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Microwave technics to characterize impurity content in biofuels for on-line monitoring applications

Floriane SPARMA and Pierre SABOUROUX

Aix-Marseille-University

***Institut Fresnel**, Marseille, France*

2nd BIOFMET Stakeholders' Workshop

28 and 29 of March 2023, PTB, Braunschweig (Germany)

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Microwave technics to characterize impurity content in biofuels for on-line monitoring applications

1. **REMINDER OF ELECTROMAGNETIC CHARACTERIZATION METHOD**
2. **LIQUID BIOFUELS : IMPURITIES IN BIODIESEL**
3. **SOLID BIOFUELS : MOISTURE IN WOOD PELLETS**

Floriane SPARMA and Pierre SABOUROUX

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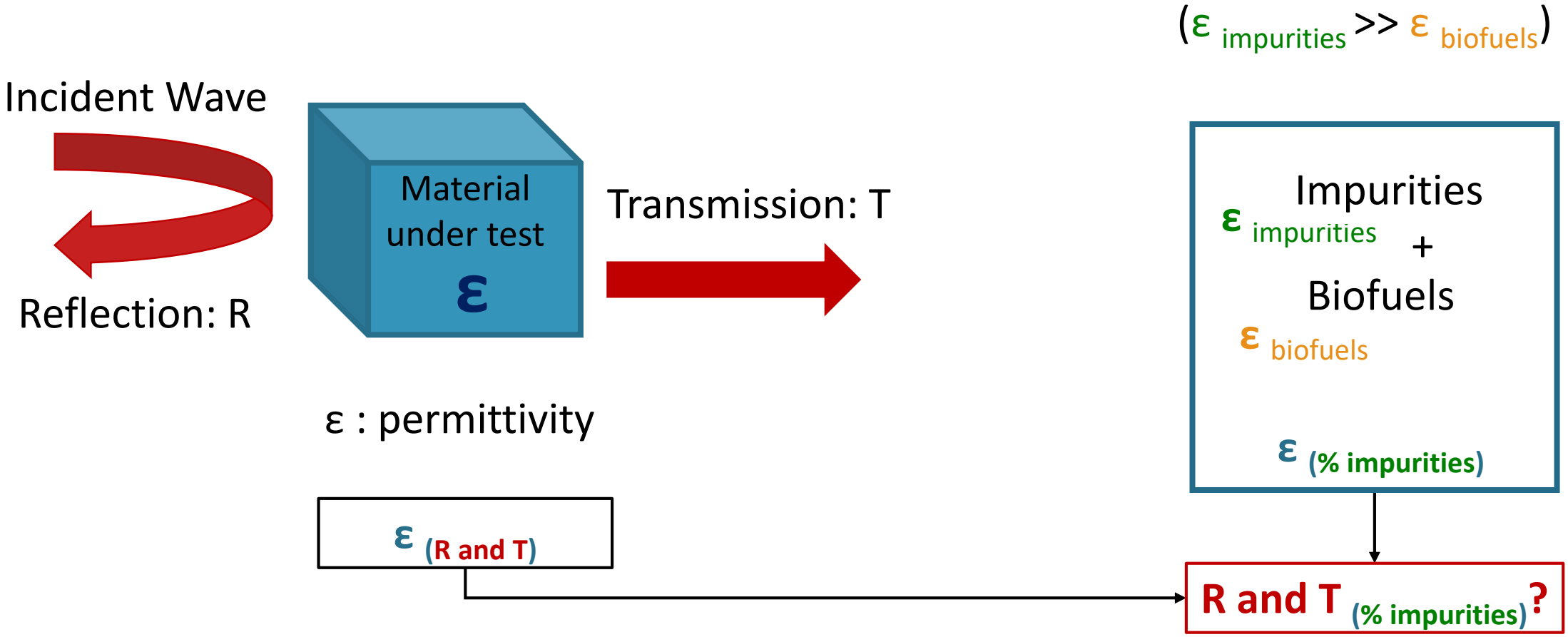
Dielectric characterization of materials : biofuels

($\varepsilon_{impurities} >> \varepsilon_{biofuels}$)



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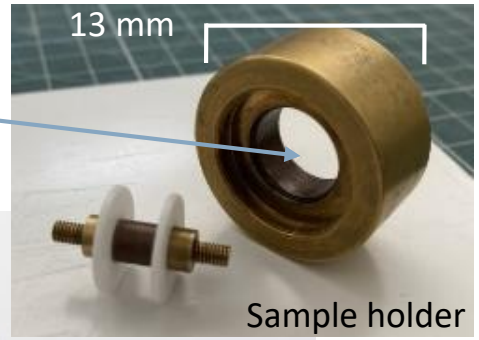
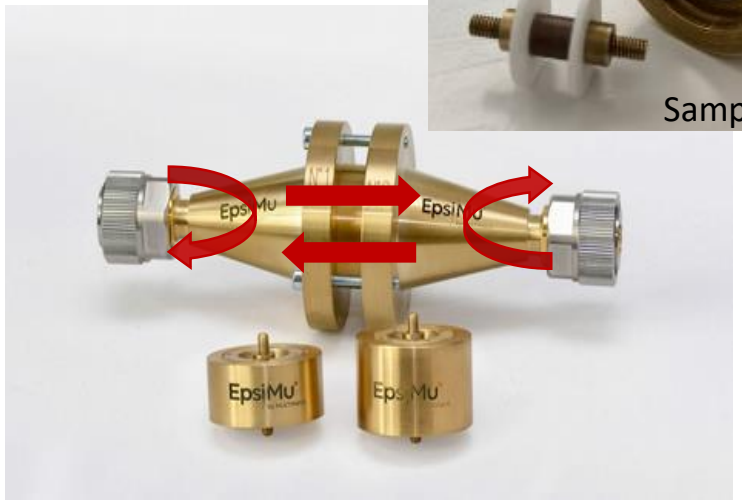
2.1 DIELECTRIC CARATERIZATION OF LIQUID BIOFUELS (BIODIESEL) WITH IMPURITIES

Biofuels with impurities





EpsiMu Cell

Coaxial transmission line

Inorganics impurities

Inorganic impurity	FQD	EN 228	EN 15376
water	NS	NS	≤ 0.300 % (m/m)
sulfur	≤ 50 mg/kg ≤ 10 mg/kg	≤ 10 mg/kg	≤ 10 mg/kg
phosphorous	NS	NS	≤ 0.15 mg/L
lead	≤ 0.005 g/L	≤ 5.0 mg/L	NS
manganese	≤ 2 mg/L	≤ 2 mg/L	NS
copper	NS	NS	≤ 0.100 mg/kg
chlorine	NS	NS	≤ 0.15 mg/kg
sulfate	NS	NS	≤ 3.0 mg/kg
additives	NS	NS	NS
Group I (Na&K)	NS	NS	NS
Group II (Ca&Mg)	NS	NS	NS
arsenic	NS	NS	NS
cadmium	NS	NS	NS
chromium	NS	NS	NS
mercury	NS	NS	NS
nickel	NS	NS	NS

Organics impurities

Organic impurity	FQD	EN 228
benzene	≤ 1.0 % (V/V)	≤ 1.0 % (V/V)
polycyclic aromatic hydrocarbons	≤ 11.0 % (m/m)	NA
methanol	≤ 3.0 % (V/V)	≤ 3.0 % (V/V)
ethanol	≤ 5.0 % (V/V)	≤ 10.0 % (V/V)
iso-propyl alcohol	≤ 12.0 % (V/V)	≤ 12.0 % (V/V)
tert-butyl alcohol	≤ 10.0 % (V/V)	≤ 15.0 % (V/V)
iso-butyl alcohol	≤ 7.0 % (V/V)	≤ 15.0 % (V/V)
ethers (>5 C/molec)	≤ 10.0 % (V/V)	≤ 22.0 % (V/V)
polysaturated (≥4 double bonds) methyl esters		
other oxygenates	≤ 15.0 % (V/V)	≤ 15.0 % (V/V)
FAME	NA	NA
linoleic acid methyl ester		
monoglycerides		
diglycerides		
triglycerides		
free glycerol		
total glycerol		

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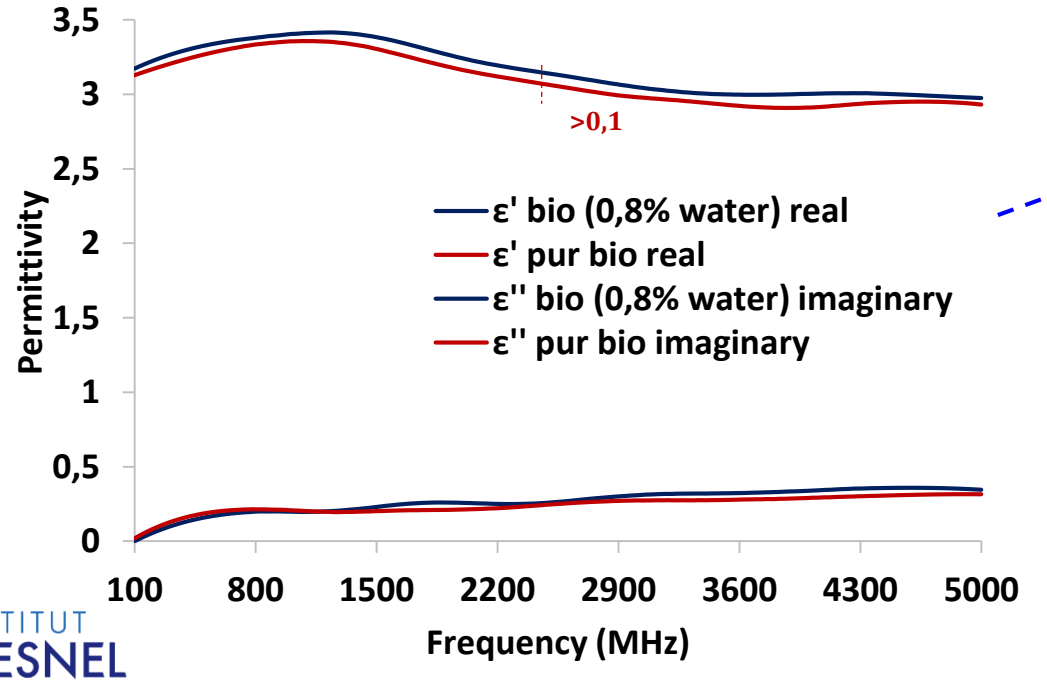
2.1 DIELECTRIC CARATERIZATION OF LIQUID BIOFUELS (BIODIESEL) WITH IMPURITIES

Inorganic impurity : water

Inorganic impurity	FQD	EN 228	EN 15376
water	NS	NS	≤ 0.300 % (m/m)
sulfur	≤ 50 mg/kg ≤ 10 mg/kg	≤ 10 mg/kg	≤ 10 mg/kg
phosphorous	NS	NS	≤ 0.15 mg/L

Permittivity of biodiesel (bio) with water



Water bubbles in biodiesel

ε' water = 78

ε' biodiesel = 3

Inhomogeneous liquids materials

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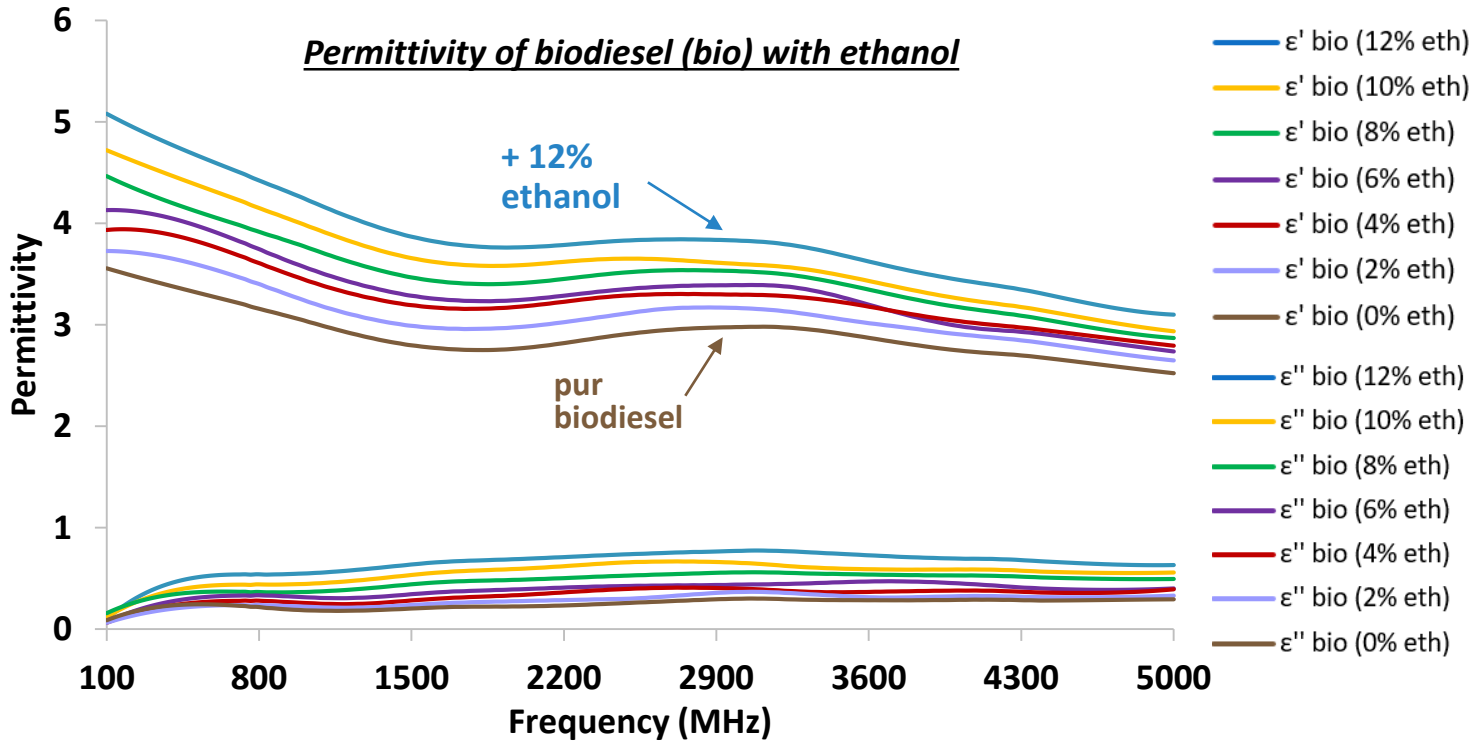
2.1 DIELECTRIC CARATERIZATION OF LIQUID BIOFUELS (BIODIESEL) WITH IMPURITIES

Organic impurity as « alcohol »: ethanol

methanol	≤ 3.0 % (V/V)	≤ 3.0 % (V/V)
ethanol	≤ 5.0 % (V/V)	≤ 10.0 % (V/V)
iso-propyl alcohol	≤ 12.0 % (V/V)	≤ 12.0 % (V/V)



homogeneous liquids mixtures :
biodiesel/ ethanol mixtures

POC (Proof Of Concept)

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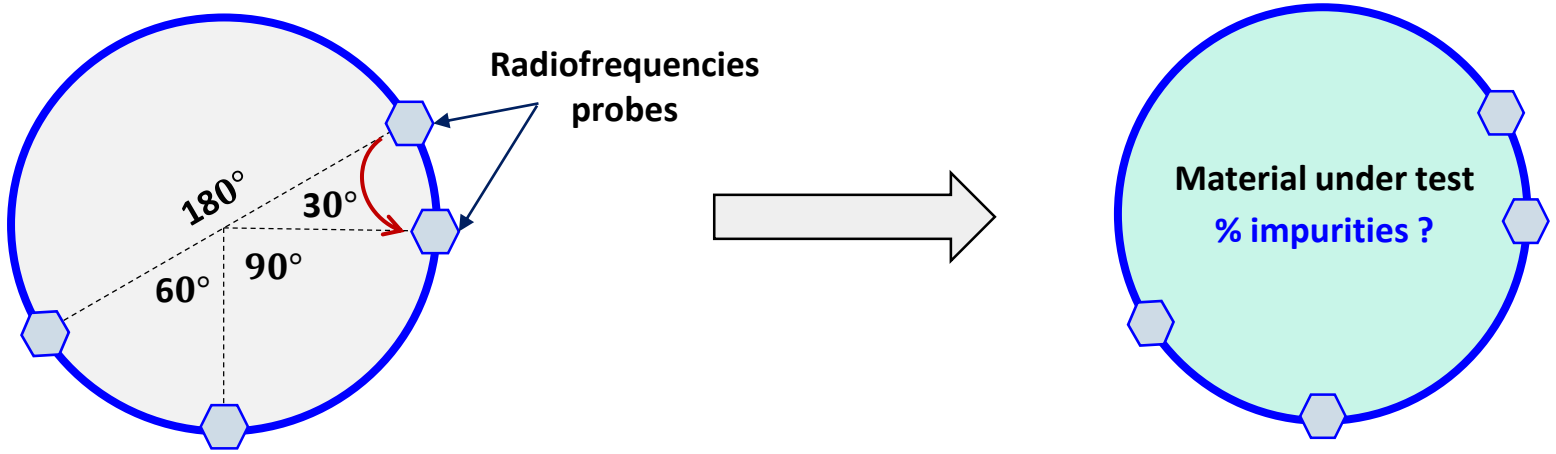
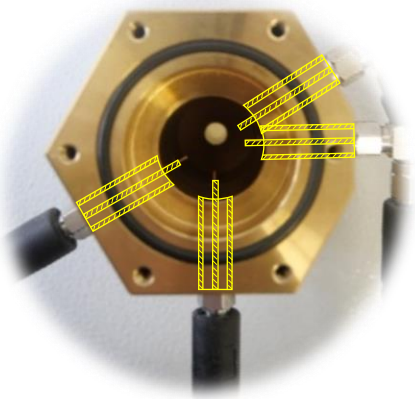
2.2 MATERIAL AND METHOD OF POC (PROOF OF CONCEPT)





POC

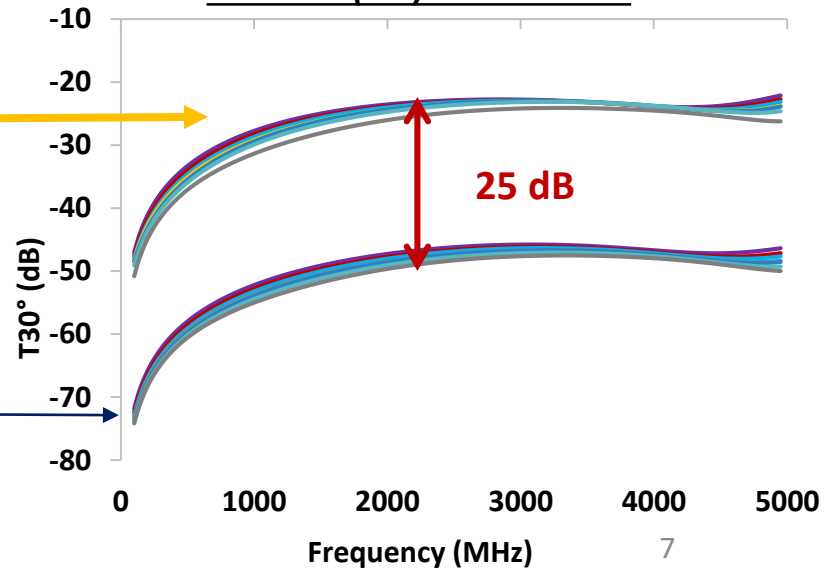
material under test

Probes with monopole antenna

material under test

Probes without monopole antenna

Simulations of 30° transmission of POC with biodiesel (bio) with ethanol



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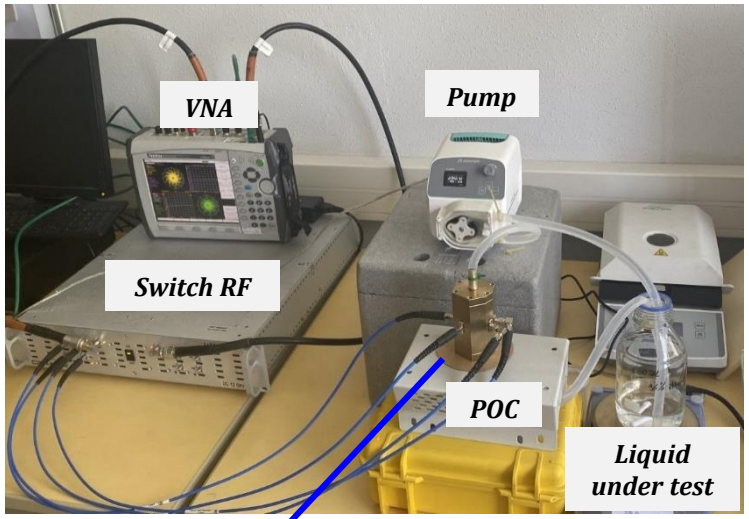
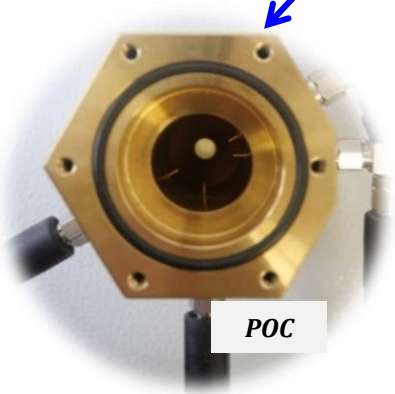
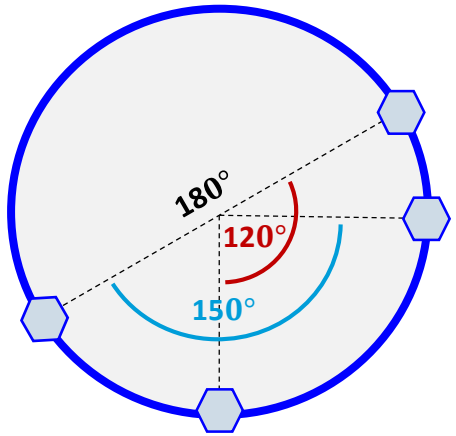
2.3 ONLINE MEASUREMENTS OF BIODIESEL/ETHANOL MIXTURES WITH THE POC

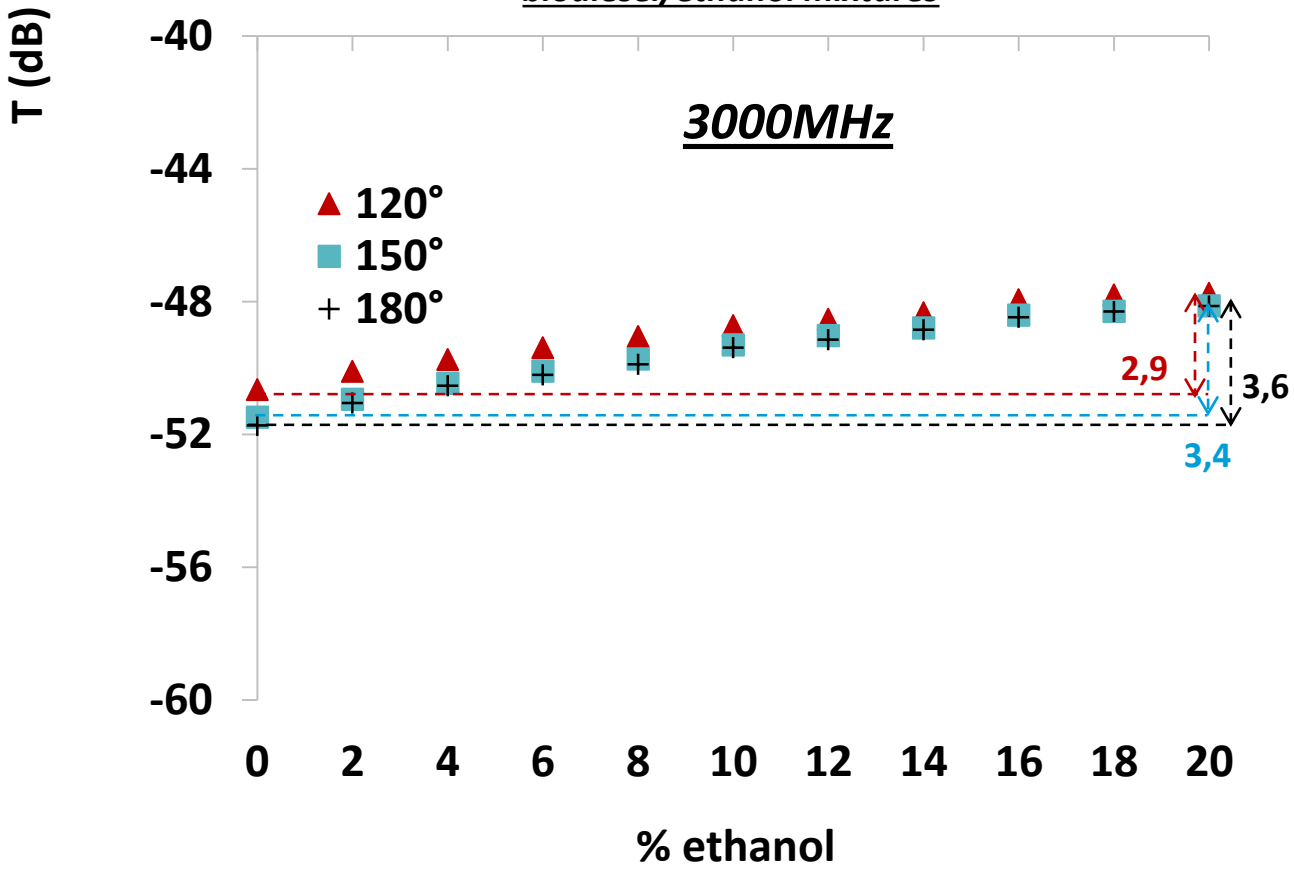
Bench of measurements of biodiesel/ethanol mixtures







Results of Transmission (T) at 120°, 150° and 180° biodiesel/ethanol mixtures



⇒ **A sensitivity of a 2% ethanol in biodiesel**

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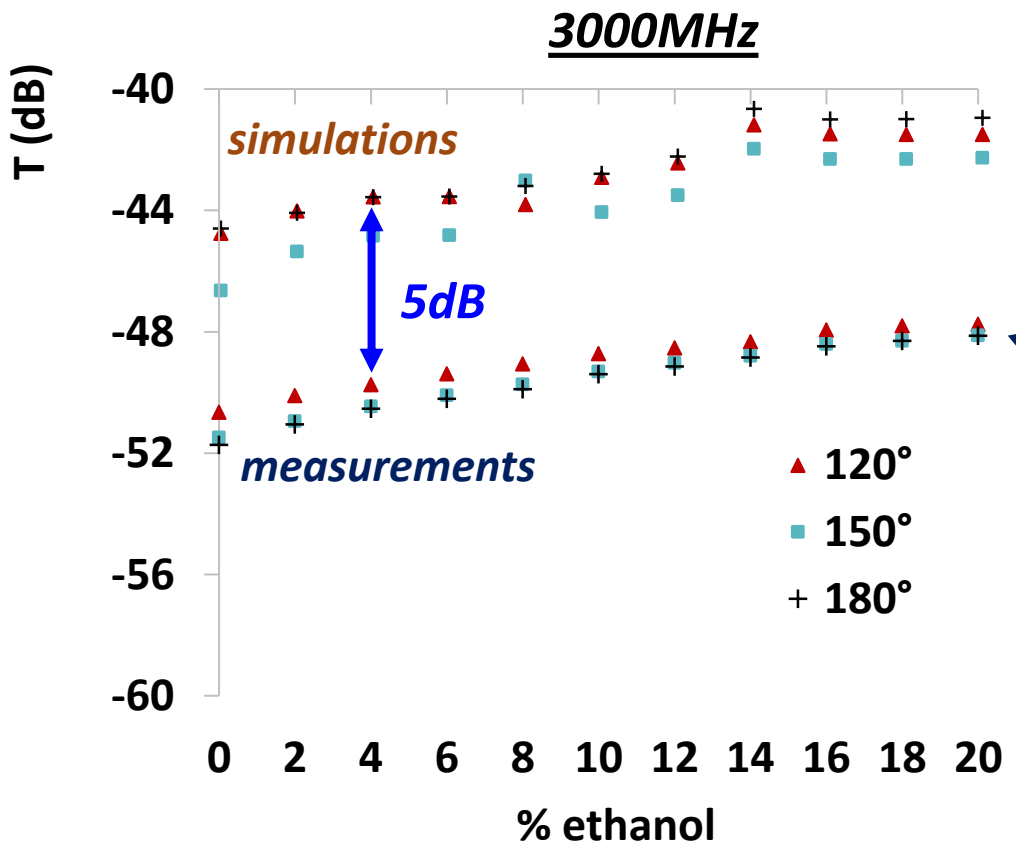
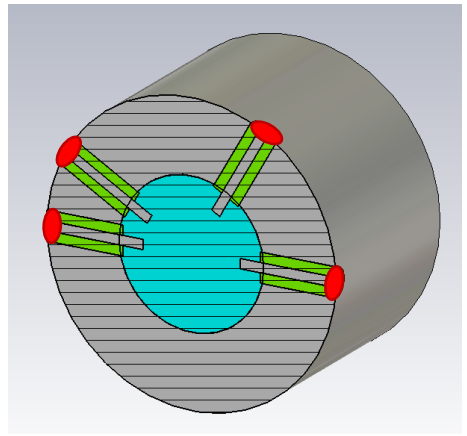
2.4 A CALIBRATION STUDIES WITH THE POC

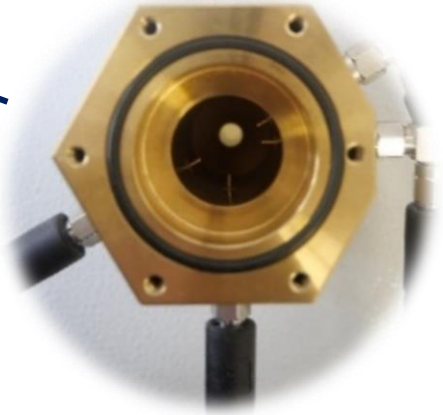
Results of measurements and simulations transmission at 3000MHz



Results area of simulations



Results area of measurements



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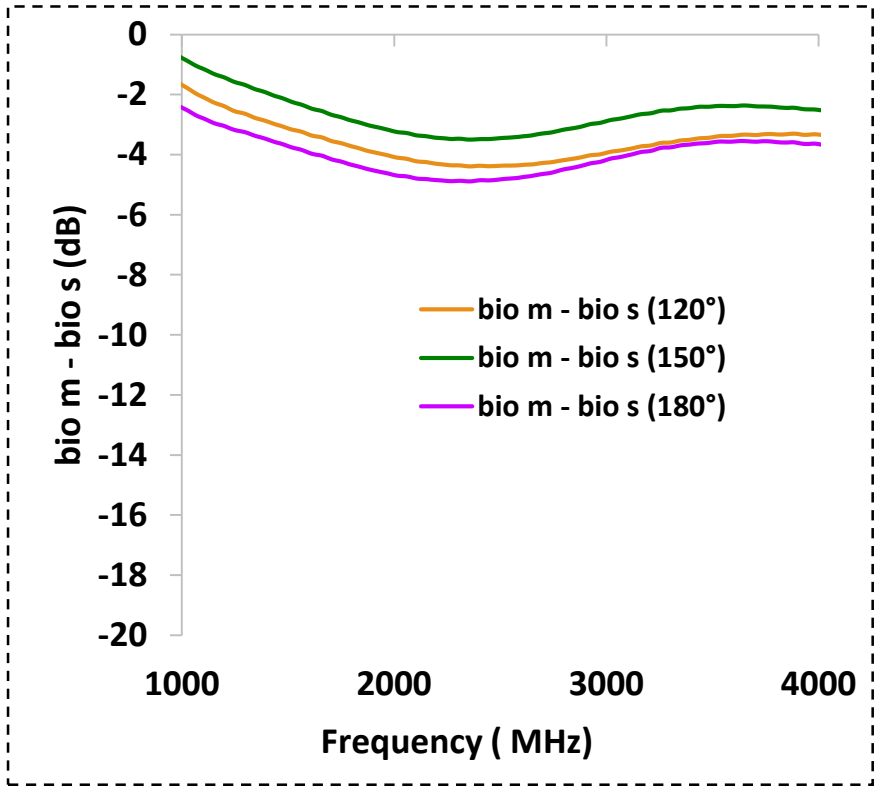
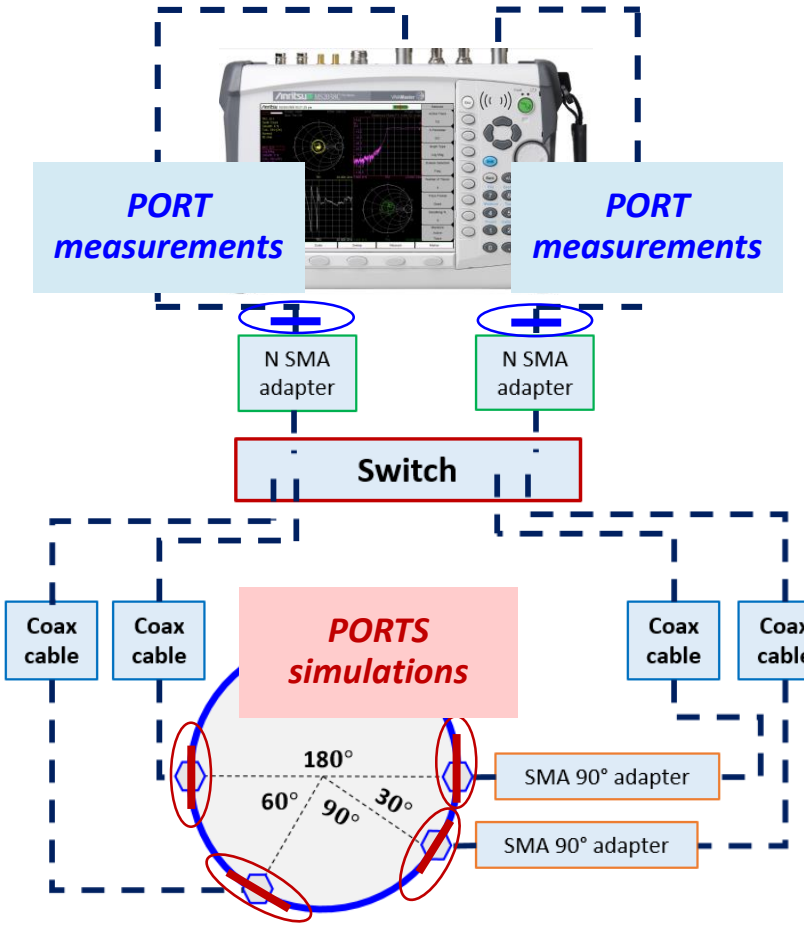
2.4 A CALIBRATION STUDIES WITH THE POC

Difference between simulation (s) and measurements (m

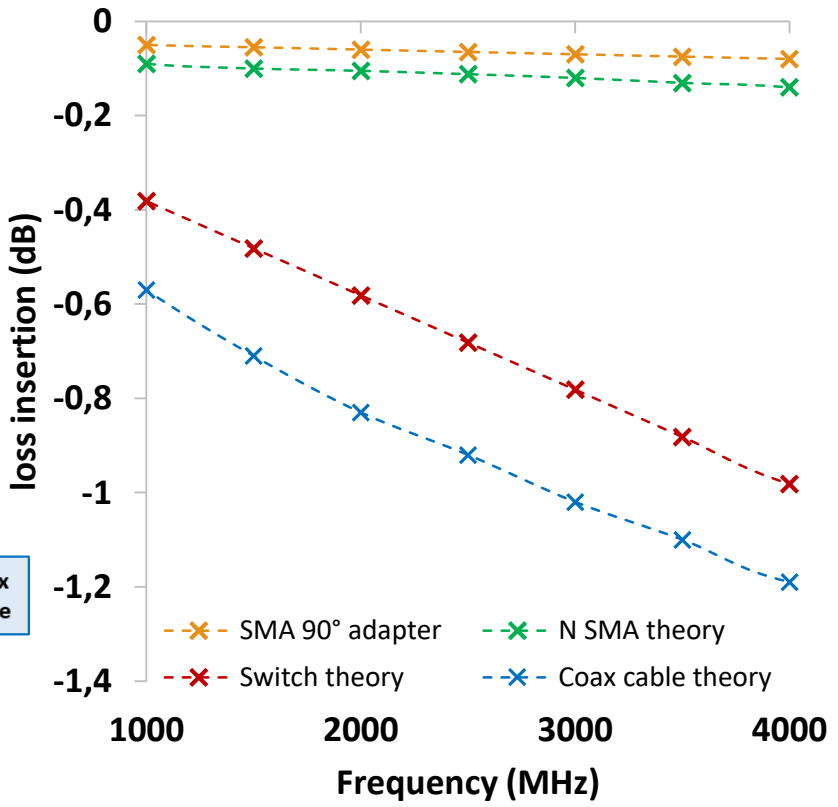


measurements (m) ≠ simulations (s)

Connection in the bench of measurements



Losses insertion (datasheets) connection



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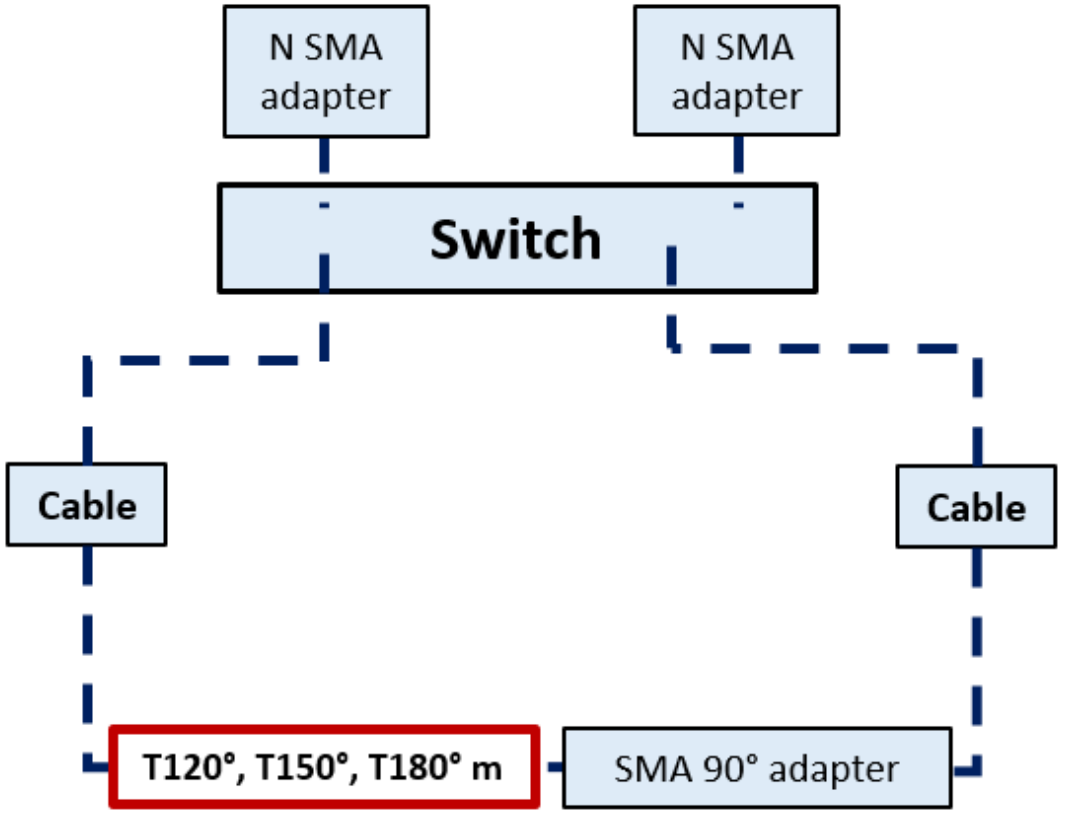
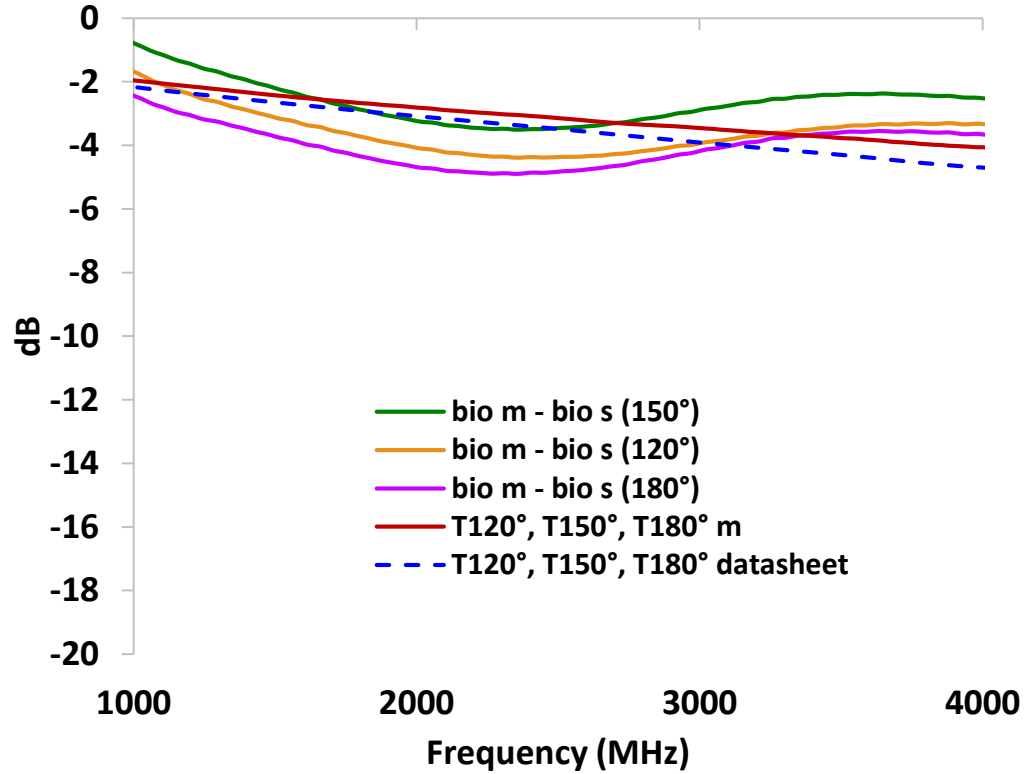
2.4 A CALIBRATION STUDIES WITH THE POC



Connection in the bench of measurements



Comparison of datasheets, connection measurements and measurements (m) – simulation (s)

⇨ **datasheets ~ connection measurements ~ comparison simulations/measurements**

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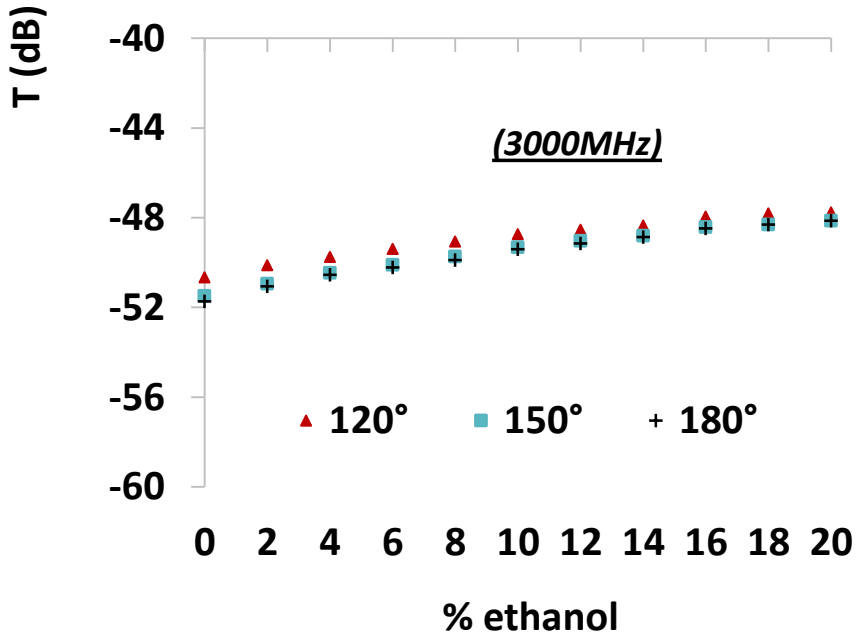
2.5 DISCUSSIONS AND CONCLUSION OF POC (PROOF OF CONCEPT)

Biofuels applications

⇨ Validation of the characterization electromagnetic method of liquids only on **biodiesel / ethanol mixtures**

⇨ **Optimization** of the POC and validation for biodiesel application : **sensitivity at 2% of ethanol**

⇨ **Verification simulations/ measurements**



Extra applications

⇨ **Agro-food application** : water content caracterization in honey

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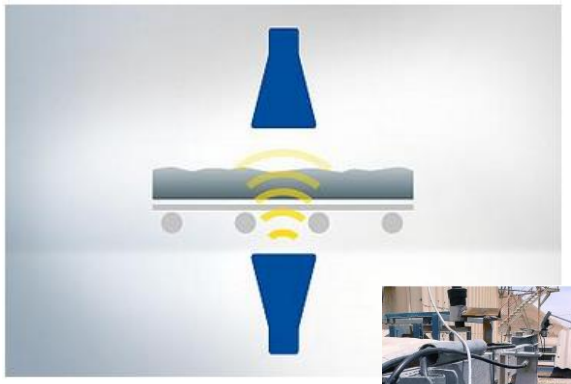
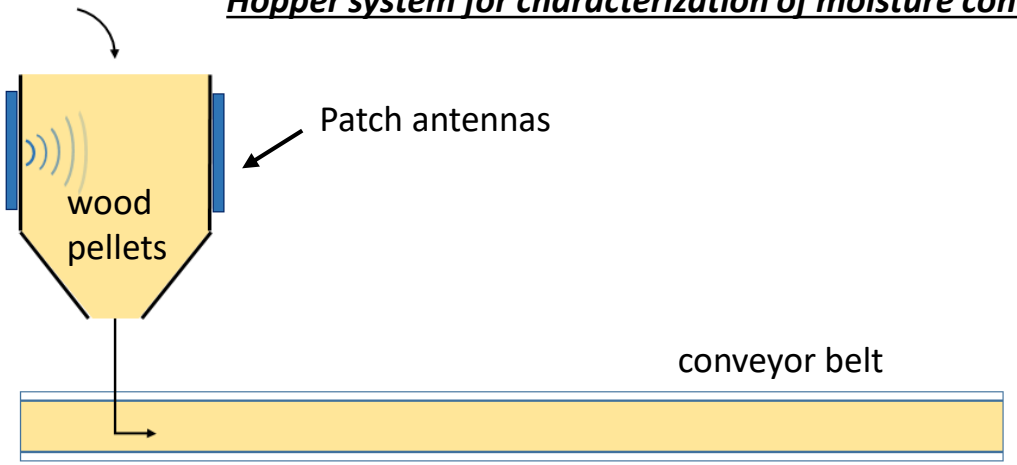
3.1 MICROWAVE TRANSMISSION METHOD FOR ONLINE MOISTURE MEASUREMENTS



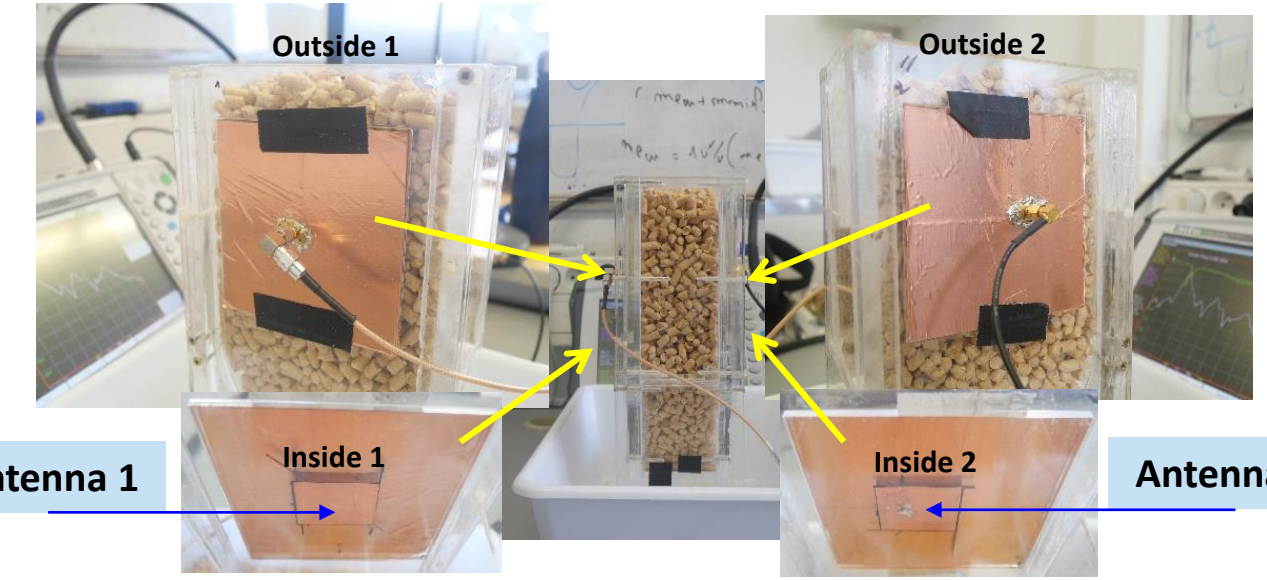
BERTHOLD
VERDO

Complementary to the setup developed by LNE-CETIAT

Hopper system for characterization of moisture content in wood pellet



- **Local measure**
- **Frequency wide band**
- **Cheap setup solution**



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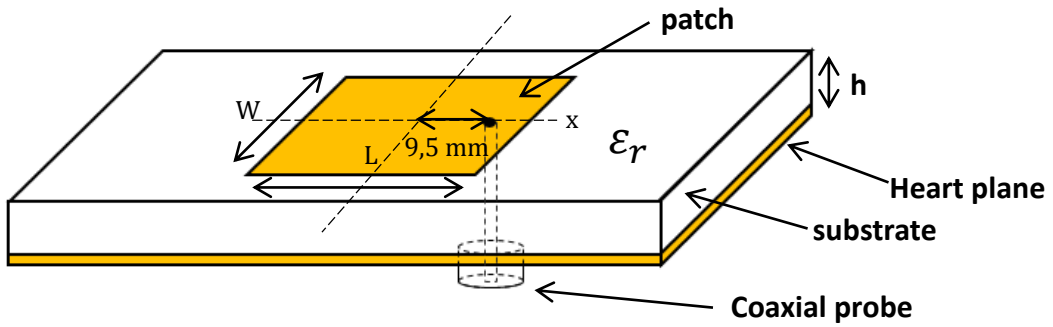
3.2 CONCEPTION OF A RECTANGULAR PATCH ANTENNA

Rectangular patch antenna

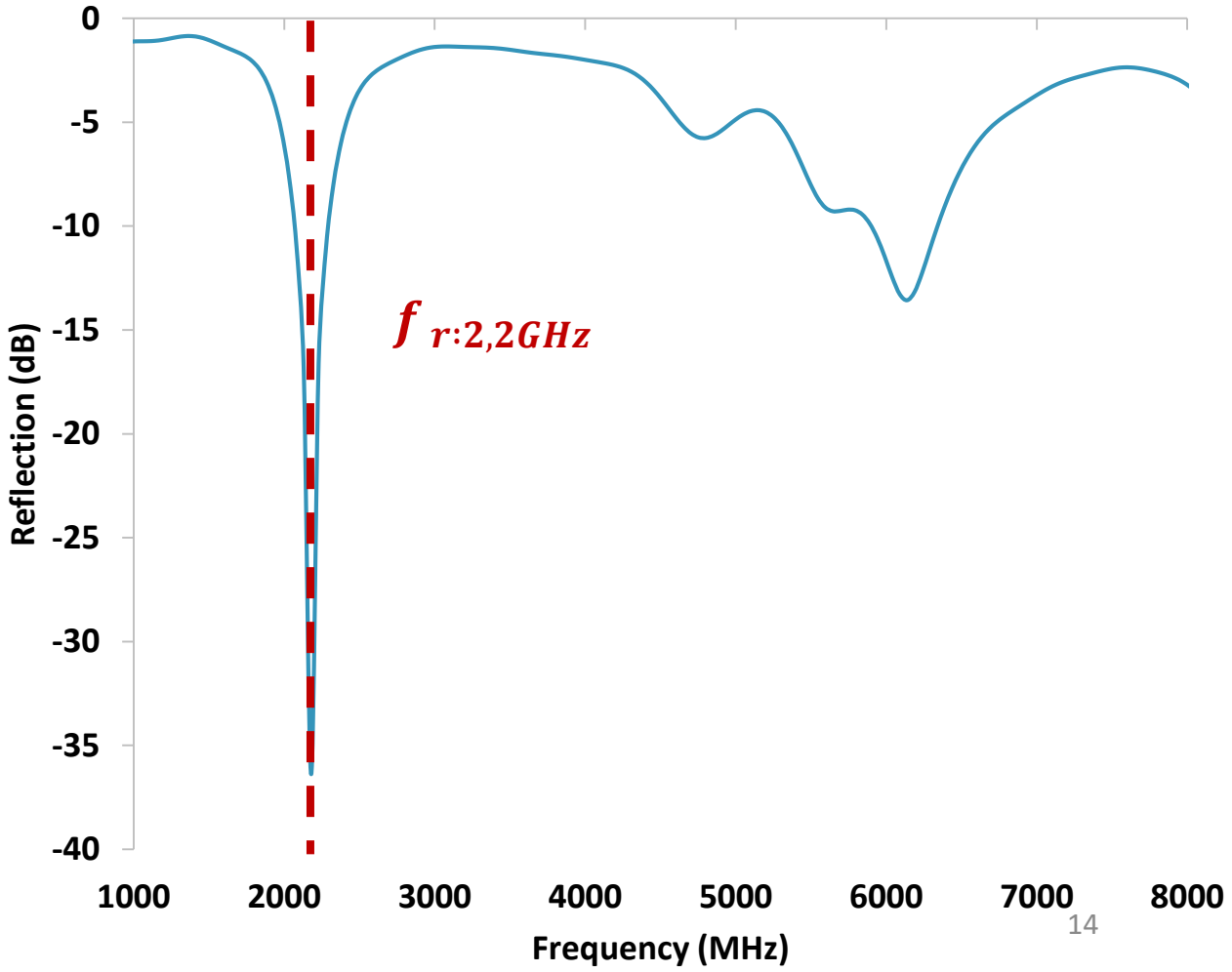


The patch caracteristics

For a resonance frequency of $f_{r:2,5GHz}$

L	35 mm
W	29,2 mm
ε_r	3,4
Substrate thickness	5 mm
Position of coaxial probe	9,5 mm

Simulation of the rectangular patch antenna



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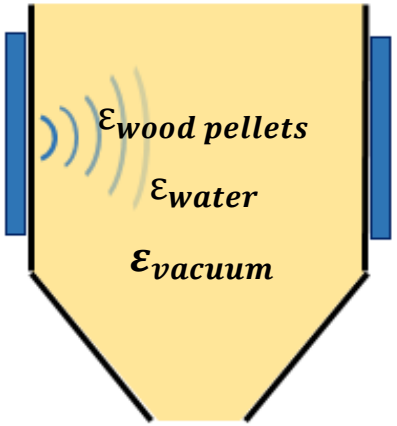
3.3 STUDIES OF THE HOPPER SYSTEM : FIRST MEASUREMENTS



Transmission measurements (*T*) of the system with :

- *Hopper with w.p.* → $T_{w.p.}$ *(dB)*
- *Empty hopper* → T_{vacuum} *(dB)*

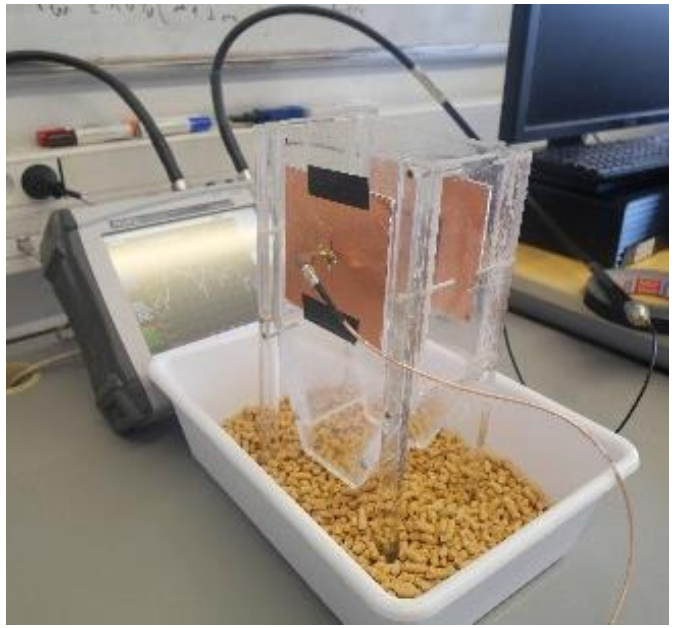
$MC_{w.p}$ **(%) :** Moisture Content of wood pellets *(w.p.)*

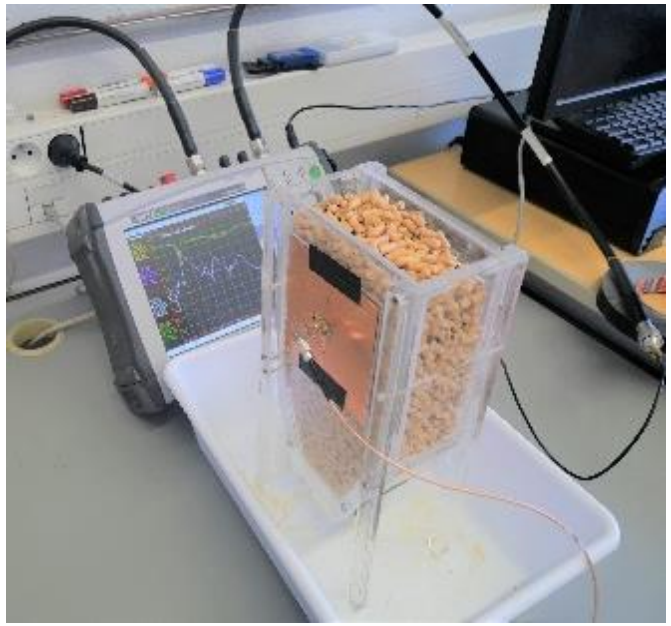
Mettler Toledo HB43

Setting : 1H, 130°C

$d_{w.p.}$ **:** density of wood pellets

$$T_r = T_{w.p.} - T_{vacuum}(dB)$$





System measurements with vacuum and wood pellets

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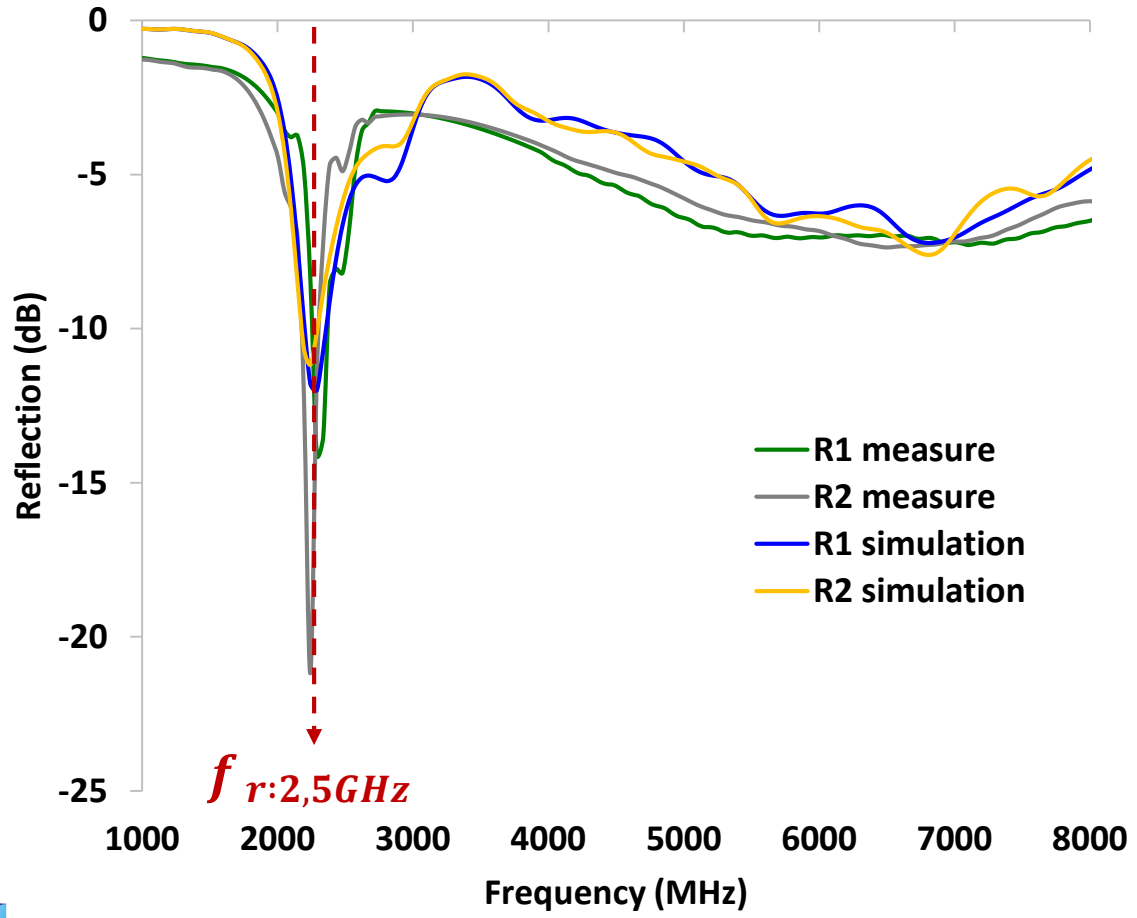
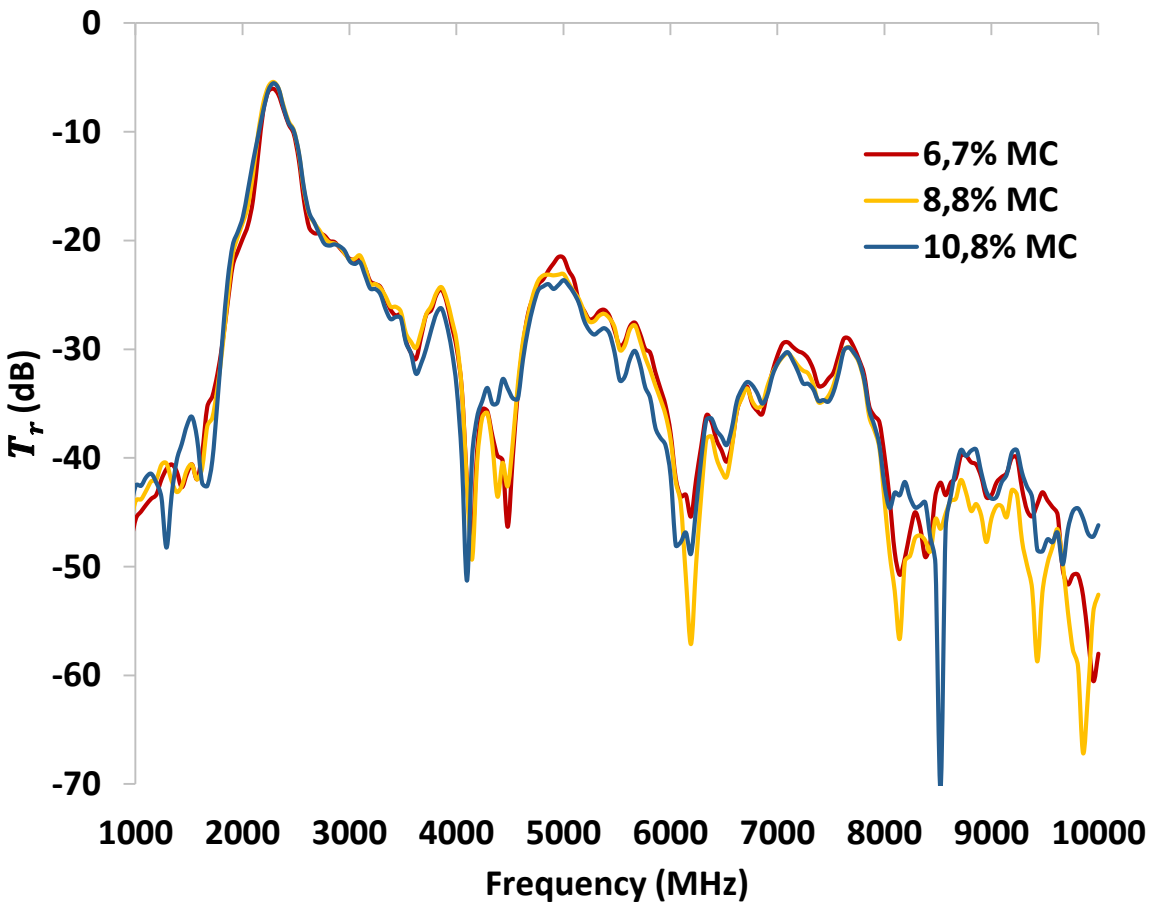
3.3 STUDIES OF THE HOPPER SYSTEM : FIRST MEASUREMENTS

Measurement of the vacuum system



Measurement of the wood pellets in the system



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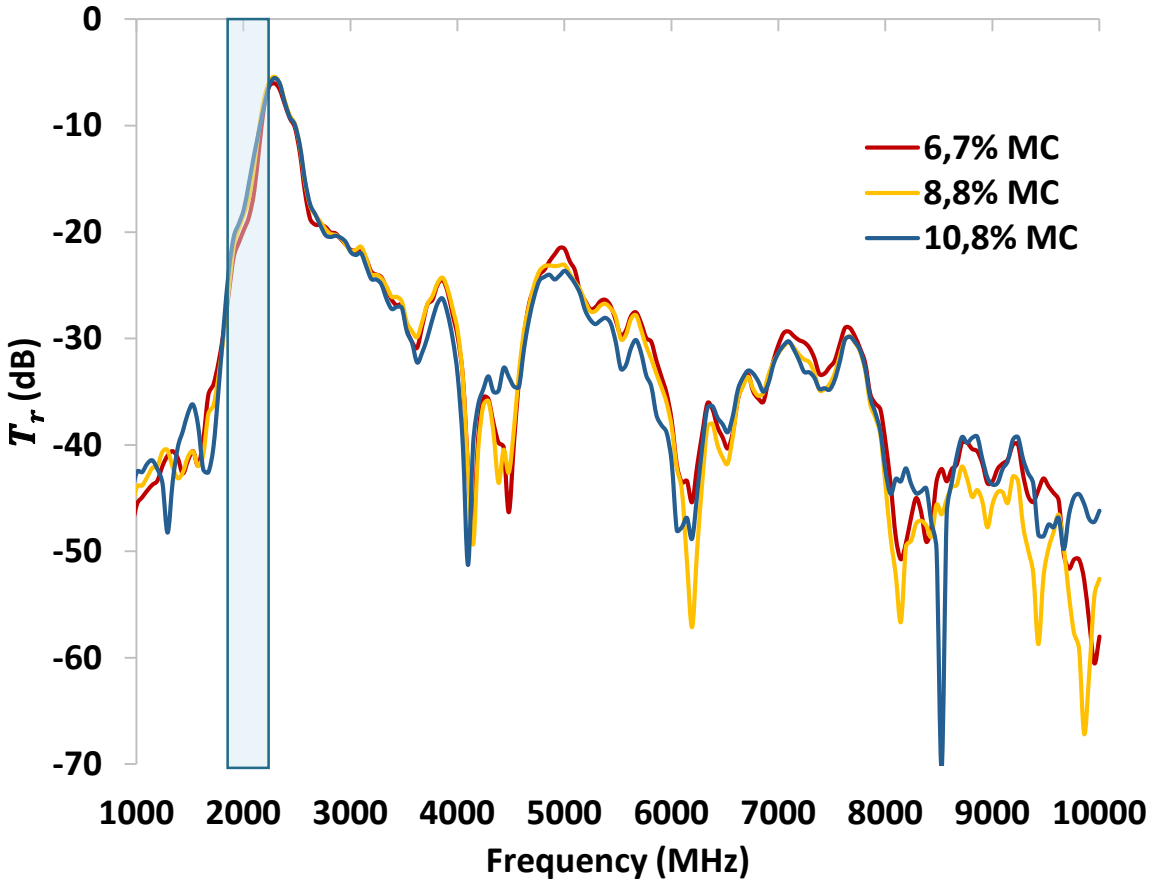
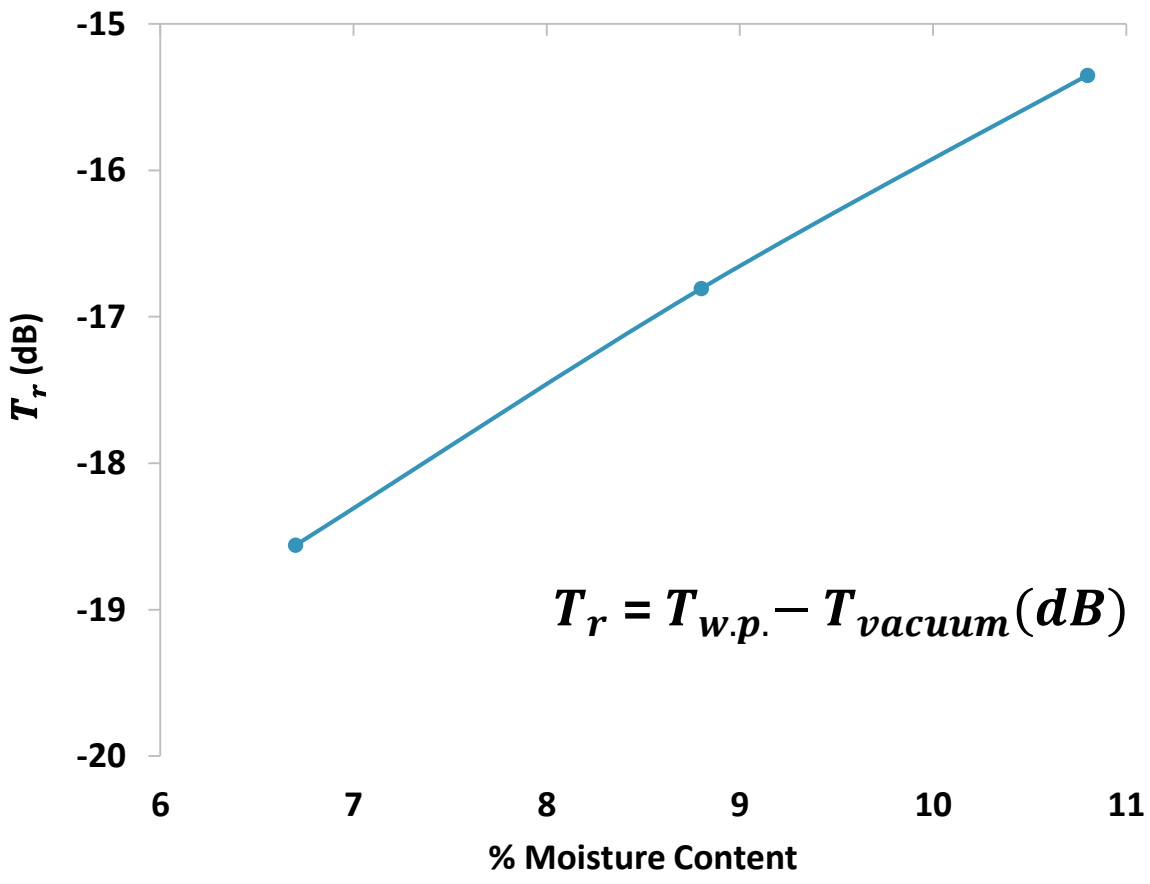
3.3 STUDIES OF THE HOPPER SYSTEM : FIRST MEASUREMENTS

Measurement of the wood pellets in the system



Results of T_r of wood pellets at 2050 MHz



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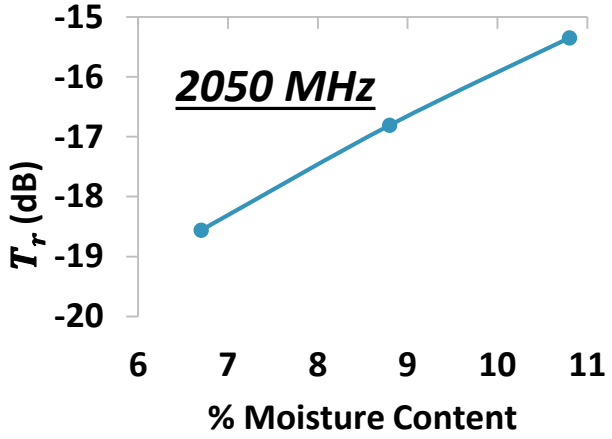
3.4 KEY OBSERVATIONS

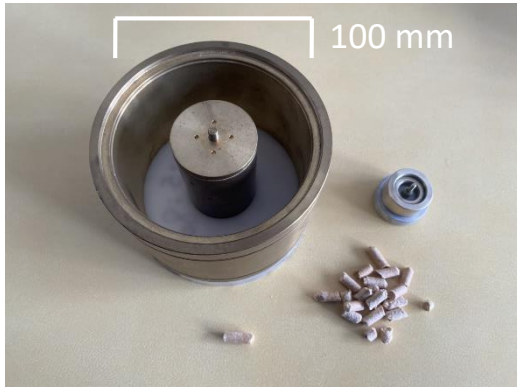


⇒ Measurements repeatability

→ Detection and analysis of peaks/ frequencies areas where the moisture content can be differentiating

⇒ Next step: Confirmation of experimental approach with simulation

→ Permittivity measurements of wood pellet …
Statistical approach: several samples
Higher sample holder dimensions



CONCLUSION

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LIQUID BIOFUEL

→ Dielectric characterization:
- Ethanol
- Biodiesel (Tubitak)
- Mixtures

→ Moisture Content and impurities detection: Proof of Concept

EXTRA APPLICATION: LIQUID AGRO MATERIAL

→ Dielectric characterization of Honey: MC detection

SOLID BIOFUELS

→ Preliminary study of a new setup to characterize MC in wood pellets

→ *To do: Dielectric characterization of wood pellets*

CONCLUSION

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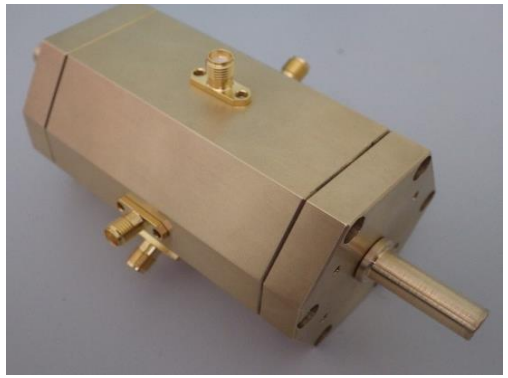

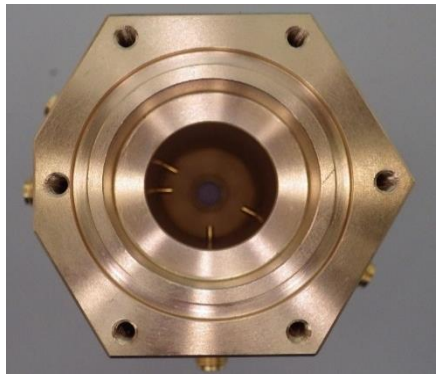

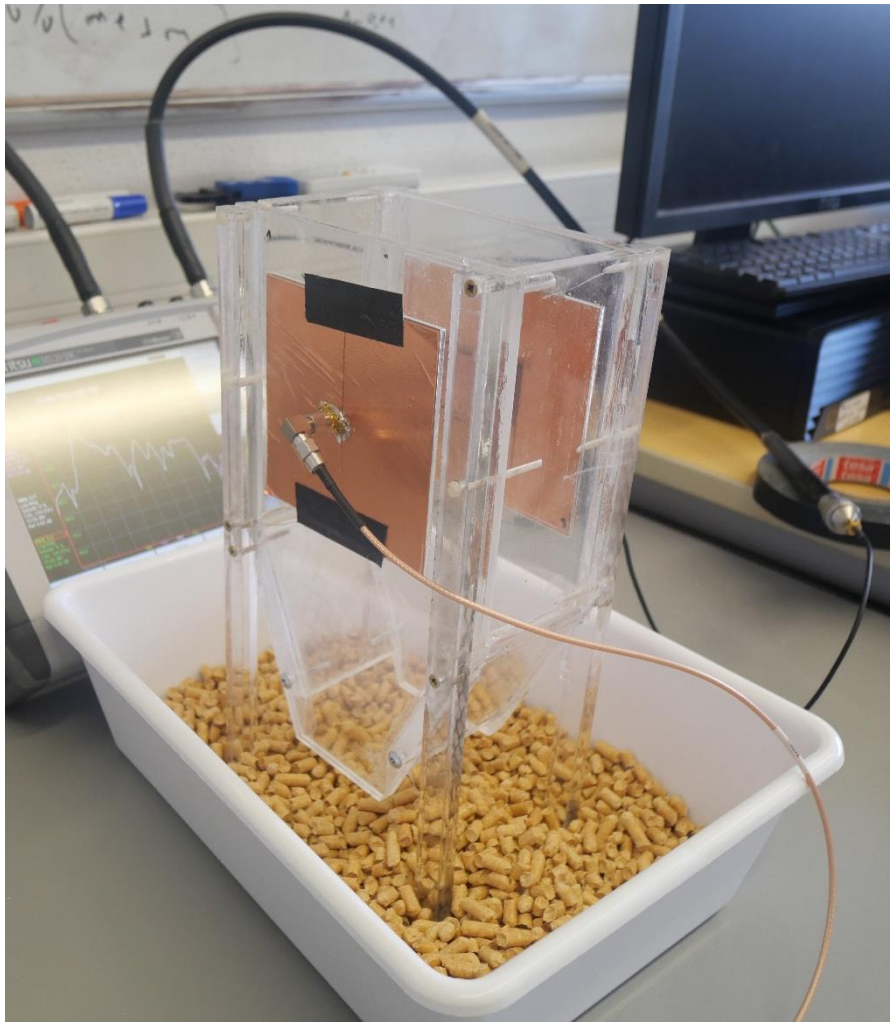

Thank you for your attention