



Project

Characterization techniques applied to the thesis: Evaluation of triborheological systems with medical applications



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FINAL

Project

Agenda

01 Introduction

General description of the thesis and the importance of characterization techniques to evaluate results.

02 Characterization Techniques

Explanation of 6 techniques that allows to evaluate different characteristics inside the research.

03 Summary

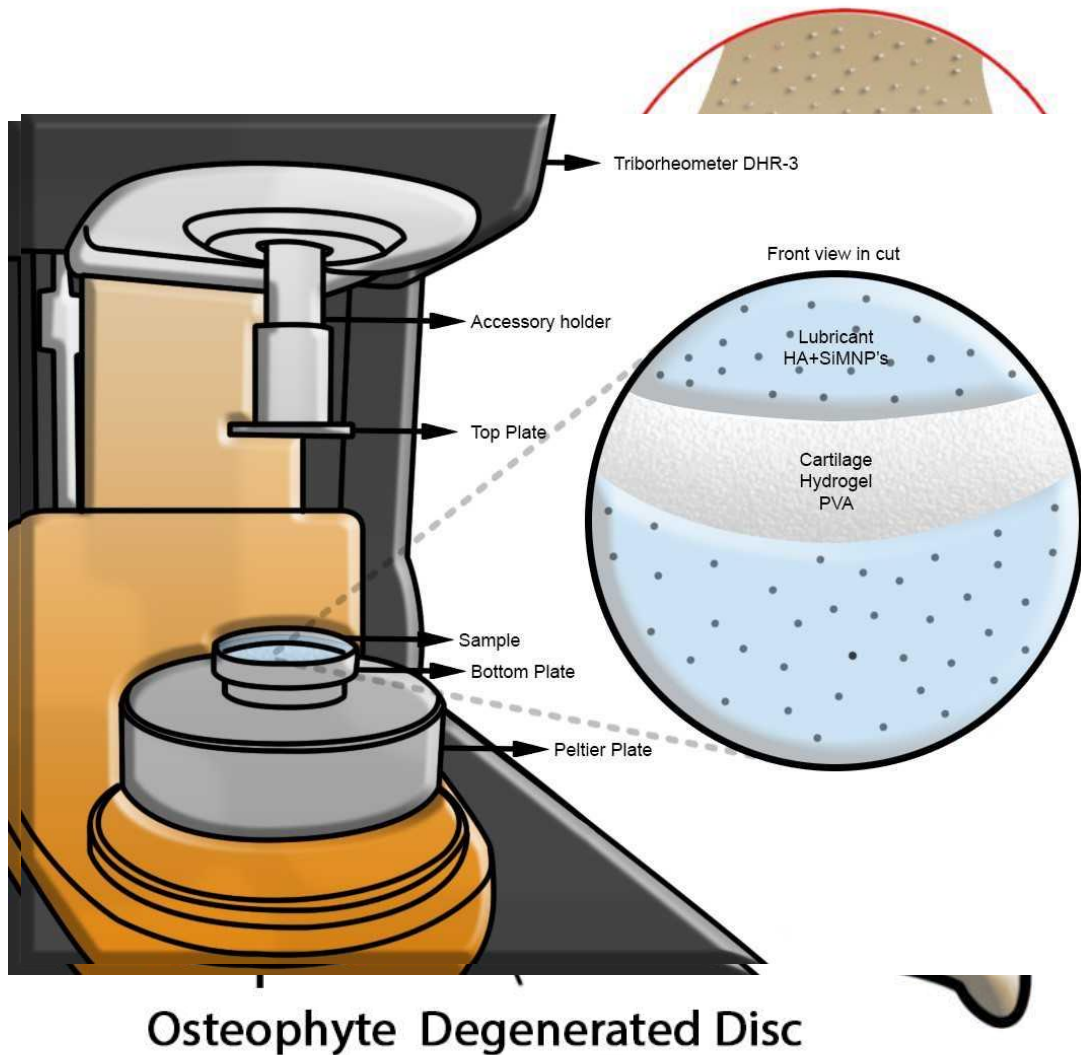
Positive effect of the techniques on the research.

04 Q/A

Questions and answers from professors and students.

INTRODUCTION

Thesis Description



Mechanical system

Synovial Joints of the human body



Osteoarthritis (OA)

Degenerative condition of synovial joints



Cartilage Replacement

Polymeric hydrogels (polyvinyl alcohol - PVA)



Lubricants

Fluids with additives (Hyaluronic acid - HA with mesoporous silica nanoparticles – SiMNP's)



Importance of characterization techniques

Porosity, surface distribution, wear tracks and performance.

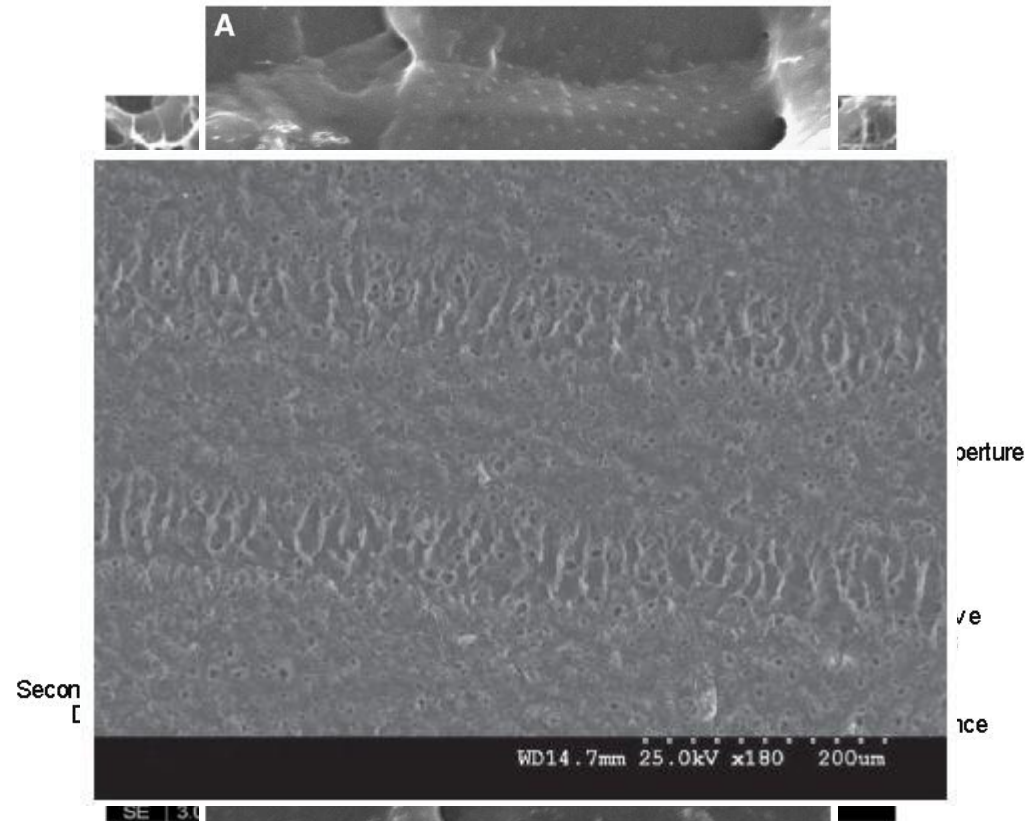
Scanning electron microscopy (SEM)

General description

A beam of electrons scans across the material surface, producing high-energy back-scattered electrons, low-energy secondary, auger electrons and cathodeluminescence. The back-scattered electrons provides a physical image of the surface whit their analysis

Sample preparation

Dry sample with conductivity properties. Coating with a tiny film of gold



1

Porosity

2

Surface distribution

3

Wear tracks after triborheological tests

4

Nanoparticles distribution in the lubricant

Li et al., Chen et al., "Preparation of Surface Mechanical Properties and Bovine Serum Albumin Adsorption Properties of Polyvinyl Alcohol Hydrogel as an Artificial Cartilage", *Advances in Materials Science and Engineering*, pp. 1 – 10, 2017.

Maiolo et al., "Development and characterization of Poly (vinyl alcohol) based hydrogels for potential use as an articular cartilage replacement", *Materials Science and Engineering C*, vol. 32, pp. 1490 – 1495, 2012.

SEM derivation

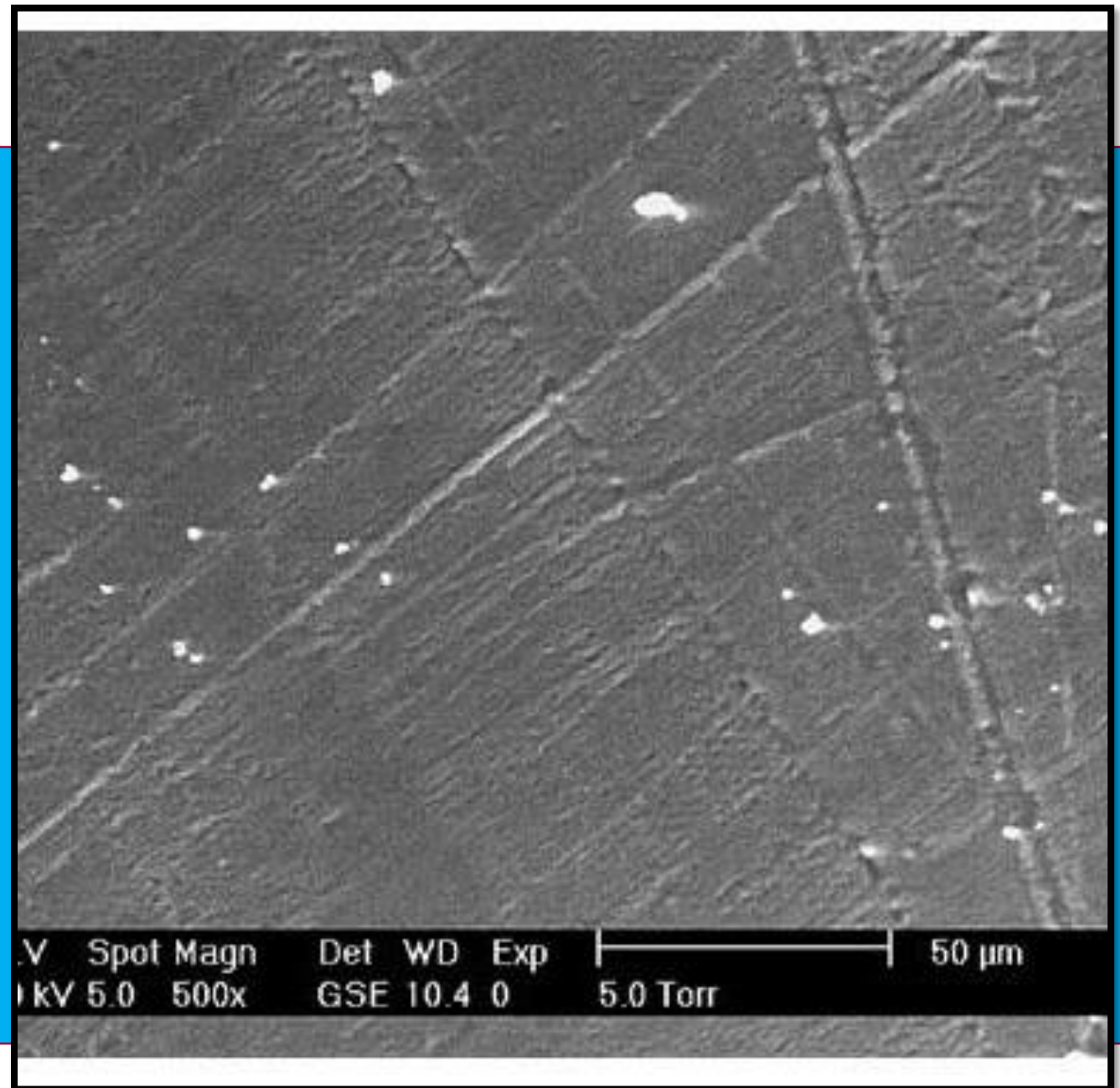
Environmental scanning electron microscopy (ESEM)

Wet-mode

Maintain the natural hydrated state of the sample without additional preparation

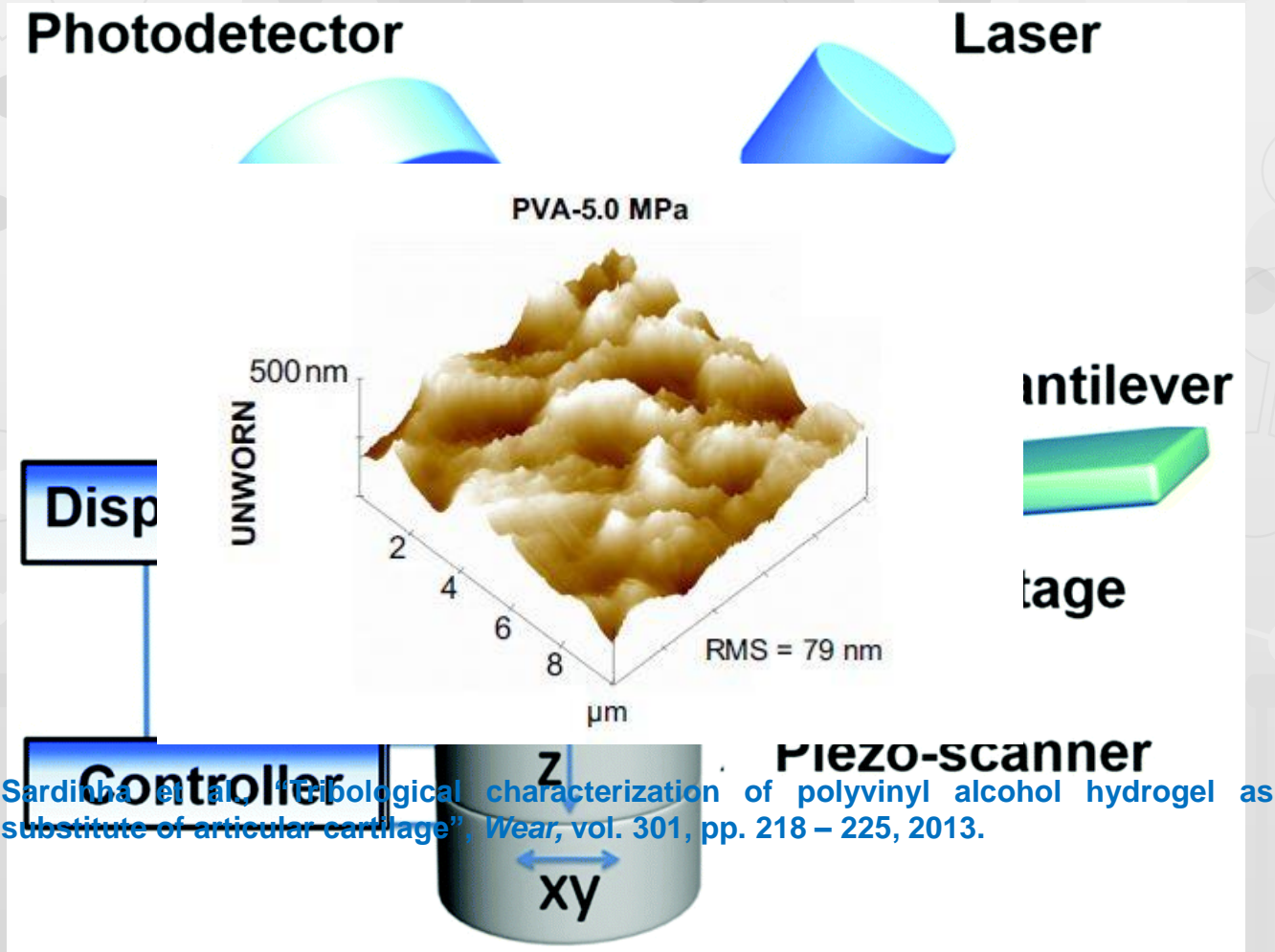
Uses

Pores, wear and cracks



Li et al., "Influence of dynamic load on friction behavior of human articular cartilage, stainless steel and polyvinyl alcohol hydrogel as artificial cartilage", *J Mater Sci: Mater Med*, vol. 21, pp. 147 – 154, 2010.

Atomic force Microscopy (AFM)



A laser beam is reflected from a cantilever that has a tip (probe). As the tip approaches the sample, the cantilever experiences deflection towards or away from the surface, causing changes on the reflection of the detected beam. The changes are detected by a photo-diode while a piezo scanner moves the tip over the sample.

- * **Sample preparation** : Hydrated state, tapping mode, distilled water, 37°C.
- * **Use** : Visualization of nano-roughness after test.

Shang, Yuping and Wang, Hongda, "The structure and function of cell membranes examined by atomic force microscopy and single-molecule force spectroscopy", *Chemical society reviews*, vol. 44, pp. 3617 – 3638, 2015.

Differential scanning calorimetry (DSC)

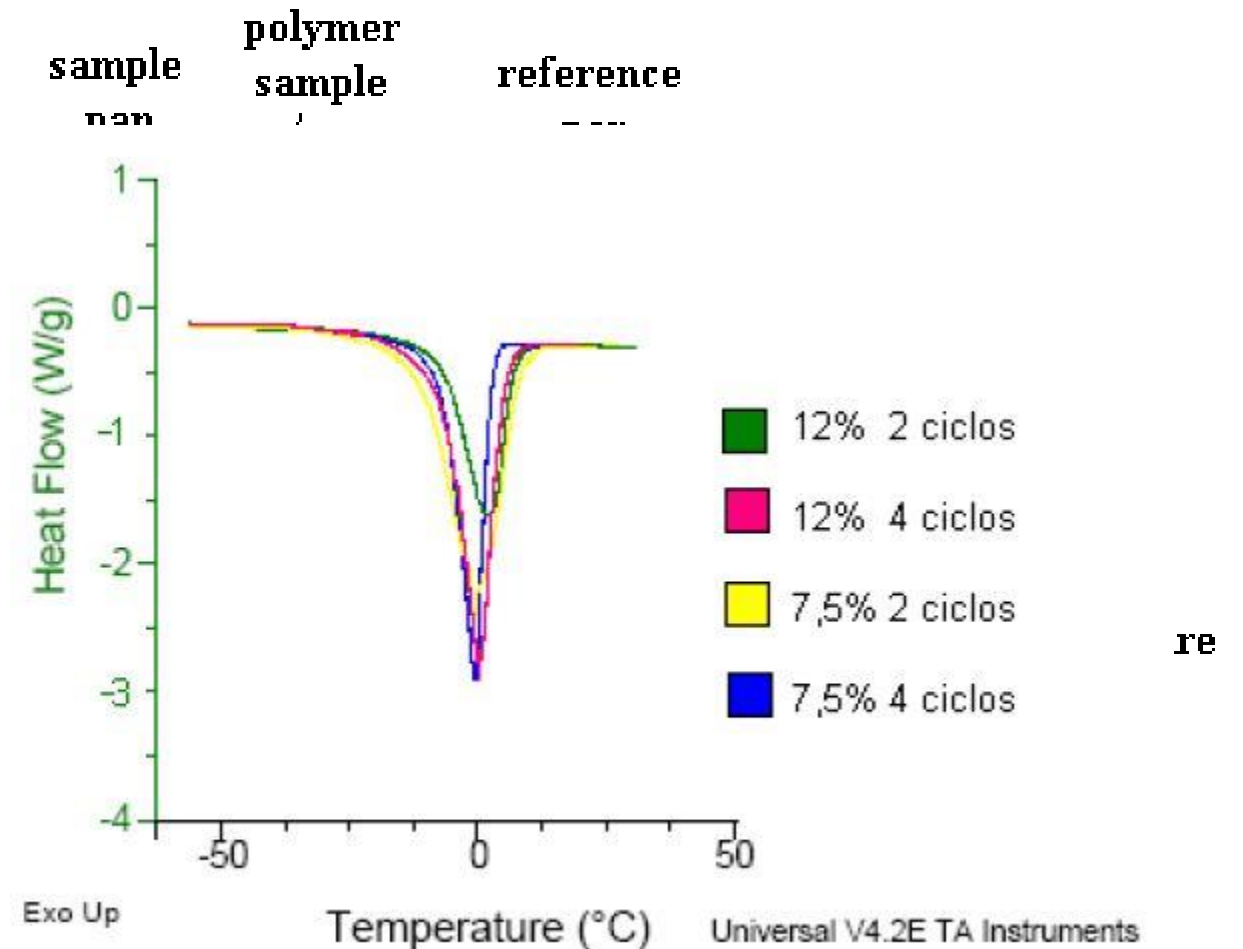
Measurements of thermodynamic properties (heat capacity) of thermally induced transitions, applied to a variety of biological elements such as biopolymers. During experiments, parameters like post scan temperature, the number of scans, their range, scan rate, and feedback strength are important and can be changed to obtain the best results.

Sample preparation

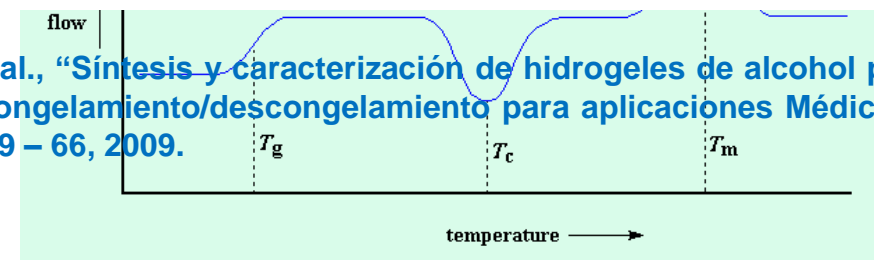
Distilled water, low scan rate (10°C/min), nitrogen atmosphere, 40 – 250 °C

Uses

- 01 Change in water content (hydrophilic behavior)
- 02 Peak temperature values (Support at body temperature)



Echeverri et al., “Síntesis y caracterización de hidrogeles de alcohol polivinílico por la técnica de congelamiento/descongelamiento para aplicaciones Médicas”, *Revista EIA*, vol. 12, pp. 59 – 66, 2009.



Polymer science learning center, “Differential scanning calorimetry”, 2019.

Recompile knowledge about molecular information (functional groups) and crystalline structures. A laser irradiate the sample with monochromatic radiation, with excitations in the UV, visible and near-IR spectral region (785-1064nm). The irradiation generates a Raman scattered light (spectrum) that is detected with a camera.

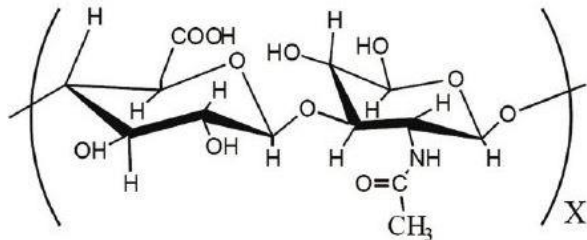
Raman spectroscopy

Sample preparation

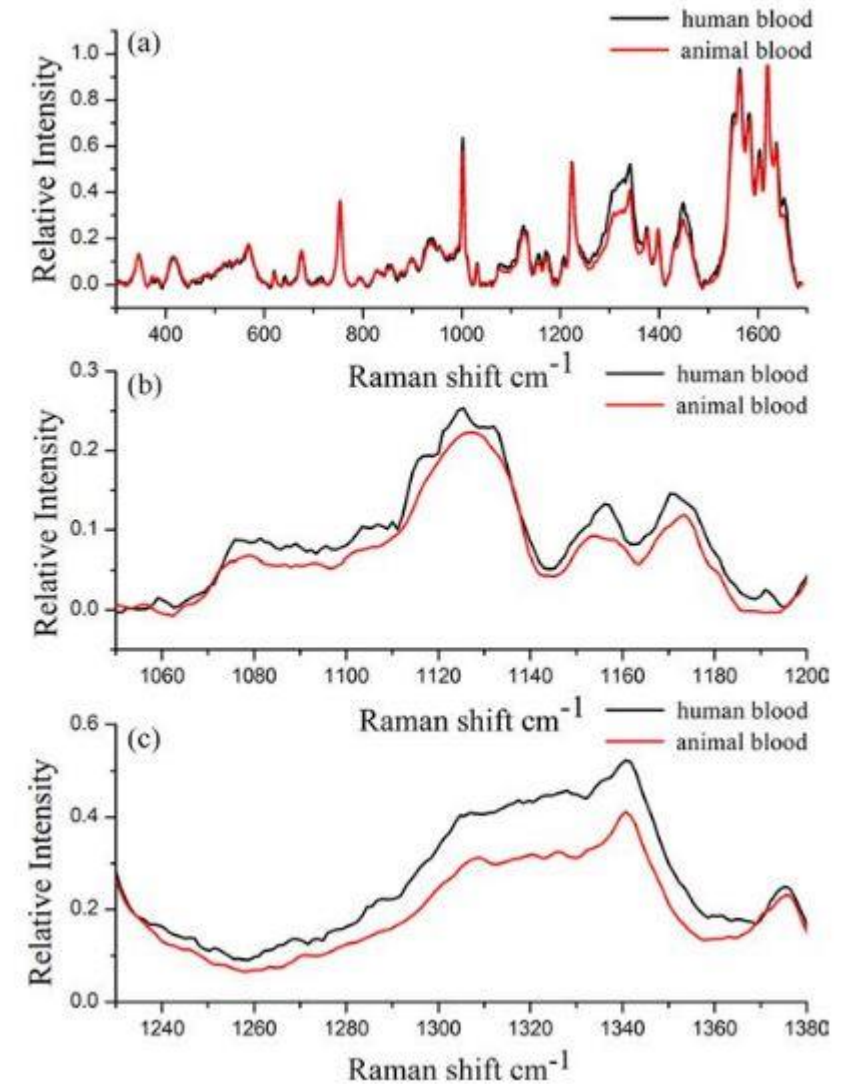
Work with metal slide at a excitation of 785 nm

Use

See variations in the fluid structure after the addition of nanoparticles.



Kim et al., "Effect of pH on Swelling Property of Hyaluronic Acid Hydrogels for Smart Drug Delivery Systems", *Journal of Sensor Science and Technology*, vol. 21, pp. 256 – 262, 2012.



108, pp.

Baia et al., "Discrimination of Human and Nonhuman Blood by Raman Spectroscopy and Partial Least Squares Discriminant Analysis", *Analytical letters*, vol. 50, pp. 379 – 388, 2017.

Thermomechanical analysis (TMA)



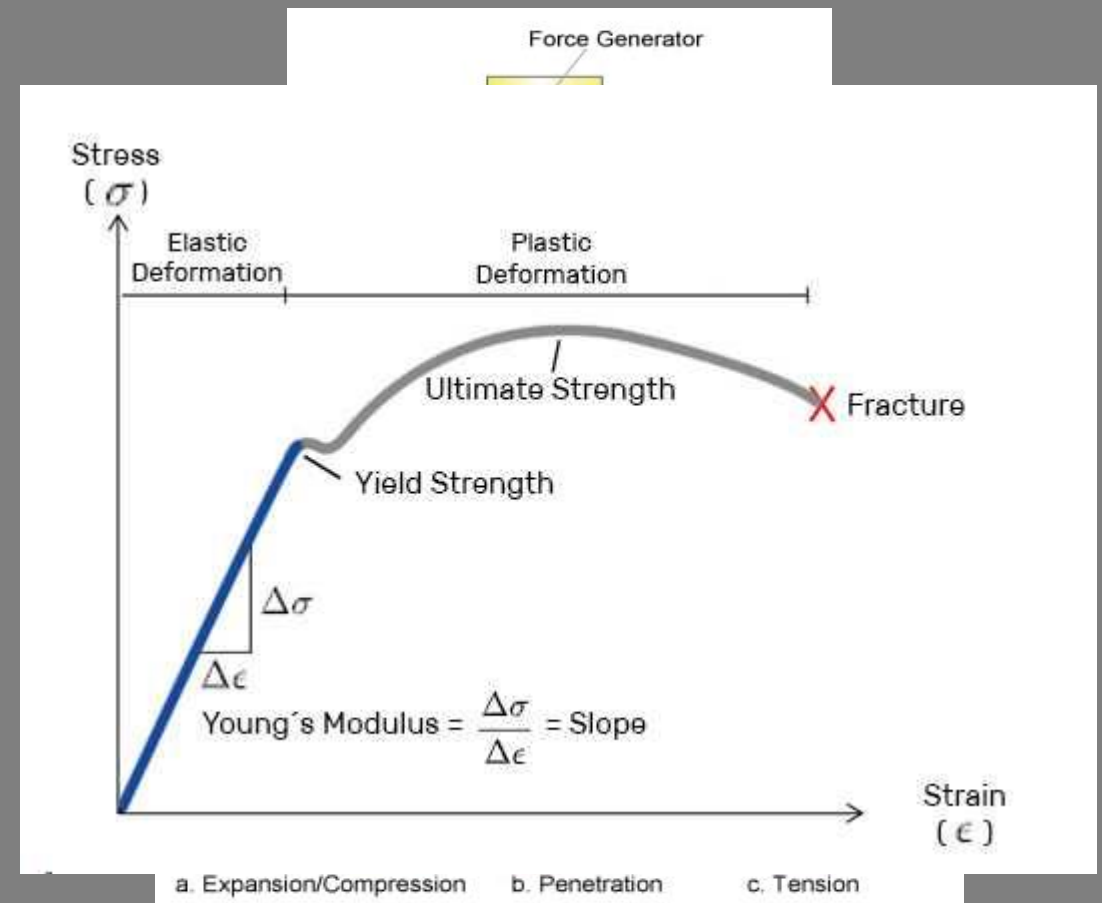
Changes in a dimension of a sample as a function of temperature, time, force and atmosphere . Force can be applied in compression, tension, flexure or torsion modes using different probes (quartz glass, silica, alumina and metals)

Sample preparation

Smooth, less of 10mm of height, silica probe, heating rate of 5k/min, nitrogen atmosphere, ramped force

Use

Mechanical properties of the hydrogel at body conditions (Elastic modulus – 0.45 a 0.8MPa)



The Summary

Characterization techniques in medical applications

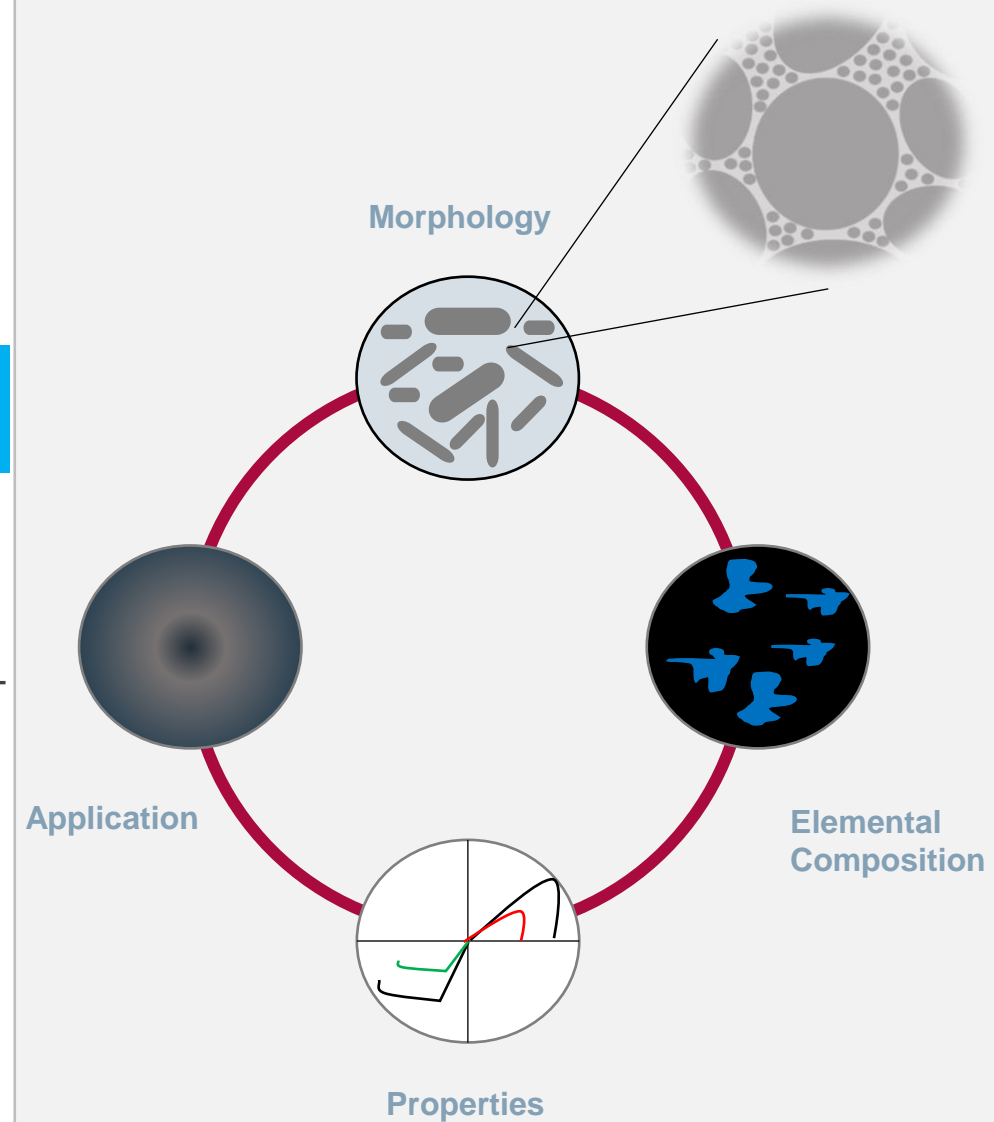
- Challenges of joint replacement processes
- Study of synthetic articular cartilage and lubricant with nano-additives

1

Wear, composition, porosity and performance

2

Mechanical properties that guide the applications



FINAL

Project

Characterization of materials

Thank You

Q/A