NIRS measurement at field level to measure rubber and resin content of guayule plants

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Research activities on Guayule at CIRAD/UR Biowooeb

Materials and Methods

Résults

Conclusion & Perspectives
Interest of NIRS measurement applied to guayule resins and rubber quantification

- Help geneticists for the search of most performing lines of guayule plants,

- Follow up field production and best time to harvest,

- Fast and accurate determination of rubber and resins production per hectare
CIRAD ACTIVITIES ON GUAYULE IN FRANCE


- EUNARS-G commitment (2014) with ETRMA/WUR/CTTM/VERSALIS/MAPA with a target to replace 10% of NR imports from Asia (120,000 T.) by GR (200,000 ha) by 2030.

- Fast Track Innovation (FTI) project proposal (October 2016) : guayule planting (5-10 ha) in Andalucia/Huelva, Spain; building of a latex and resins extraction guayule pilot plant in 2017.

- FEADER (EU & LR regional funded) project with more guayule planting in France (>2ha) on four locations with farmers in South of France (LR and PO).

GUAYULE (Parthenium argentatum)

Guayule is adapted to Mediterranean climate (South of EU and North of Africa)
PI; Lipids (40; 3%)

PI (25-35%)

FLOW LATEX

WOOD

CAMBIIUM

LATICIFER TUBES

BARK

PI; Resin (9; 9%)
MATERIALS AND METHODS
TYPE OF EQUIPMENT USED

- **ASE 350 (Dionex/Thermofisher)**
  Détermination of a reference method for extraction of resins and PI
  PhD Sunisa SUCHAT (2012)

- **NIRS Laboratory VECTOR V22 (Brucker)**

- **NIRS field portable ASD (LabSpec4)**
ADVANTAGES OF NIRS MEASUREMENT

**Parthenium argentatum (guayule) biomass composition**

- Water
- PI
- Cellulose
- Protein
- Resins: Sesquiterpene (Guayulin A, B, C, D)
  - Triterpenoids
  - Triglyceride
  - Terpenes

**Laboratory**

- Long time & many processes

**NIRS**

- Fast ~ 1 min
- Developing & set up

**Why NIRS?** Advantages

- Fast
- Nondestructive
- Low cost
- Environmental Friendly

*Source PhD Sunisa Suchat*
The NIRS method is based on the Phenomenon that functional groups such as C-H, N-H and O-H absorb near infrared light.
Résines :

2210 nm
Strain of the link -C=O of a carboxyle group

2308 nm
Strain and distortion of the link -C-H of -CH₃ from a lipid

Polyisoprène :

1716 nm
Strain of link C-H of -CH₃

Moisture :

1450 nm /1940 nm
Strain of link O-H of H₂O

Structure of PI, Resin :

Source PhD Sunisa Suchat
METHODOLOGY

- **Laboratory : (Vector V22 Brucker)**
  - Branches samples dried (moisture < 9%) and size particles < 500µm)
  - Scan reflectance mode: 400nm à 2500nm (interval 2nm)
  - Average of 32 spectra with correction of light dispersion
  - Calculation of second derivative for 5 points polynomial lissage
  - PLS regression used to establish calibration curves
  - SECV calculation with prediction on 25% of samples with a calibration developed with 75% remaining samples

- **Field and Laboratory : (ASD Labspec4)**
  - Use of a reflectance probe
  - 5 spectra per branch and 10 branches per plant, or 50 spectra per plant

**TOTAL:** 70 branches selected or 350 NIRS measurements at field level to establish a model LAB NIRS/FIELD NIRS.
PRINCIPLE OF NIRS MEASUREMENT

1. NIRS analysis
   Lab Vector V22 Brucker

2. Reference Lab data
   Extraction
   Quantified by Gravimetry

3. NIRS lab Portable ASD (LabSpec4)

4. ASD (Dionex)

5. Mathematical analysis

6. Calibration Equation, Validation
RESULTS
PRE-TREATMENTS OF SPECTRA

- To correct scaling differences coming from pathlength effects, scattering effects, source or detector variations, or other general instrumental sensitivity effects: SNV (standard normal variate) and Detrend

- To reduce scatter effects by removing a linear baseline shift: Second derivative (5 points polynomial)

- Different absorbance curves between Nirs apparatus: Vector and ASD.
- Difference in spectra between laboratory measurements and field measurements with ASD.
Calibration and validation: RESIN

Resin content between 4% to 12%

Vector V22
- 206 samples, $R^2 = 0.96$
- Error of cross validation: RMSECV = 0.81
⇒ Resin content = predicted value ± 0.81

ASD Laboratory
- 123 samples, $R^2 = 0.96$, RMSECV = 0.77

ASD In situ
- 123 samples, $R^2 = 0.96$, RMSECV = 0.85
**Calibration and validation: POLYISOPRENE**

Polyisoprene content between 3% to 10%

**Vector V22**
- 174 samples, $R^2 = 0.82$
- Error of cross validation: RMSECV = 1.03

**ASD Laboratory**
- 91 samples, $R^2 = 0.77$, RMSECV = 0.83

**ASD In situ**
- 91 samples, $R^2 = 0.89$, RMSECV = 1.24
**SUMMARY**

- Identical results for the resin content: same $R^2$ 0.96 with an error of about 0.80

- Efficiency of models for polyisoprene is less than those for resin: $R^2$ between 0.77 to 0.89 with an error between 0.8 to 1.2

- Polyisoprene models built with a low number of samples, and/or the reference values with ASE 350 are difficult to assess

<table>
<thead>
<tr>
<th></th>
<th>Number of samples</th>
<th>Model variables</th>
<th>Calibration</th>
<th>Cross validation</th>
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<tr>
<td><strong>Vector V22</strong></td>
<td></td>
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<tr>
<td>Resin</td>
<td>206</td>
<td>5</td>
<td>0.96</td>
<td>0.81</td>
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<td>Polyisoprene</td>
<td>174</td>
<td>4</td>
<td>0.82</td>
<td>1.03</td>
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</tbody>
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| **ASD Labo**   |                   |                 |             |                 |
| Resin          | 123               | 7               | 0.96        | 0.77            |
| Polyisoprene   | 91                | 3               | 0.77        | 0.83            |

| **ASD InSitu** |                   |                 |             |                 |
| Resin          | 123               | 7               | 0.96        | 0.85            |
| Polyisoprene   | 91                | 6               | 0.89        | 1.24            |
CONCLUSION & PERSPECTIVES
- Capability to determine by NIRS measurement, guayule resin content directly by field measurement (non-destructive and fast determination);

- Uncertainty of determination of ±0.8 for a resin content range of 4% to 12%;

- Model for polyisoprene less efficient with an uncertainty of 1.2% for a rubber content range of 3% to 10%;

- Need to increase the number of measurements to improve the database and to build more powerful models;

- Need to investigate the best area of NIRS measurement on a guayule plant, the effect of seasonality on NIRS measurement, the relationship between the depth of NIRS measurement and the presence of polyisoprene.