning**: to 120 °C for 4 hours, a common pre-treatment to enhance leather dimensional performances under environmental stresses.

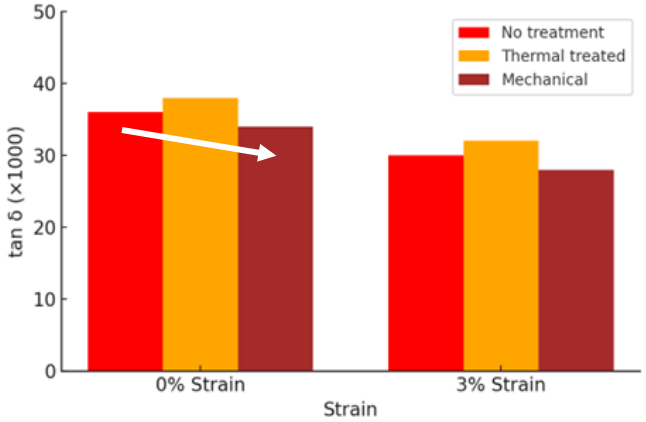
Both treatments induce configurational modifications in the fibrous structure of leather, resulting in alterations of its viscoelastic properties

The possibility of detecting these changes was assessed by means of micro-indentation technique

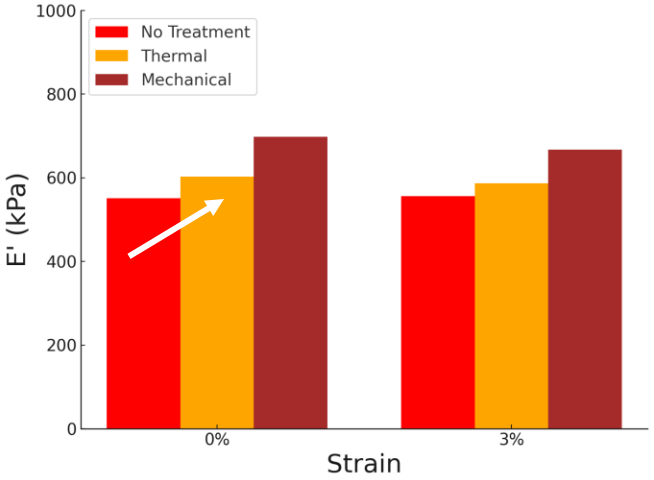
Discussion of Results

Mechanical conditioning leads to alignment of collagen fibers in the load direction, resulting in an increase in storage modulus (E') and a decrease in damping factor (tan δ).



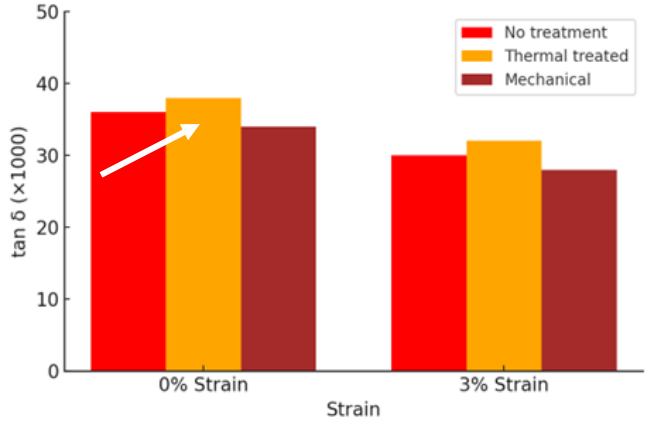


Discussion of Results



Mechanical conditioning led to alignment of collagen fibers in the load direction, resulting in an increase in storage modulus (E') and a decrease in damping factor (tan δ).

Thermal conditioning determined an increase of both E' and tan δ due to the a “fiber sticking” effect. Heat promotes the formation of additional cross-links and physical bonds between collagen fibrils, which stiffen the structure but also enhance energy dissipation through internal friction.



The micro-indenter successfully allowed to measure the changes in viscoelastic behaviour of the material after the thermal and mechanical treatment

Conclusions

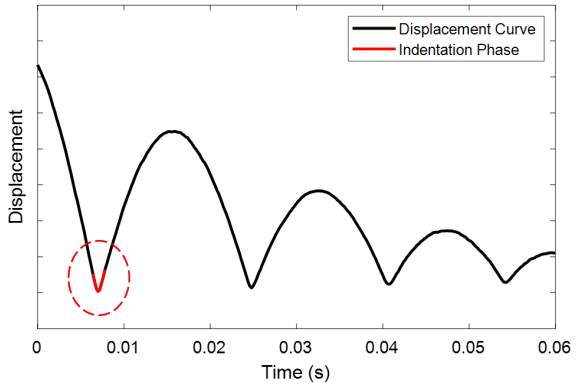
VISCOELASTIC CHARACTERIZATION OF LEATHERS PROPERTIES:
A NOVEL NON DESTRUCTIVE METHOD USING MICRO-INDENTATION TECHNIQUE

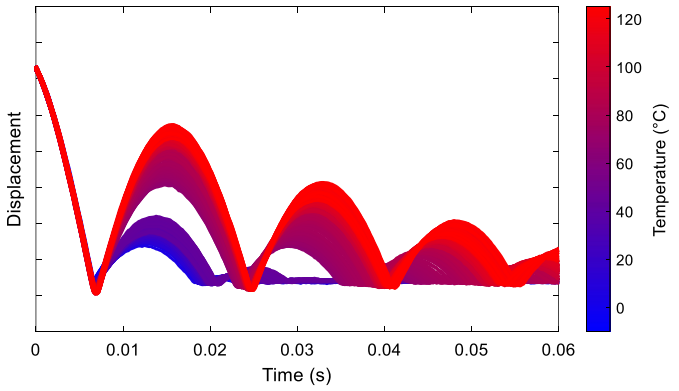
Conclusions

- In this work a **micro-indentation** technique developed within the motorsport domain was applied for the viscoelastic characterization of leathers.
- Four different leather samples for different use were characterized in terms of **Storage Modulus (E')** and **Damping factor (tan δ).**
- Tests have been carried out at **different levels of initial samples distension** (from 0 % to 10 %) and the performances of the test methods were assessed.
- For all the samples the Storage Modulus (E') increase and the Damping Factor (tan δ) decrease with the increase of initial leather distension.
- The results on **automotive** leather after **mechanical and thermal treatments** showed that the micro-indentation technique was able to detect even small variations in the viscoelastic properties.

Conclusions

This technique represents a **promising** and **efficient** approach for the in situ, non-invasive assessment of viscoelastic properties, with potential applications in quality control and material development in sectors such as automotive, footwear or apparel materials.





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