PREPARATION AND CHARACTERIZATION OF BIO-BASED THERMOSET NANO COMPOSITES

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NSO Nanostructures and Self-organization WG

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SUMMARY

Motivation

Bio-based Resins with Potentialities in Aviation

Materials

Functionalization of Carbon Nanotubes

Mechanical Characterization

Conclusions

MOTIVATION

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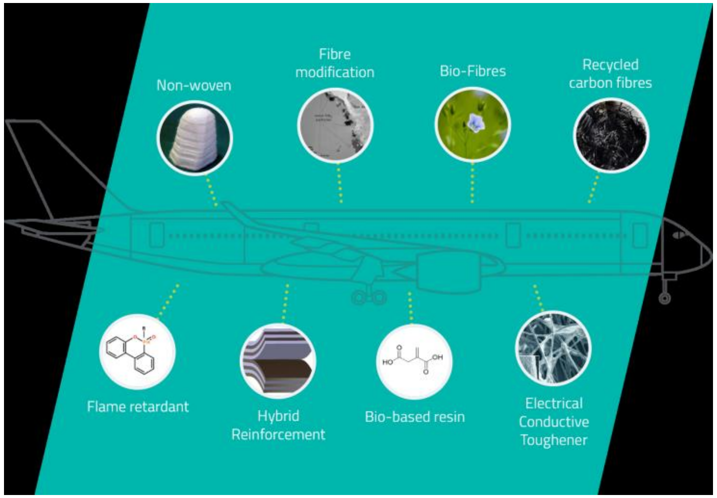
- The present work was developed within the ECOCOMPASS project



[1]

Objective:

- Investigating bio-sourced resins for developing greener composite aircraft interior components and secondary structures
- Enhancing the electrical properties of the composites by modifying the bio-based polymer resin matrix by filler incorporation

[1] Ph. Credits: https://cordis.europa.eu/result/rcn/223841_en.html, accessed on 2018/11/02

BIO-BASED RESINS WITH POTENTIALITIES IN AVIATION

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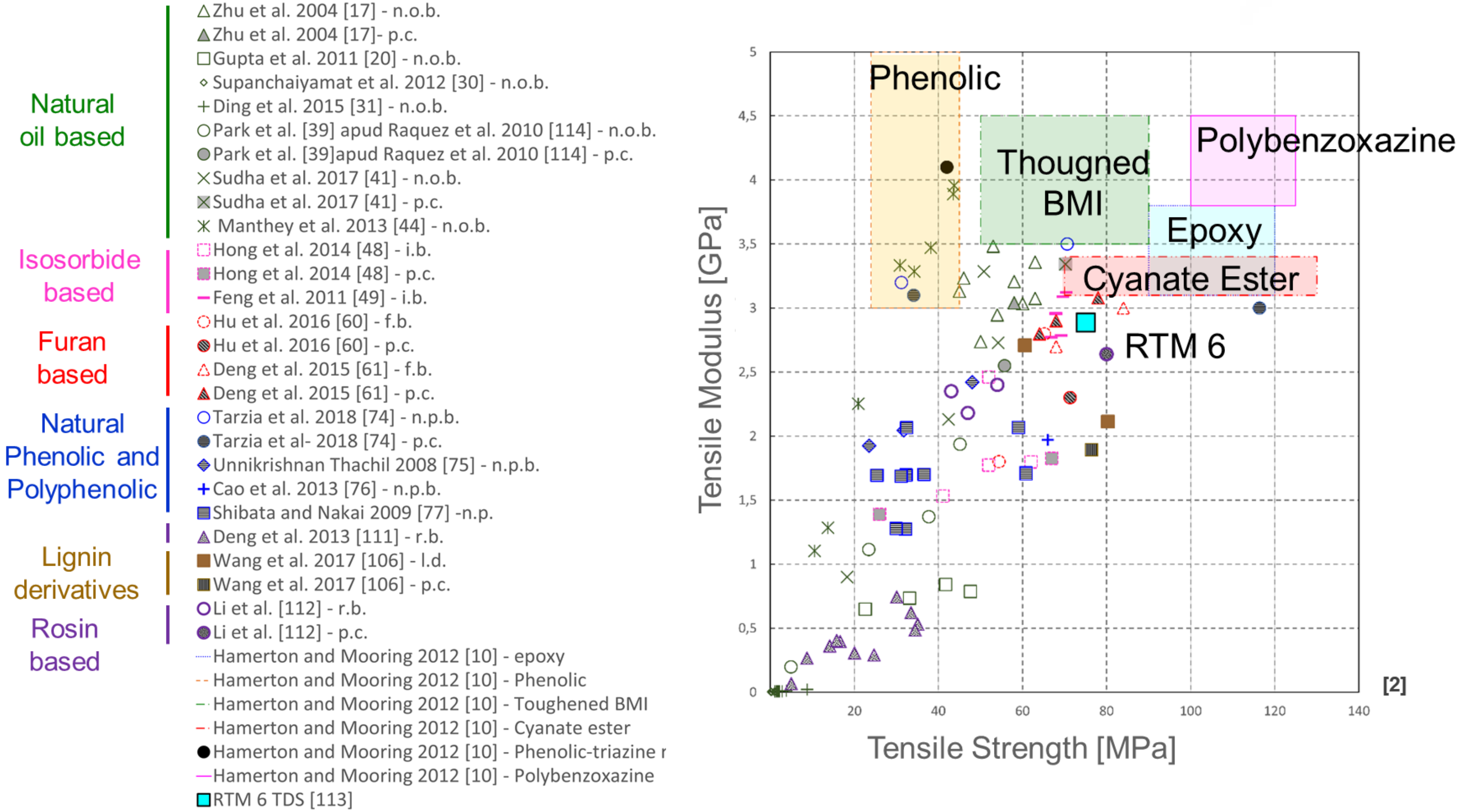
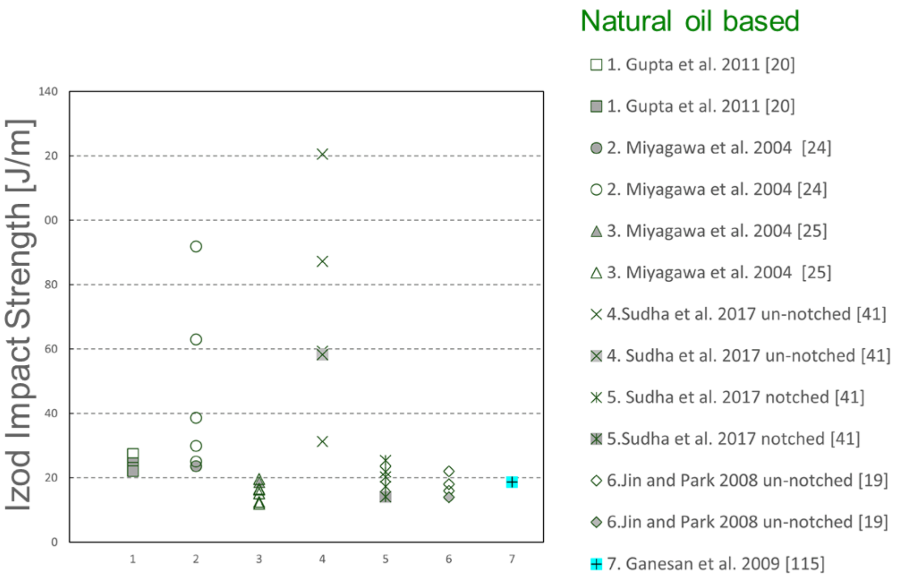
Comparison of tensile properties of bio-based and petroleum-based resins



[2] Ramon, E.; Sguazzo, C.; Moreira, P.M.G.P. A Review of Recent Research on Bio-Based Epoxy Systems for Engineering Applications and Potentialities in the Aviation Sector. *Aerospace* 2018, 5, 110

BIO-BASED RESINS
WITH POTENTIALITIES IN AVIATION

Impact properties of natural oil-based resin compared to petroleum-based resin



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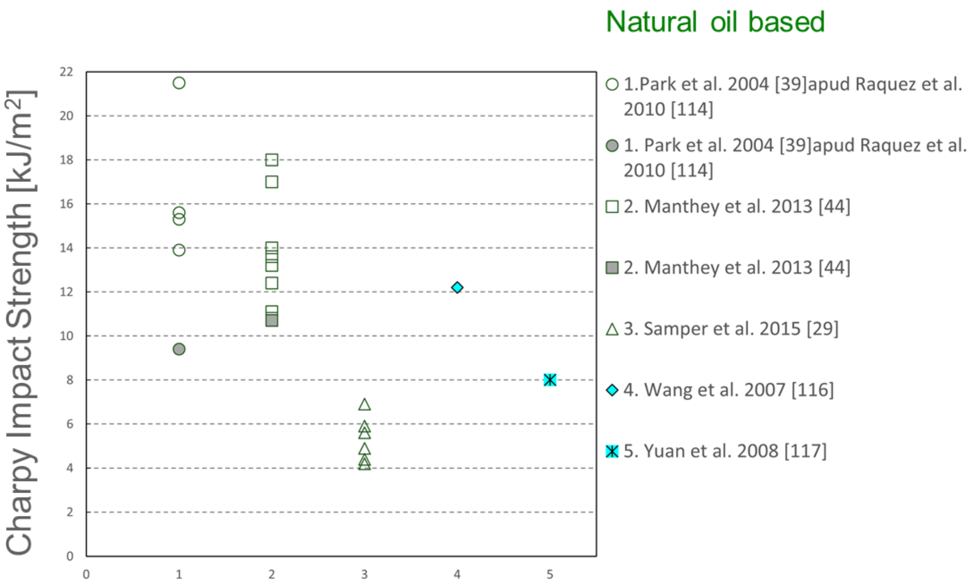

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MATERIALS

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Epoxy Matrix

- Epoxy resin with high bio-based carbon content
- 35% of molecular structure from plant origin
- Epoxy/hardner system: SR GreenPoxy 33/SZ 8525 by Sicomin – France

Fillers

- Multiwalled Carbon Nanotubes (MWCNTs) synthetized by Catalytic Chemical Vapor Deposition (CCVD) process - NANOCYL® NC7000™ - Belgium
- High electrical conductivity
- Key Applications: Transportation including Aeronautic sector and EMI-shielding

FUNCTIONALIZATION OF CARBON NANOTUBES

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Industrial grade MWCNT NC 7000™

- No surface modification → functionalization is needed
- Average Diameter 9.5 nm
- Average Length 1.5 µm
- Carbon Purity 90%
- Transition metal oxide <1%
- Surface Area 250-300 m^2/g
- Volume Resistivity 10^{-4} Ω.cm
- Low percolation threshold of 0.5 wt% and 4.5 wt%

Advantages

- Improvement of the dispersion and the interfacial bonding of MWCNTs in the epoxy matrix

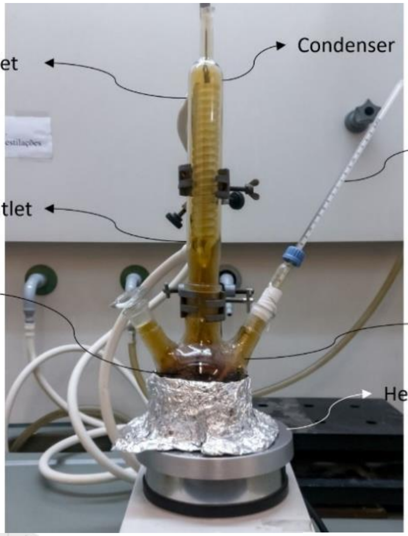
Parameters affecting the chemical functionalization of the MWCNTs

- Concentration of the mixture acids (H_2SO_4 + HNO_3)
- Time of heating
- Temperature of the mixture
- Quantity of the MWCNTs used

FUNCTIONALIZATION OF CARBON NANOTUBES

Oxidation Methodology for Functionalization by –COOH

- H_2SO_4 and HNO_3 taken in a mixture of 3:1 (100 ml)
- Time of heating 30 minutes
- Temperature of mixture from 90-100 °C
- 1 g of MWCNTs is used for functionalization
- This mixture is heated in reflux by using the shown apparatus
- After 30 minutes the mixture is cooled by means of ice bath until room temperature



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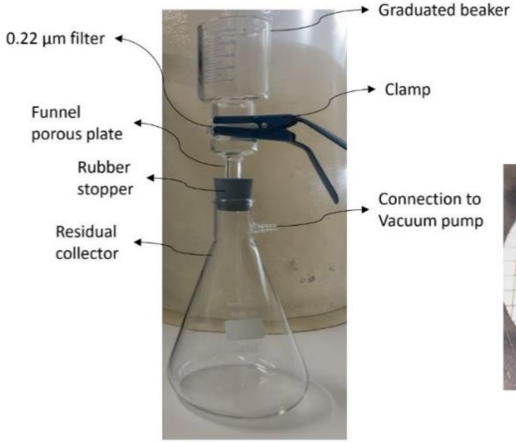
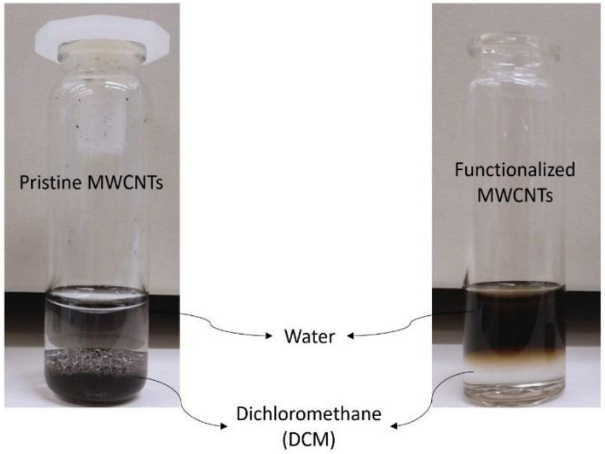
FUNCTIONALIZATION OF CARBON NANOTUBES

Filtration and Drying

- After dropping the temperature to room temperature
- Mixture is diluted by using 9:1 volume of distilled water
- Ice bath to drop down the mixture to room temperature
- Start the vacuum filtration by the shown setup
- Wash the MWCNTs at least 5 times and check for the pH to get down to 7
- MWCNTs obtained are kept in oven for drying overnight at 60 °C





MECHANICAL CHARACTERIZATION

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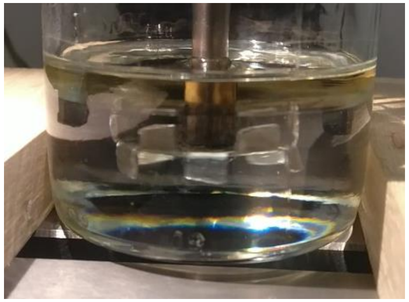
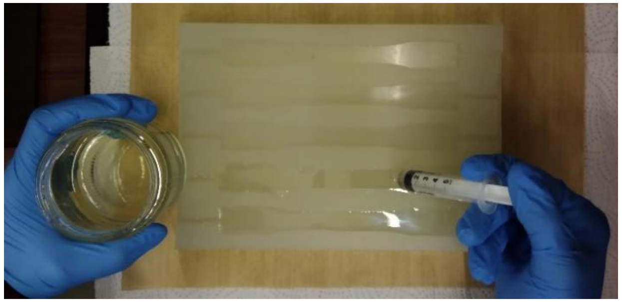
Specimens preparation

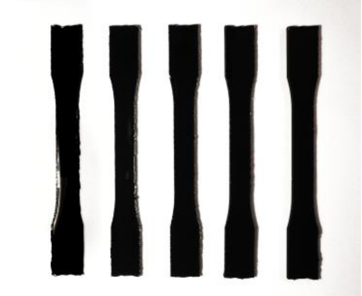
- study of the influence of the shear mixing speed and time on the epoxy resin system quality: optimum at 200 rpm for about 15 minutes
- curing cycle at 120 °C for 40 min (TDS and confirmed by DSC)

Manufacturing of specimens for tensile and compression tests from:

- pure GP epoxy resin system
- GP epoxy filled with 0.5 wt% not functionalized MCNTs
- GP epoxy doped with 0.5 wt% of functionalized MWCNTs

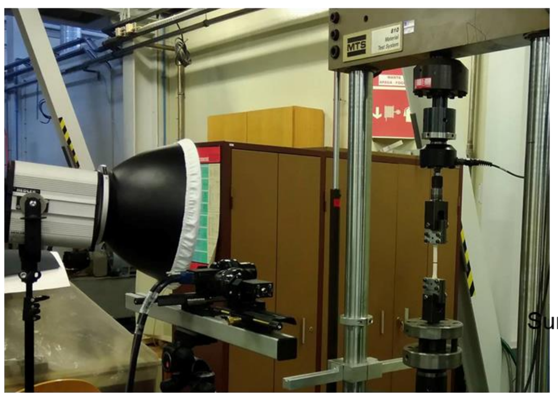




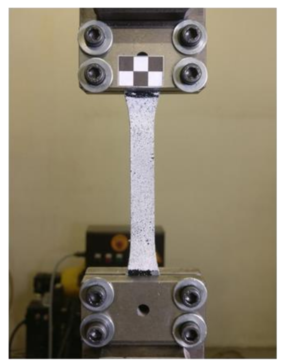


MECHANICAL CHARACTERIZATION

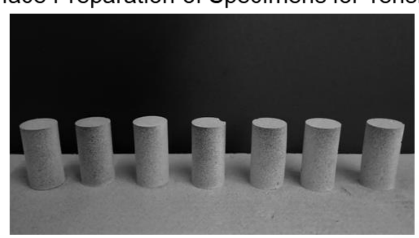
- Investigation of experimental **tensile** and **compression** behavior
- Strain-field detection by means of non-contact **Digital Image Correlation (DIC)** system is used during the experiments



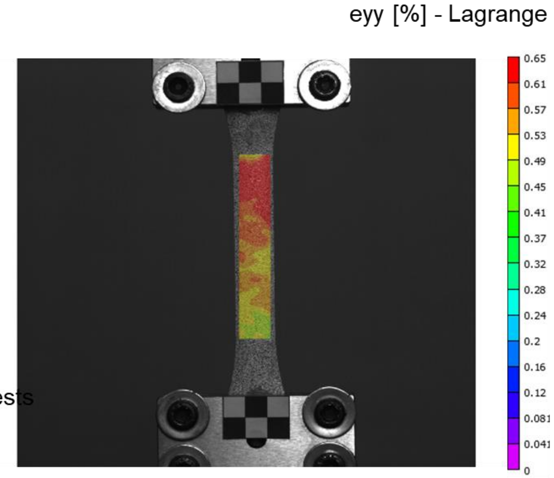
Digital Image Correlation System Set-up



Surface Preparation of Specimens for Tensile Tests



Surface Preparation of Specimens for Compression Tests

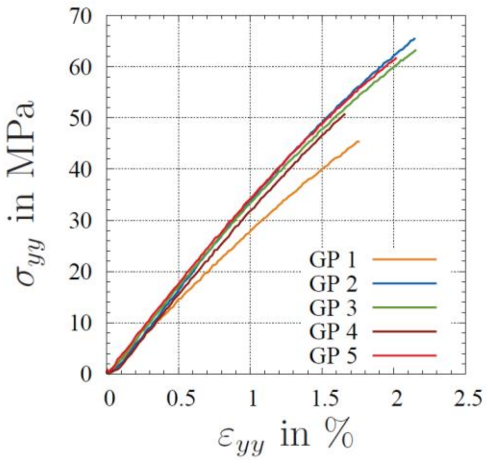


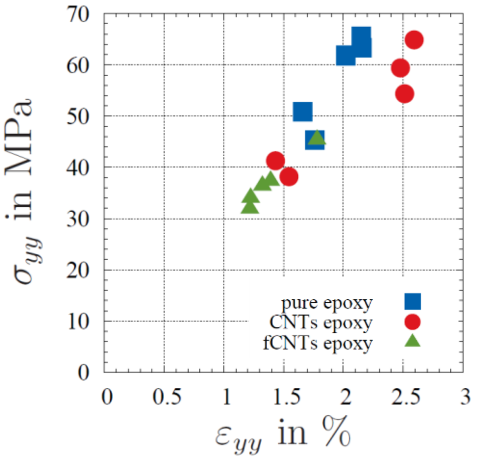
Strain Field Distribution of the Specimen before the Failure

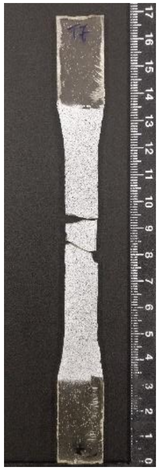
MECHANICAL CHARACTERIZATION

Experimental Tensile Tests according to ASTM D638-14

Monotonic uniaxial displacement controlled tests at room temperature and displacement-rate of 5 mm/min





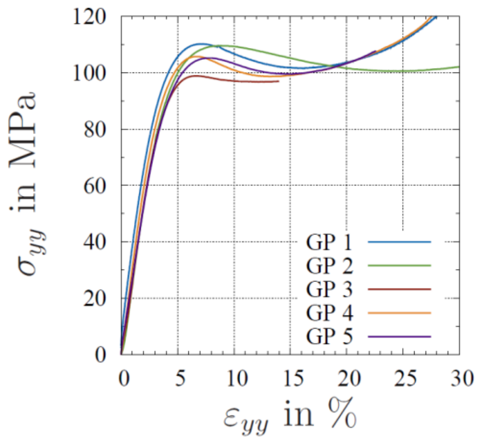


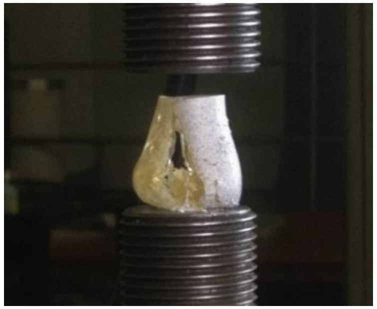
Tensile Sample after Failure

MECHANICAL CHARACTERIZATION

Experimental Compression Tests according to ASTM D695-15

Monotonic uniaxial displacement controlled tests at room temperature and displacement-rate of 1 mm/min





CONCLUSIONS

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- Recent literature formulations of bio-based resins show interesting mechanical properties for application in airplane interior and secondary structures
- An investigation on functionalization of MWCNTs and preparation of nano-filled bio-based resin was presented
- Tensile and compression behavior of samples from neat resin and resin filled with pristine and functionalized MWCNTs was carried out
- DIC system was used for obtaining the strain-field characterization
- Further work is needed for optimizing the functionalization process of MWCNTs
- Final aim is to obtain bio-based filled resin samples with improved mechanical and electrical properties

THANKS FOR YOUR ATTENTION

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