10-3.- Structural Characterization of different Composites

ECOCOMPOSITES FROM REUSED TYRES
A good solution to avoid disposal in landfills

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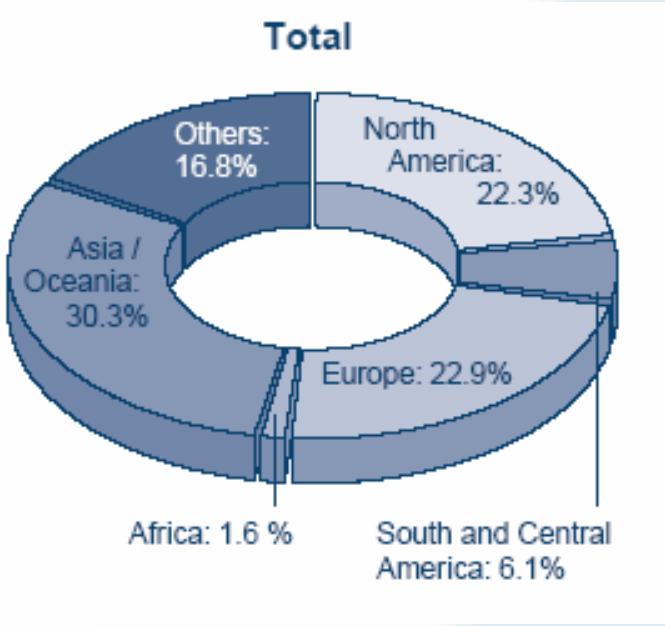
Tyres in the world



	1999-2003	Landfills
Europe	273-279 (6)	38%
USA	284-292 (8)	10,3%
Global	1129-1224 (95)	46%

Landfills Directive in the European Union

“ The EU Landfill Directive will ban the disposal of tyres to landfill. Whole tyres will be banned from July 2003 and shredded tyres from July 2006...”

What can we do with the tyres?

We propose a new composite material made up from a polyolefine matrix (recycled HDPE bottle water) and reinforced with reused tyre as another way to reduce the stock of tyres in order to obtain sustainable composites.

What's the AIM of our work?

Study the influence of different parameters:

- Surface pretreatments: chlorination, H_2SO_4, silane coupling agent
- Reused tyre content

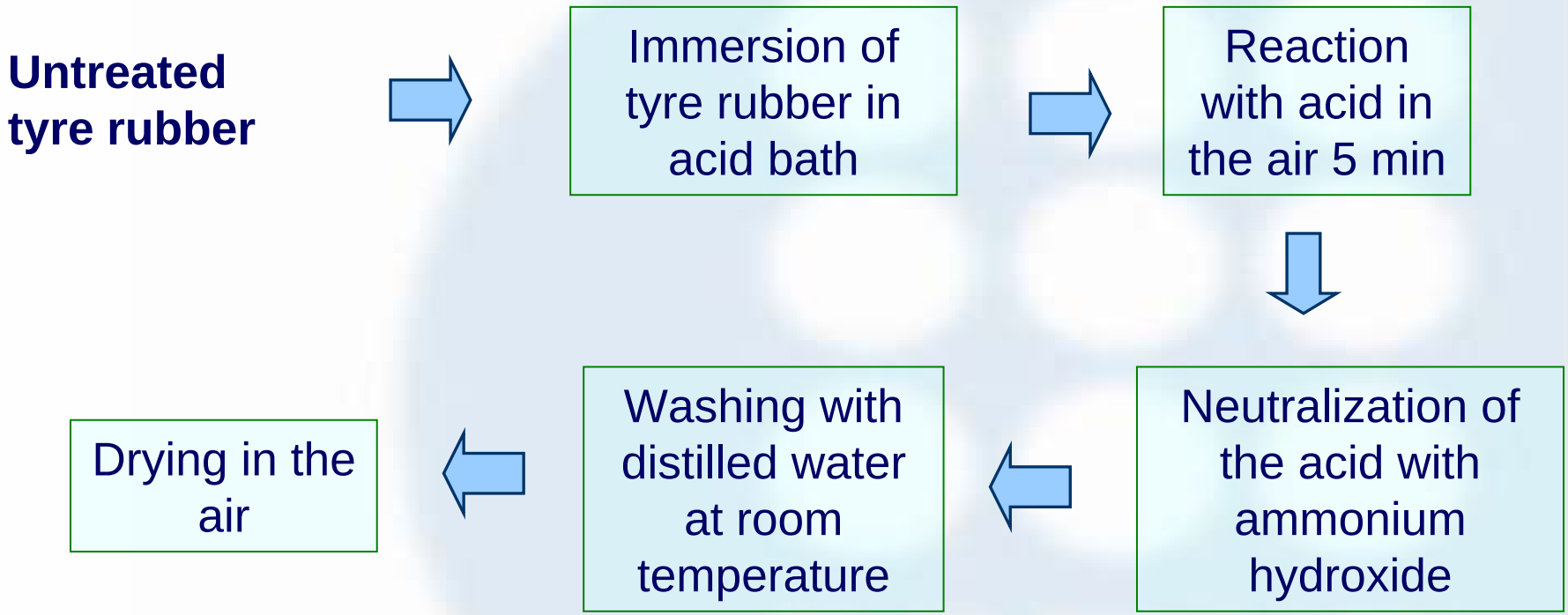
on Mechanical properties and structural changes of new composites

Materials

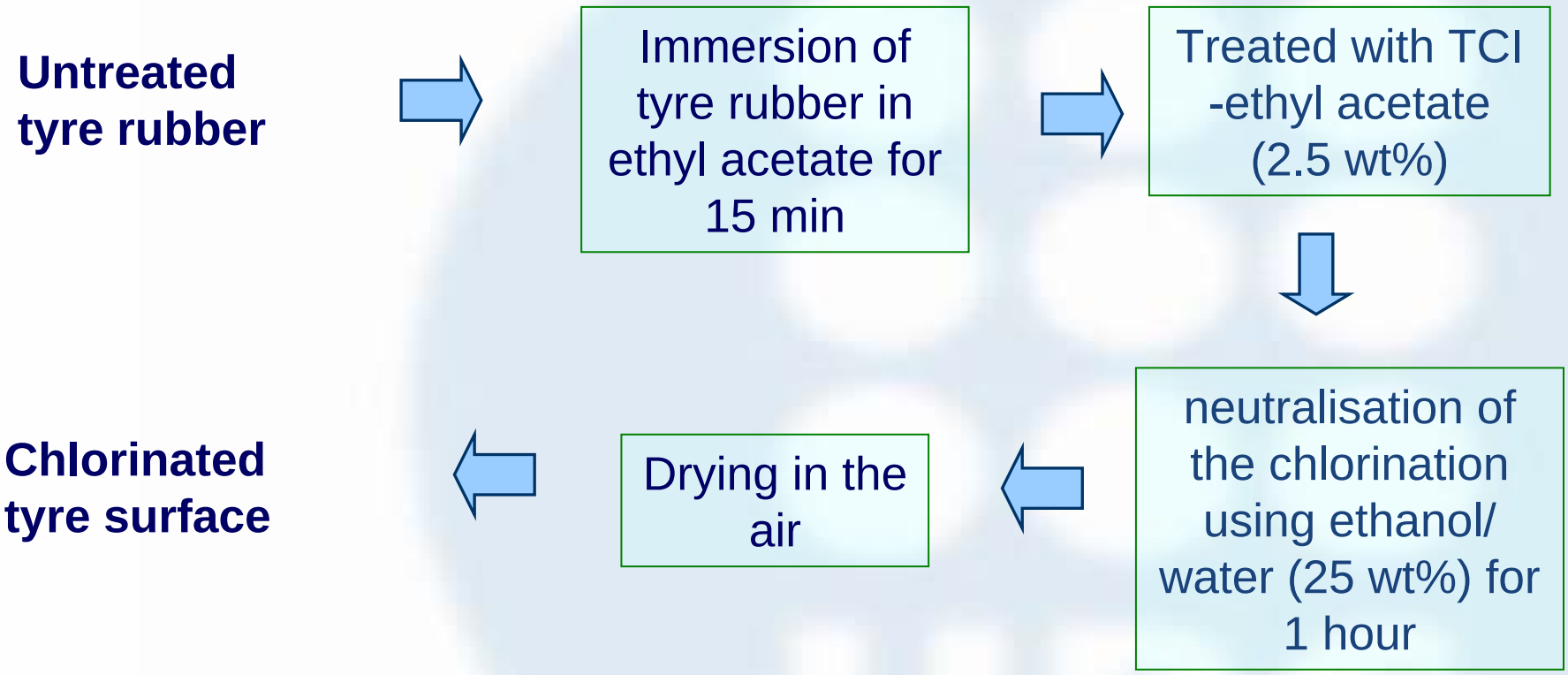
- HDPE (from recycled botlle water) — MFI = 2.75 g/min, ρ= 940 kg/m^3
- Reused tyres — micronized average particle size: 500 μm
- Treatment process — TCI, coupling agent and H_2SO_4

TCI (trichlorisocyanuric acid)

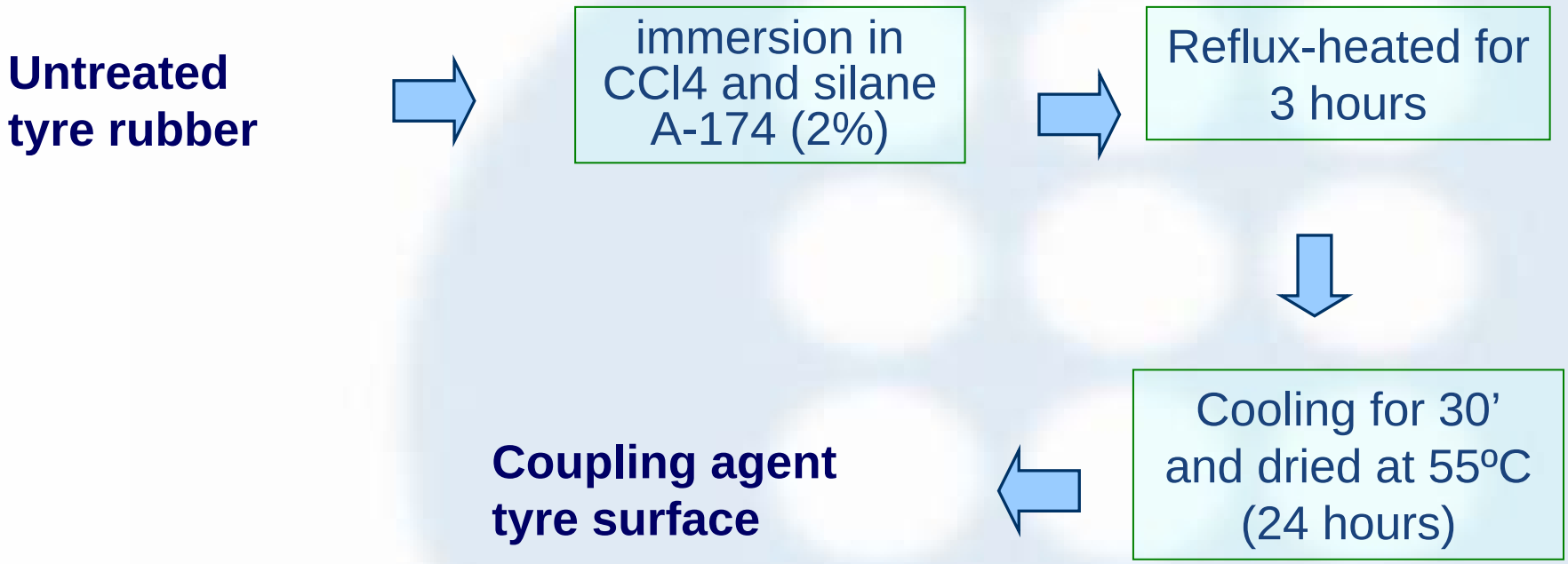
PRETREATMENTS : H_2SO_4



PRETREATMENTS : chlorination



PRETREATMENTS : silane coupling agent



Manufacturing process

Homogenity
Mesh of HDPE +
Reused tyre

Hot press moulding at 400 kN , 170ºC for 15 min.

COMPOSITE

mixing process was carried out in a two-roll mill heated at 153ºC

Five rubber-HDPE compositions (5,10,20,30,40%)

Structural Characterization

- Fourier Transform Infrared spectroscopy (FTIR)

- Thermogravimetric analysis (TGA)

Under nitrogen atmosphere

STRUCTURAL CHARACTERIZATION

Scanning Electron Microscopy

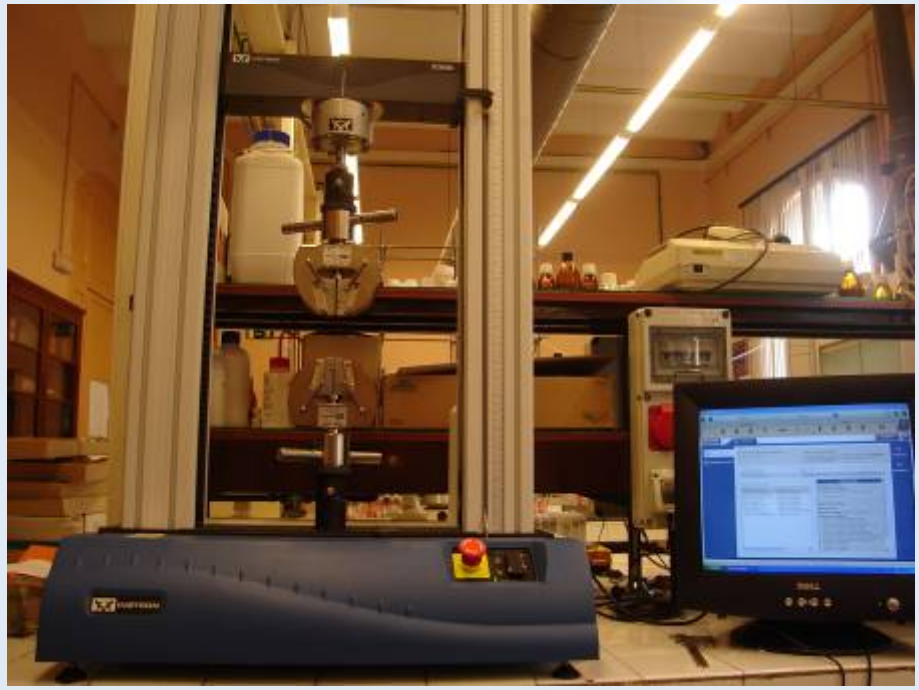
- The surface of reused tyres was examined with a Zeiss DSM 960

- Dry samples were coated with a thin layer of carbon before observation under the microscope, in order to increase the sample conductivity

MECHANICAL CHARACTERIZATION

Mechanical testing

Tensile Strength
Young's Modulus
Toughness
Elongation at break



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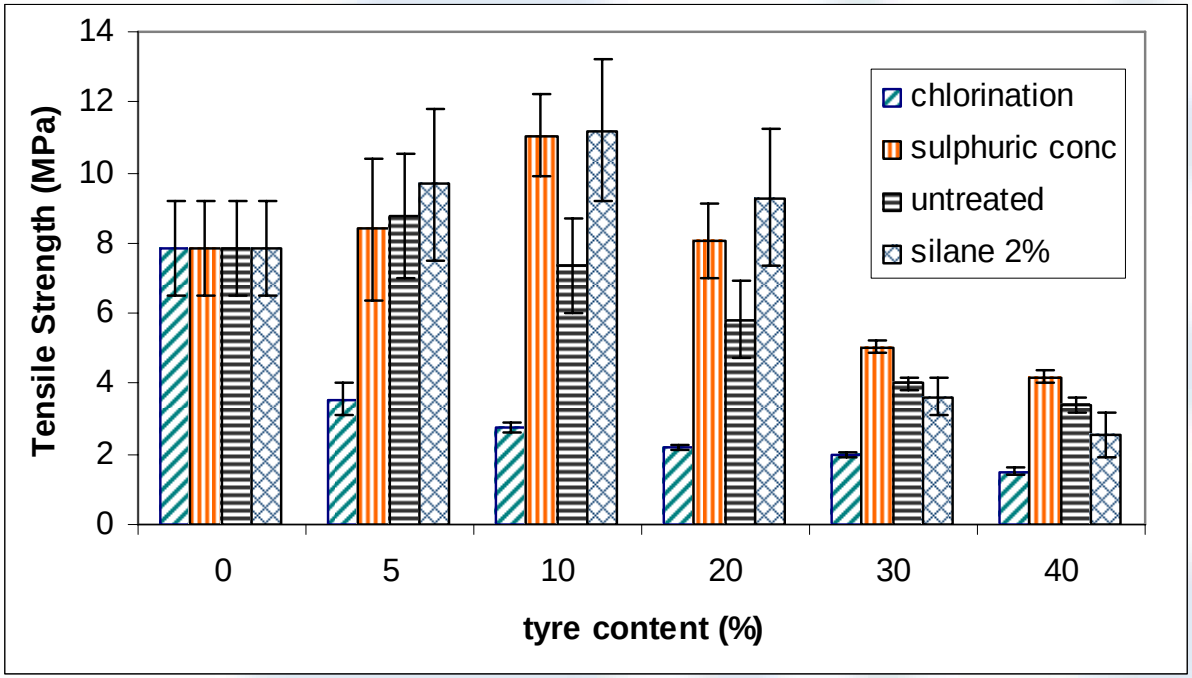
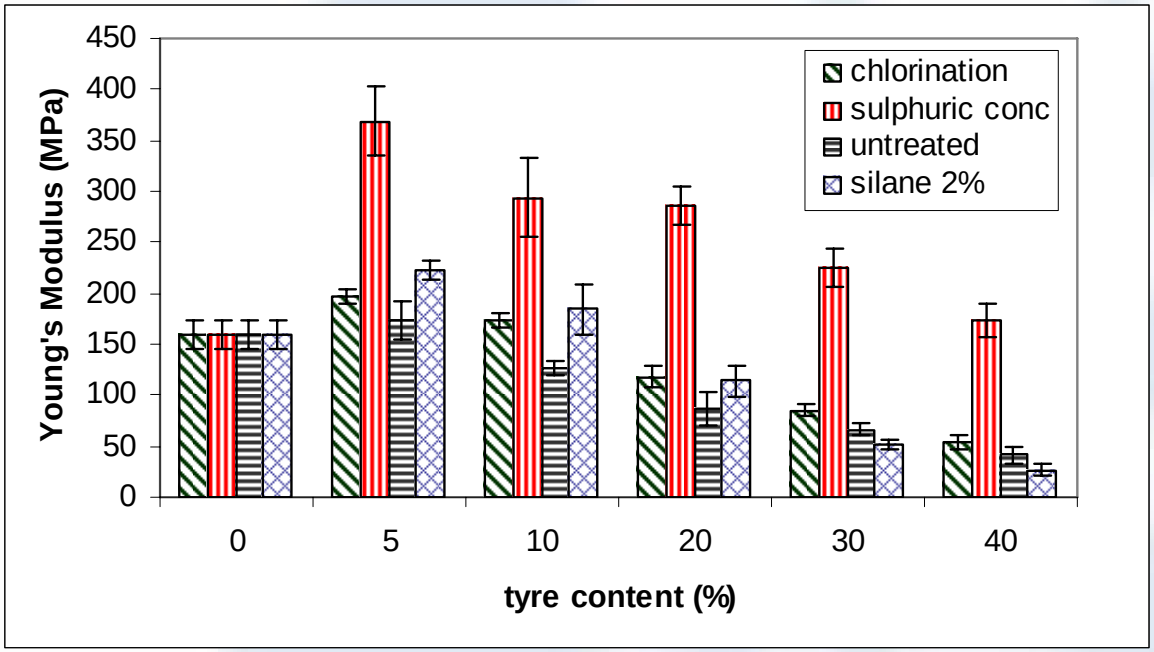
Mechanical Properties

Tensile Strenght



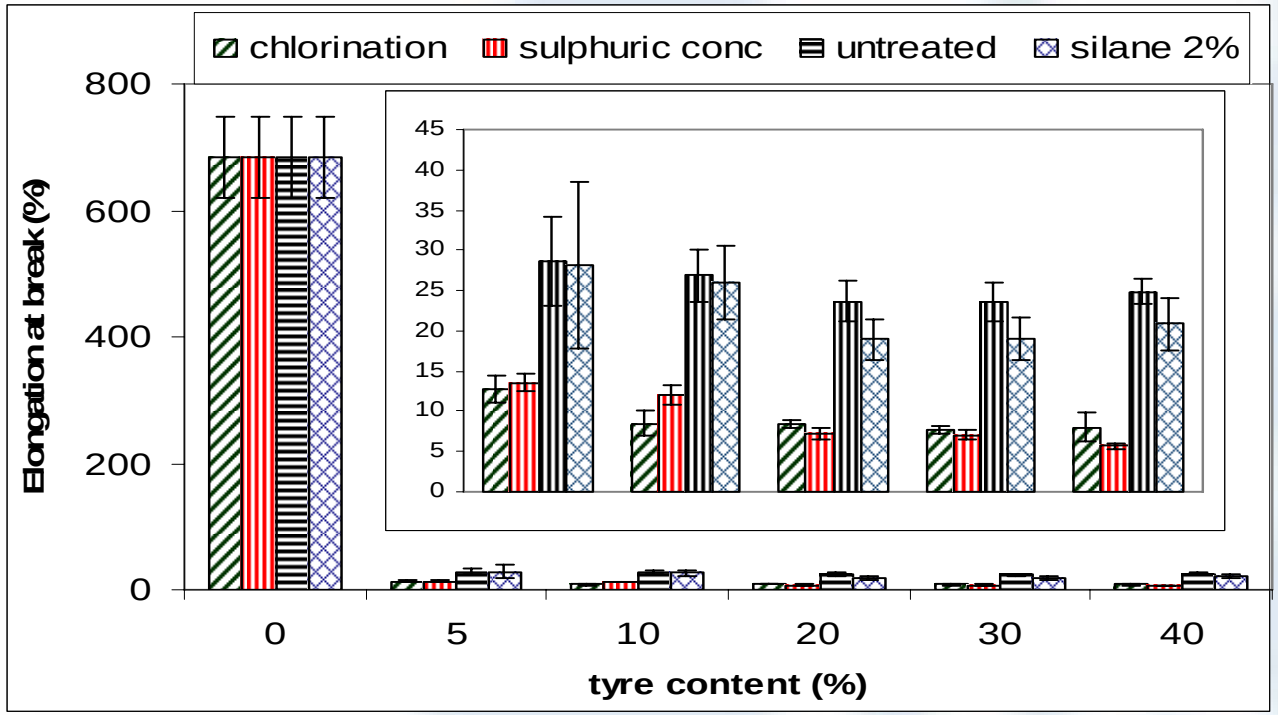
Mechanical Properties

Young's Modulus



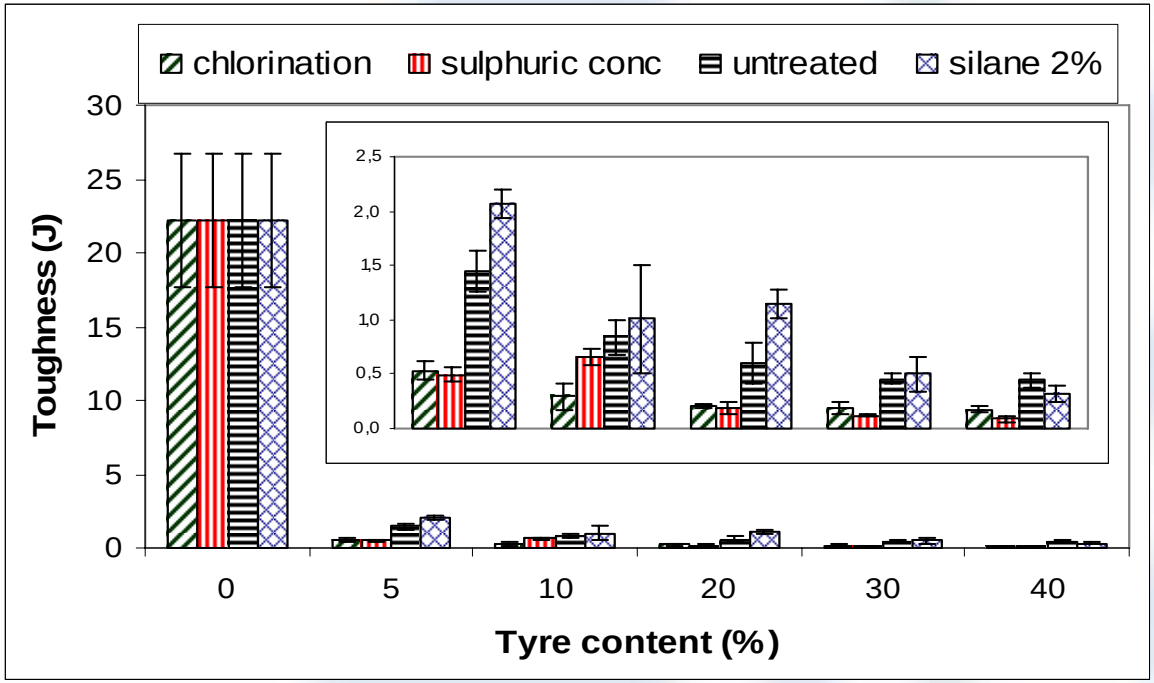
Mechanical Properties

elongation at break



Mechanical Properties

Toughness



MECHANICAL PROPERTIES REMARKS I

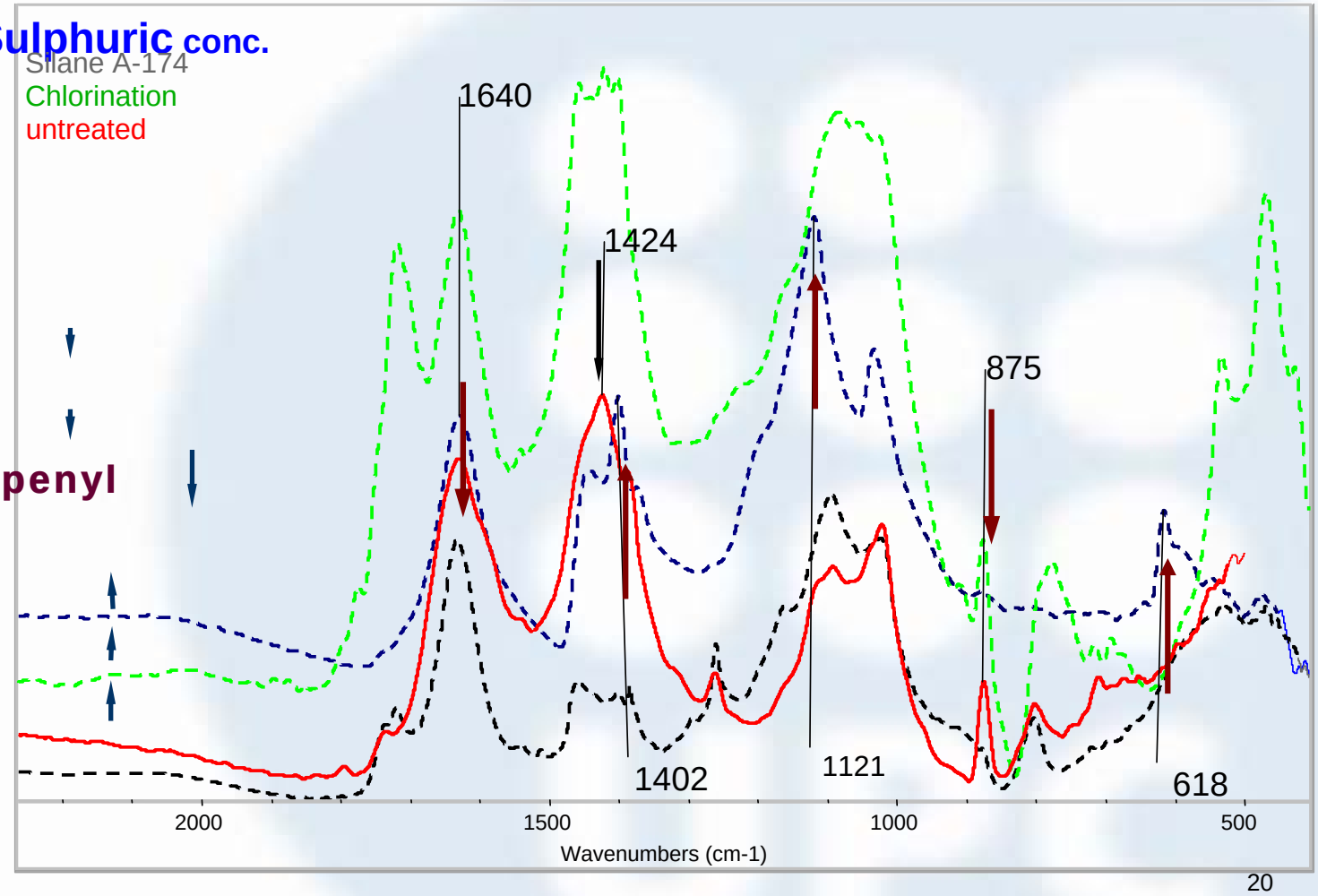
- The addition of reused tyre rubber to the HDPE in contents lower than 20% acts as a filler, improving the tensile strength stiffness and providing a brittler behaviour.

- Contents of tyre rubber above 20%, using either tyre not treated or tyre treated with different treatments, obtain composites samples with lower values of Tensile Strength, Young's Modulus, Elongation at break and toughness than recycled HDPE.

- Tyre rubber treated with H2SO4 and silane A-174 improve the interaction ability of rubber with HDPE, yielding to higher stiffness of these composites.

Spectral results of H_2SO_4 treated tyre

ref: red spectra of untreated tyre



1630 C=C

1424 CH_2

875 *t*- isopropenyl

1402 O=S=O

1121 O=S=O

618 O=S=O

Spectral results of silane treated tyre

ref: spectrum of untreated tyre

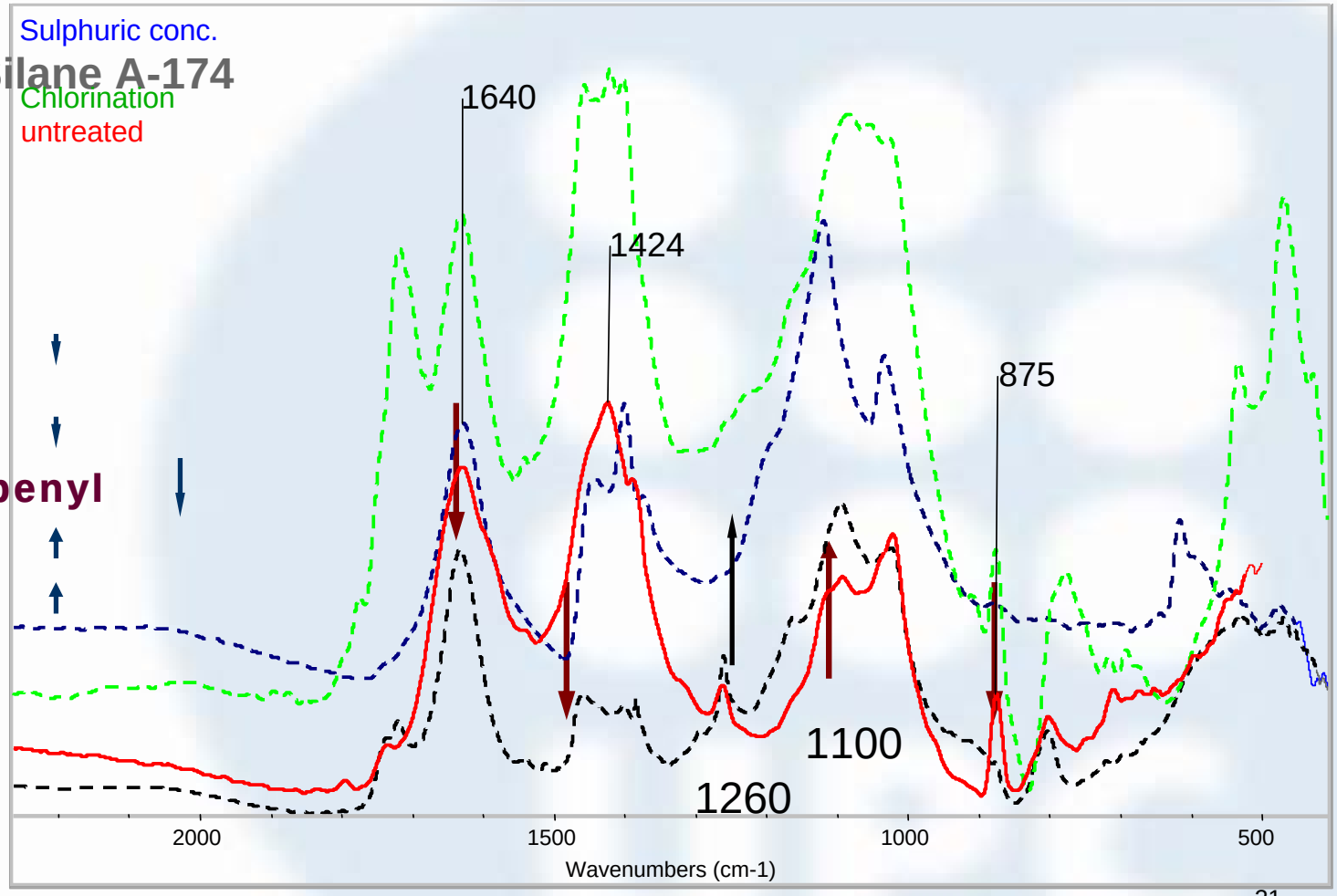
1630 C=C

1424 CH2

875 *t*- isopropenyl

1260 Si-O

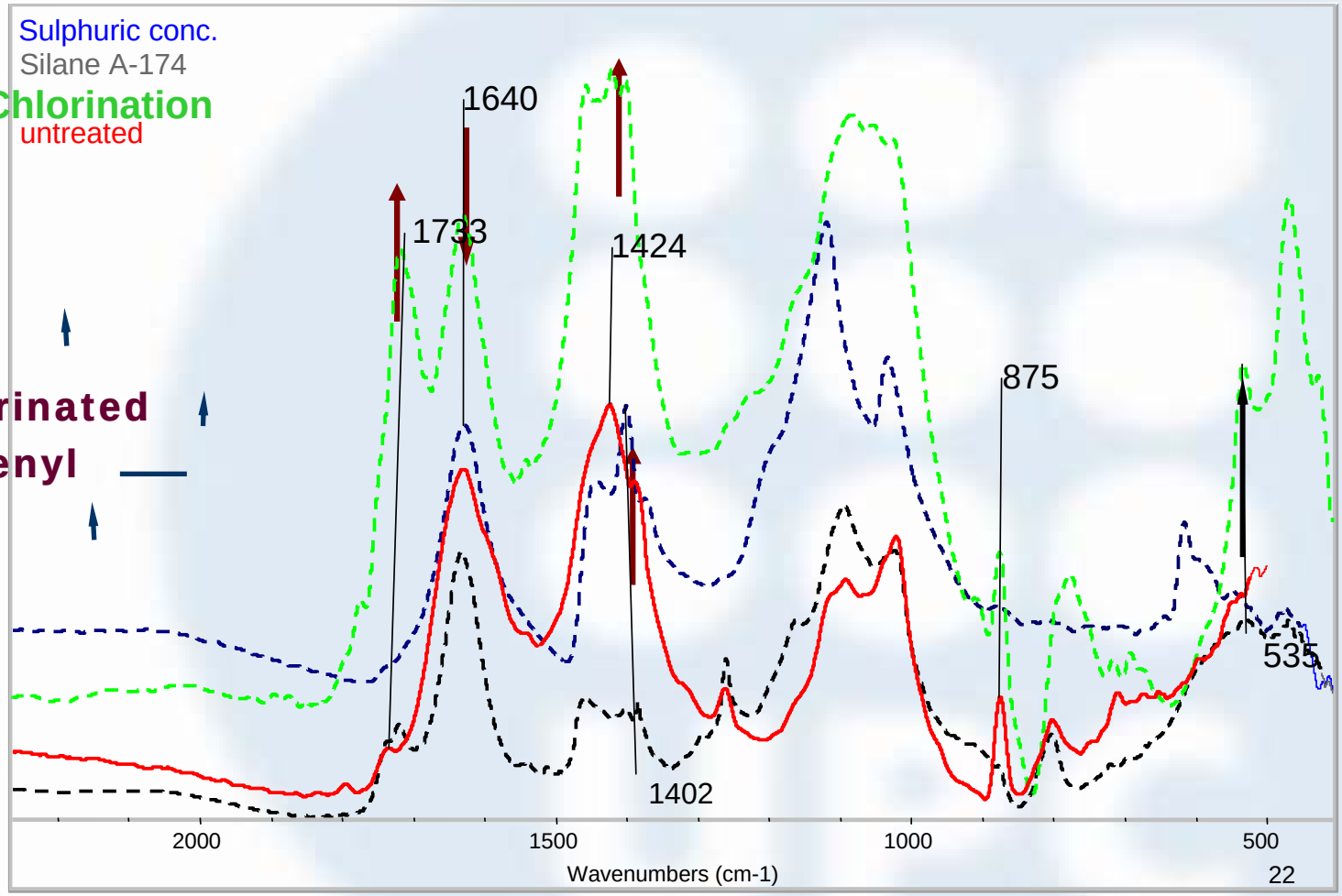
1100 Si-O



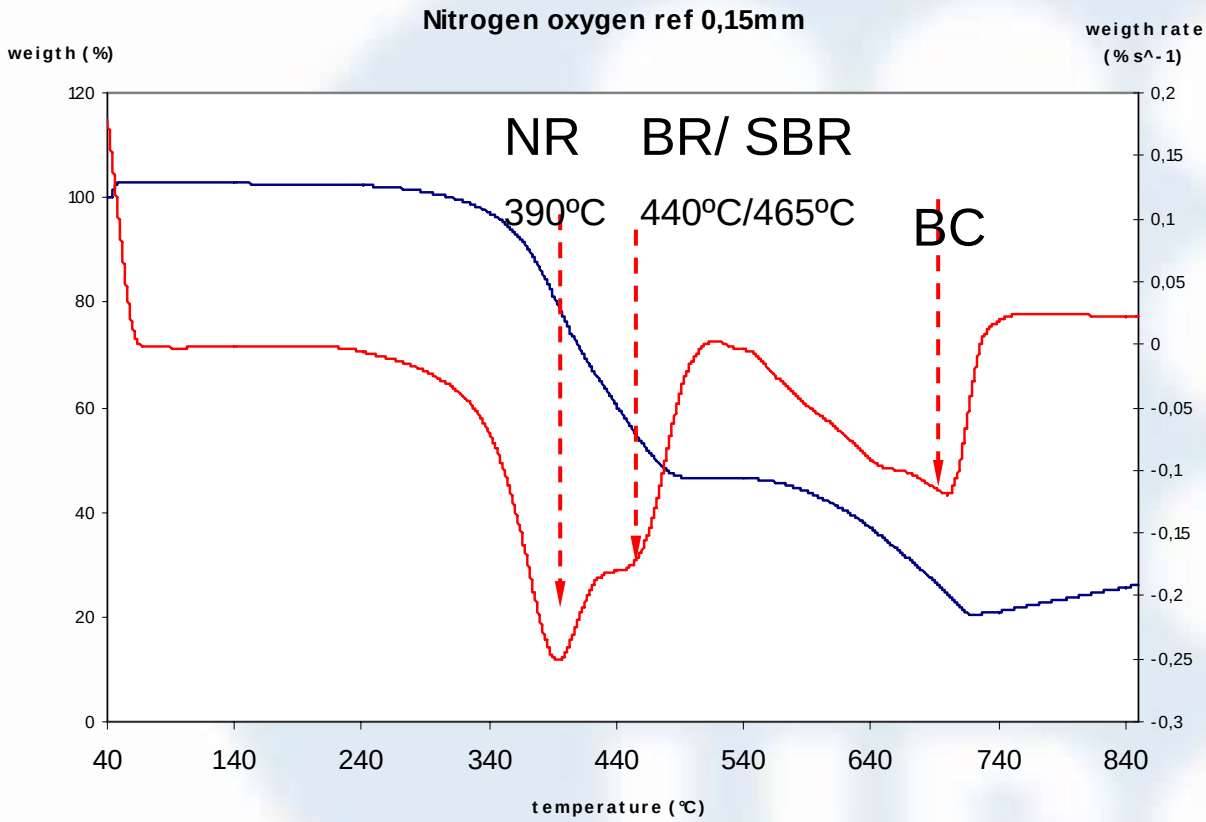
Spectral results of chlorination treated tyre

ref: spectrum of untreated tyre

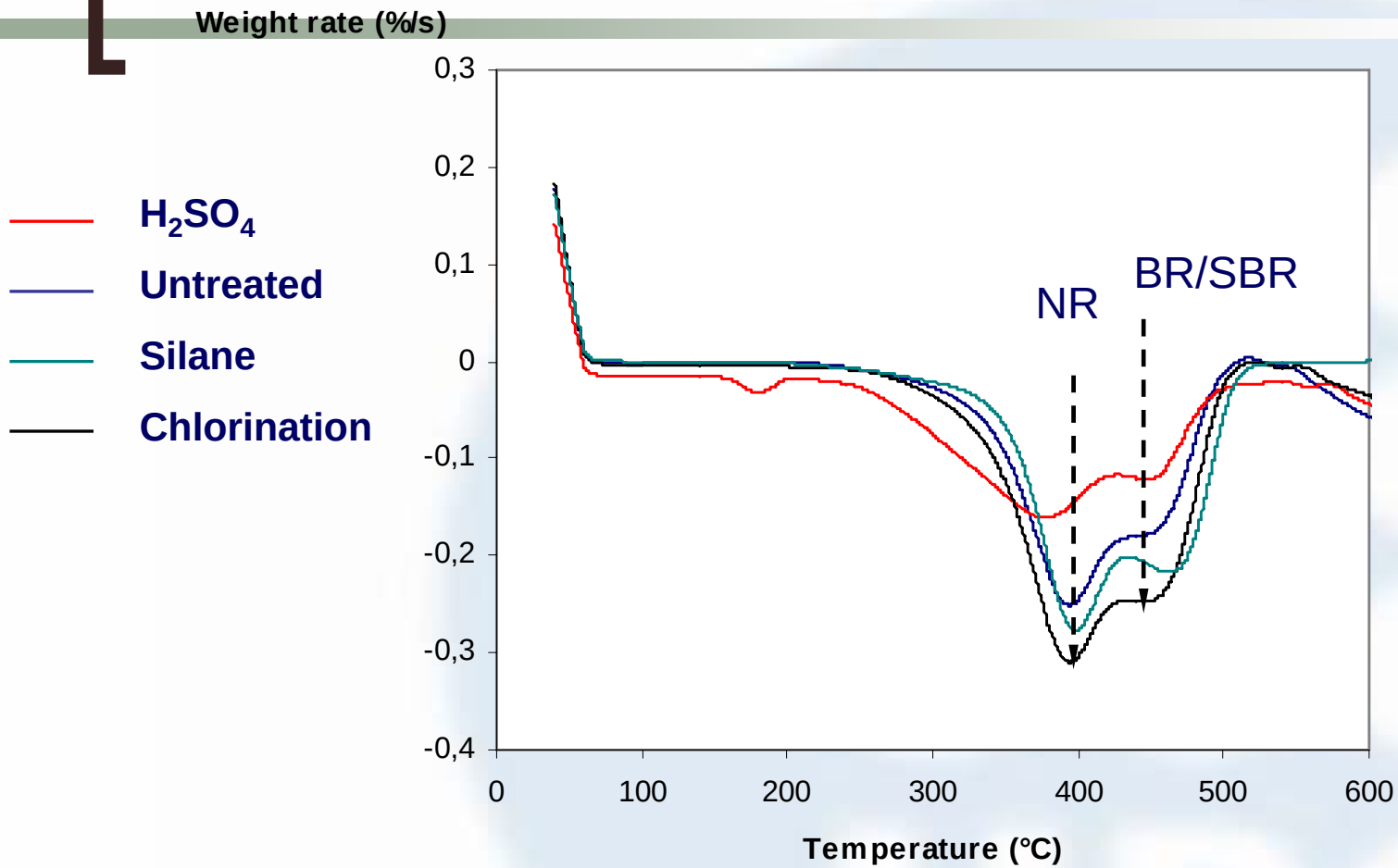
1730 C=O
1630 C=C
1424 CH2 chlorinated
875 *t*- isopropenyl
534 C-Cl



Thermo gravimetric analysis
corroborate the FTIR results



Thermo gravimetric analysis
first derivative



TGA corroborate that H_2SO_4 treatment define the largest modification

FTIR and TGA remarks

By studying the FTIR and TGA, the main results obtained were the following:

- Every acid treatment studied produces a specific chemical modification on the rubber, thus inducing the formation of specific groups;

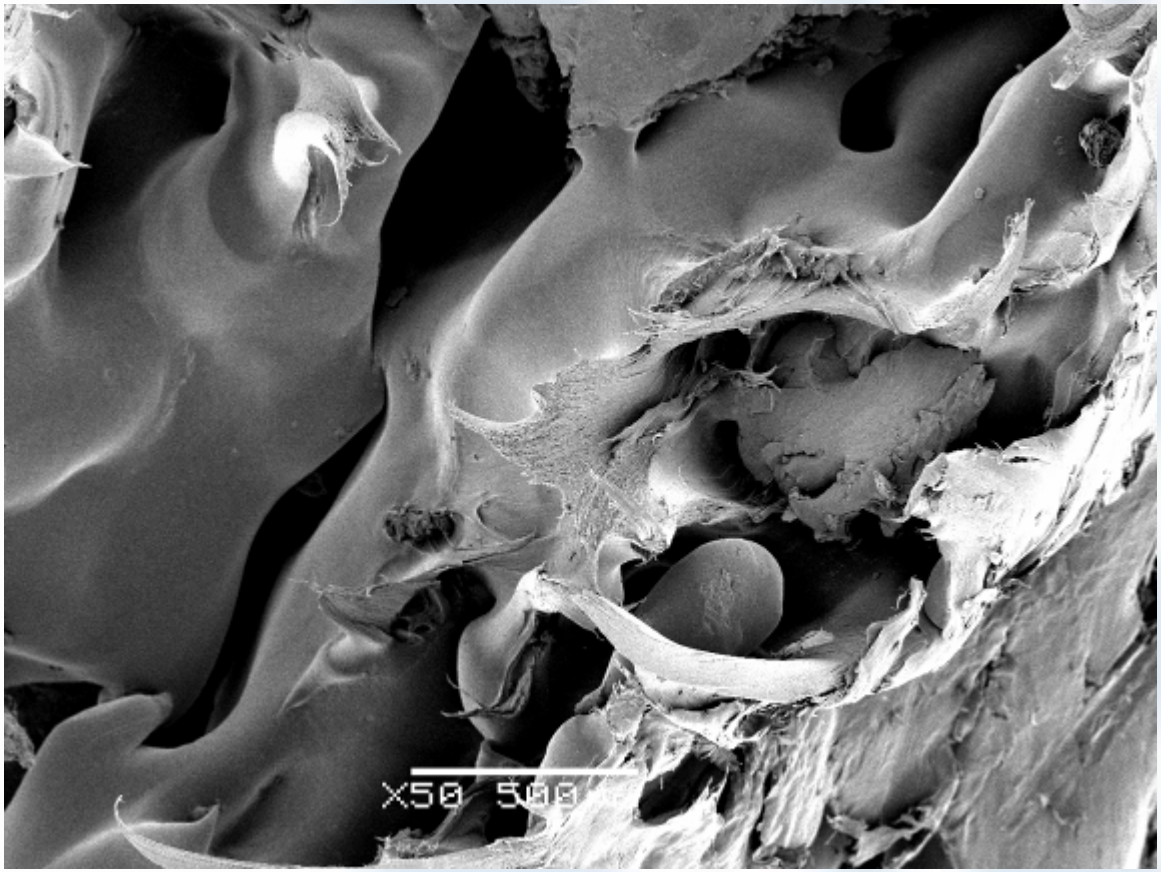
- Several degradative effects appear in a similar way and do not depend on the acid used in the treatment (mainly the decrease in double bonds and the elimination of minor components and moieties);

- TCI acid increase the energy surface and have negative influence in adhesion with recycled HDPE.

- The coupling agent developing interactions with the rubber and interpenetrates with the matrix, improving the ability to establish a link between the material's components.

SEM Characterization

reused tyre untreated

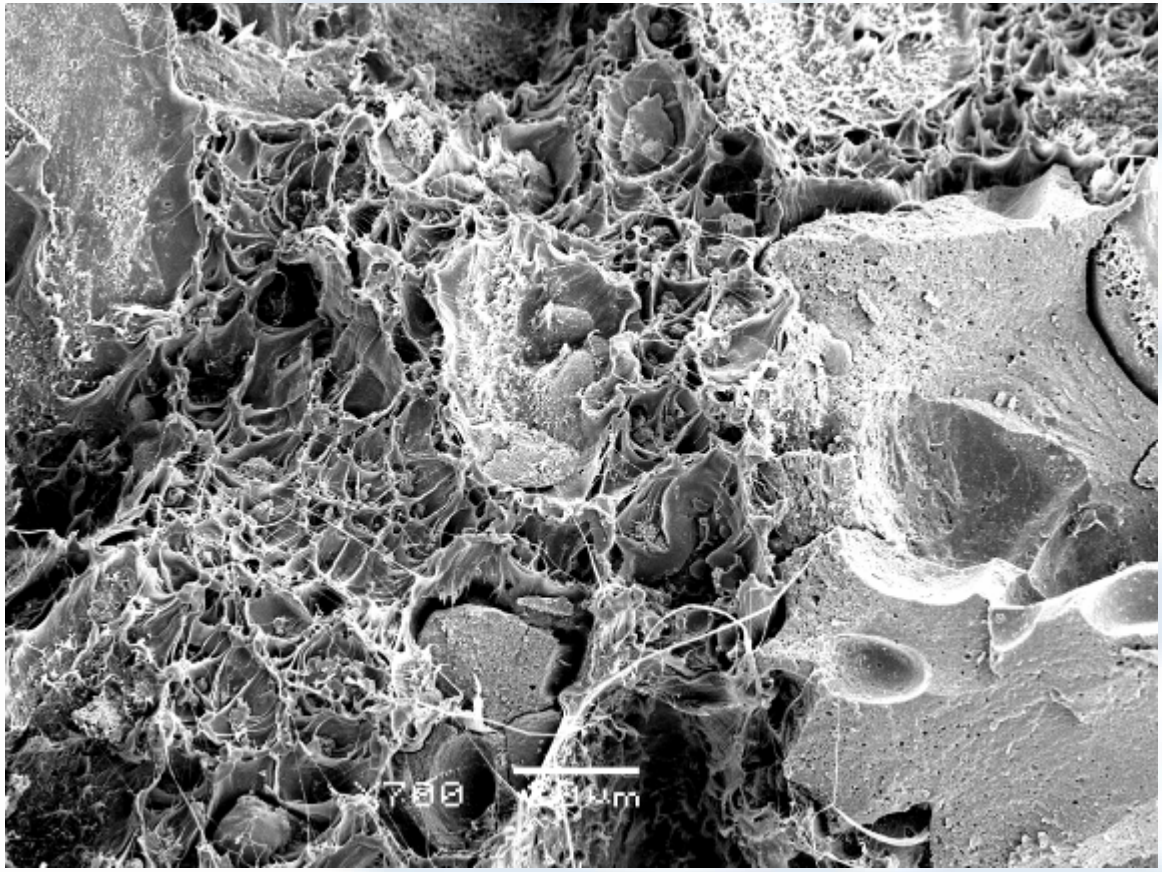


SEM Characterization

reused tyre treated with H_2SO_4

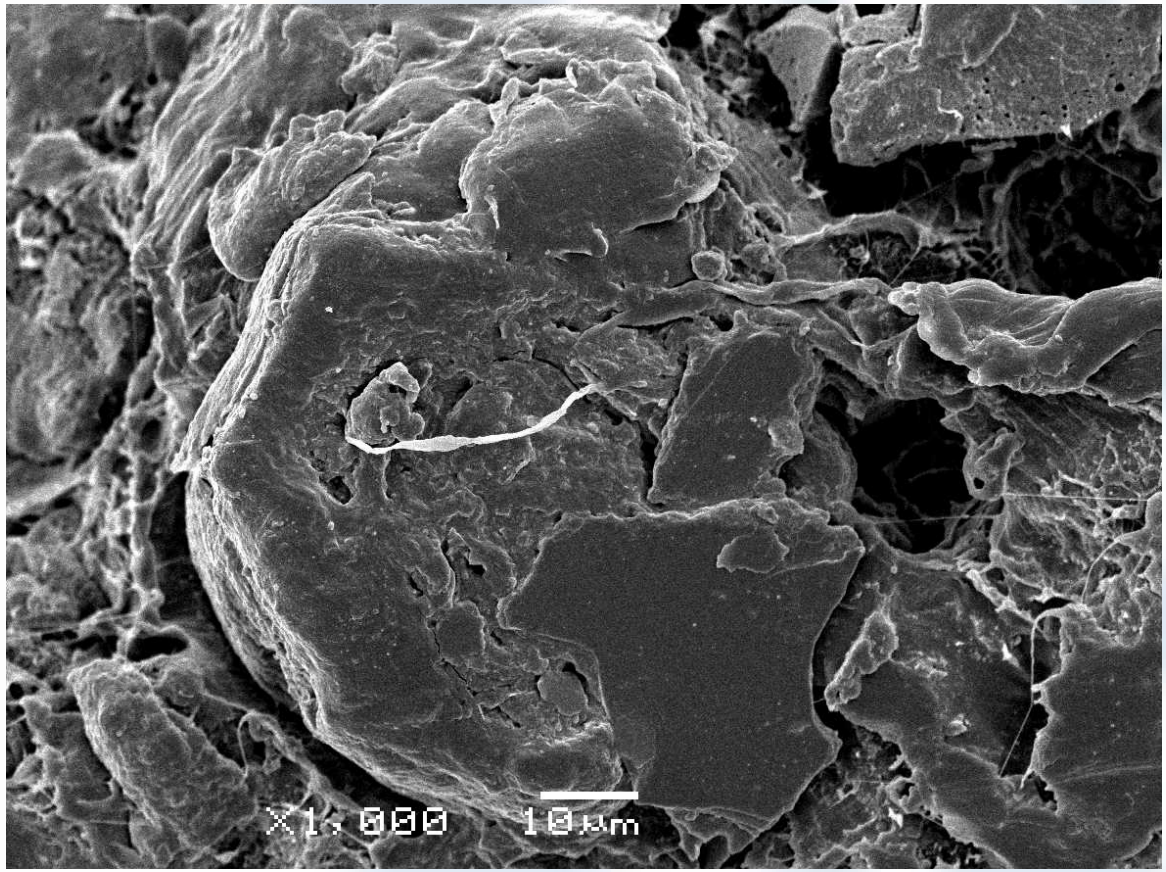
SEM Characterization

reused tyre treated with silane



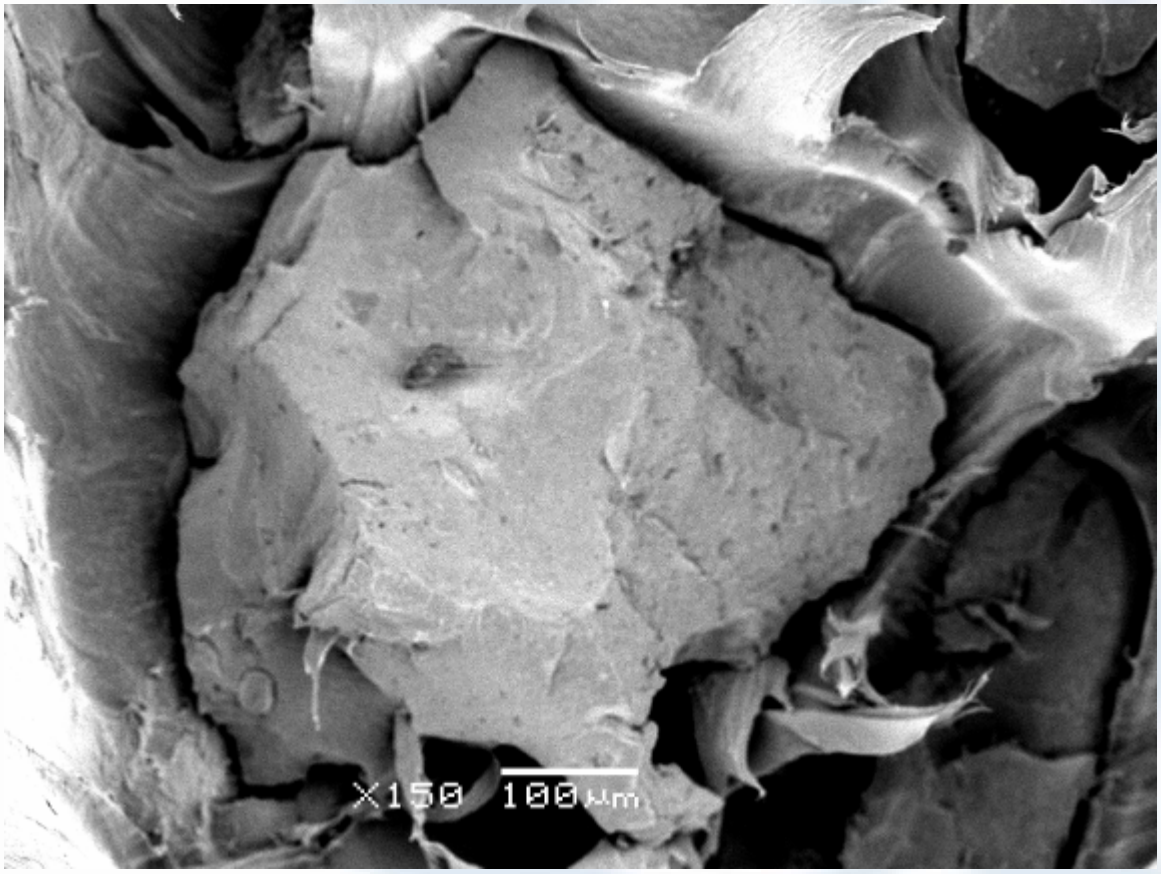
SEM Characterization

reused tyre treated with silane



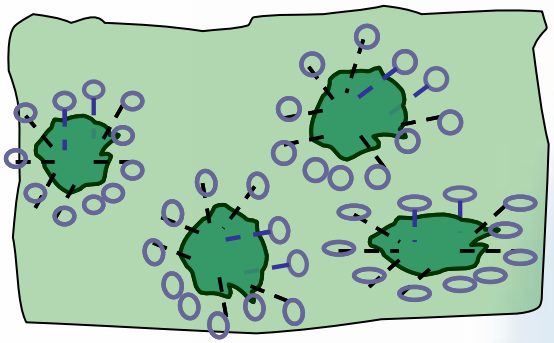
SEM Characterization

reused tyre treated with chlorination

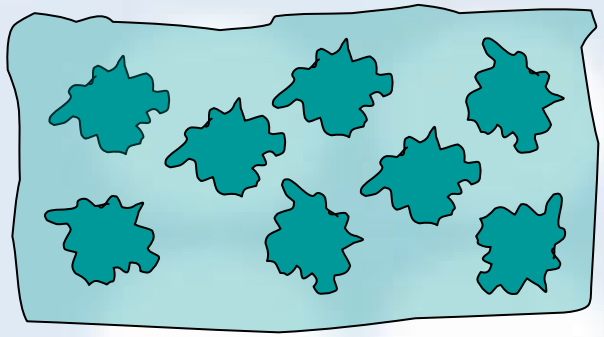


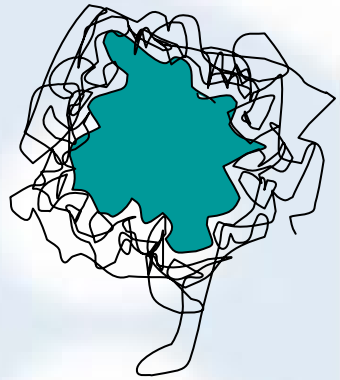
Mechanism of adhesion

Chlorination process



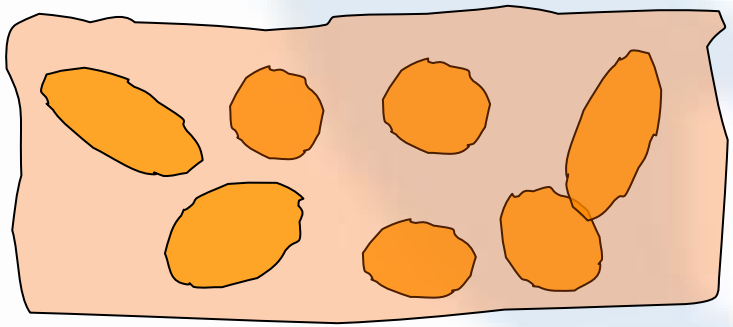
Tyre modified with H_2SO_4

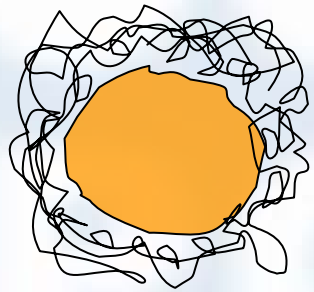




Pyshical bonding with interdiffusion of macromolecules inside the microcavities

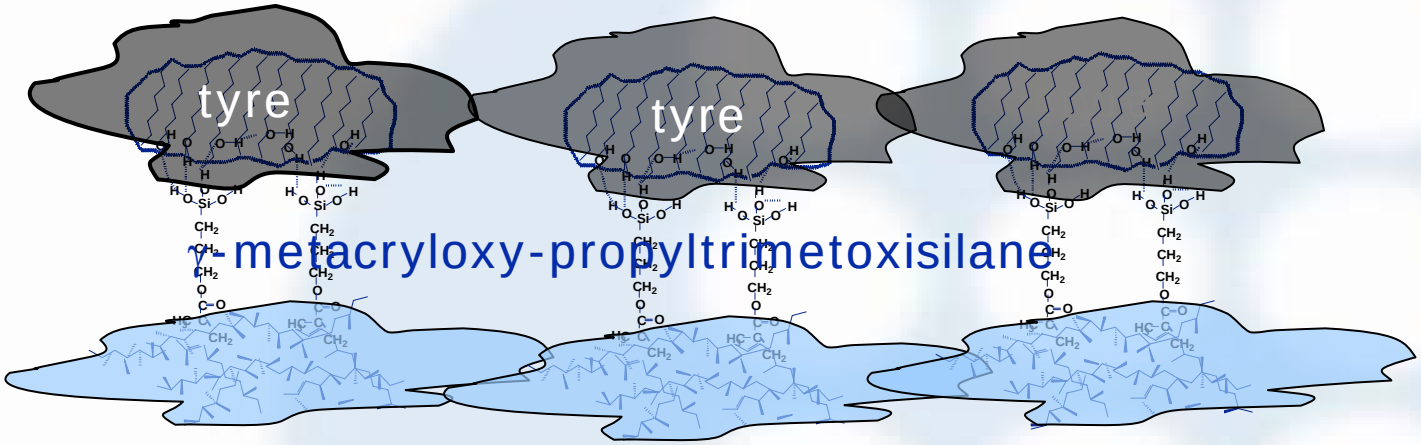
Unmodified Tyre





mechanism for silane

chemical mechanism



The composites treated with silane present a chemical mechanism of adhesion, including the formation of secondary bonds between silane and HDPE and “probably” chemical bonds with reused tyre

conclusions

• The nature and intensity of the chemical modifications produced by H_2SO_4 treatment, and the surface microroughness that it generates on the reused tyre rubber, makes the use of H_2SO_4 more convenient for adhesion with the polyolefine matrix that any other studied treatment.

• This may be a consequence of the rigidization of the rubber after the acidic treatment due to the extraction of additives and the degradation process.

• Treatment with silane improves the ability of interacting of both components obtaining better results than recycled HDPE at concentrations lower than 30%.

conclusions

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- The untreated composites present a very weak physical-mechanical adhesion due that its present a smooth surface.

- In composites treated with chlorinate process, the chemical modifications are very important, although the presence of polar groups reduce the interaction between matrix and tyre.

Thank you very much
for your attention