Agenda of the ICCTM-Stickiness TF 2018

- Introduction
  - HarCoStiC
  - RTStick: results and future
  - Comparison of results
- The effect of ETO fumigation on stickiness. René van der Sluijs
- Contest-Fibermap: Contest-Fibermap: the first year of field testing on stickiness detection. Gabrielle Salvinelli
- Embrapa: Developing a Portable Device Using NIR Image for Easy Detection of Stickiness in Cotton. Liv Severino
- KOTITI: Announcement
- Prodev: Announcement
- CIRAD: Announcement
- Conclusion

ITMF-ICCTM Stickiness task force

Results of the 2017 international round-test of stickiness measuring methods

Jean-Paul Gourlot, Serge Lassus, Axel Drieling, Karsten Froese
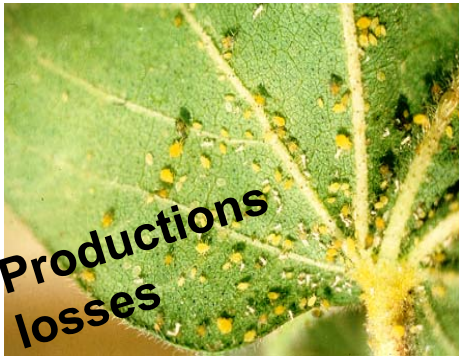
Bremen, March 2018

Stickiness in spinning mill due to entomological sugars

**These sugars or honeydew are mainly produced by *Aphis* and *Bemisia*, …
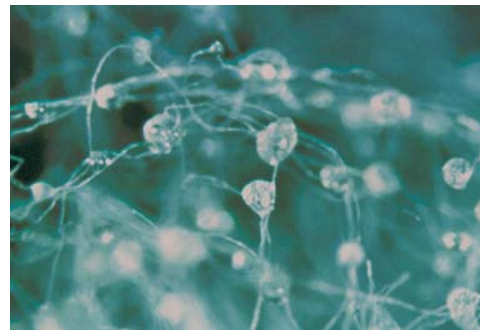but new insects are coming (mealybug, …, due to resistance, GMO…)**
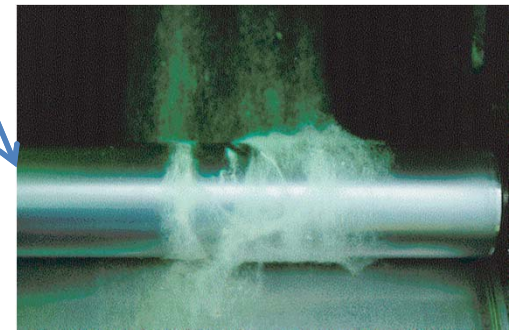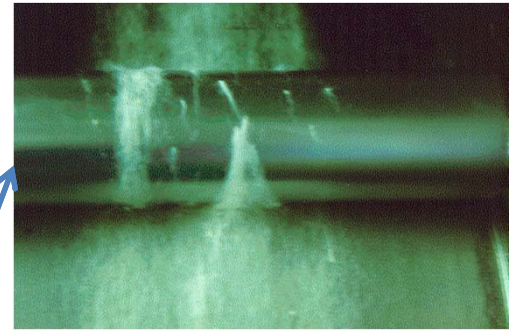
Aphis gossipii

Honeydew on open boll

Productions losses

Bemisia tabaci

Honeydew in fibers

Problems — **Productivity, quality**

**=> Need for reliable characterization (method, <u>reference material</u>,
predictive of problems in spinning…)**

Mandates

Reminder

Two of the mandates of the ICCTM are:

[…/…] **"to harmonize cotton testing results by means of:**

- a. proposition and support for the international standardization of test methods

- b. development of guidelines for testing

- **c. technical evaluations using world-wide round tests.**

and to discuss the problems related to testing of cotton fiber properties and their relations to cotton processing**."**
[…/…]

Reminder

- To check the ability of each measuring technique to reproduce itself within a same single laboratory
- To check the ability of each measuring technique to reproduce itself between several laboratories
- To give some indications about the ability of various measuring techniques to correlate to each others

One RT conducted in 2013-2014
=>report 2014 and 2016 (instruments vs micro-spinning)

➔need harmonization
➔need creation of reference materials

Reminder

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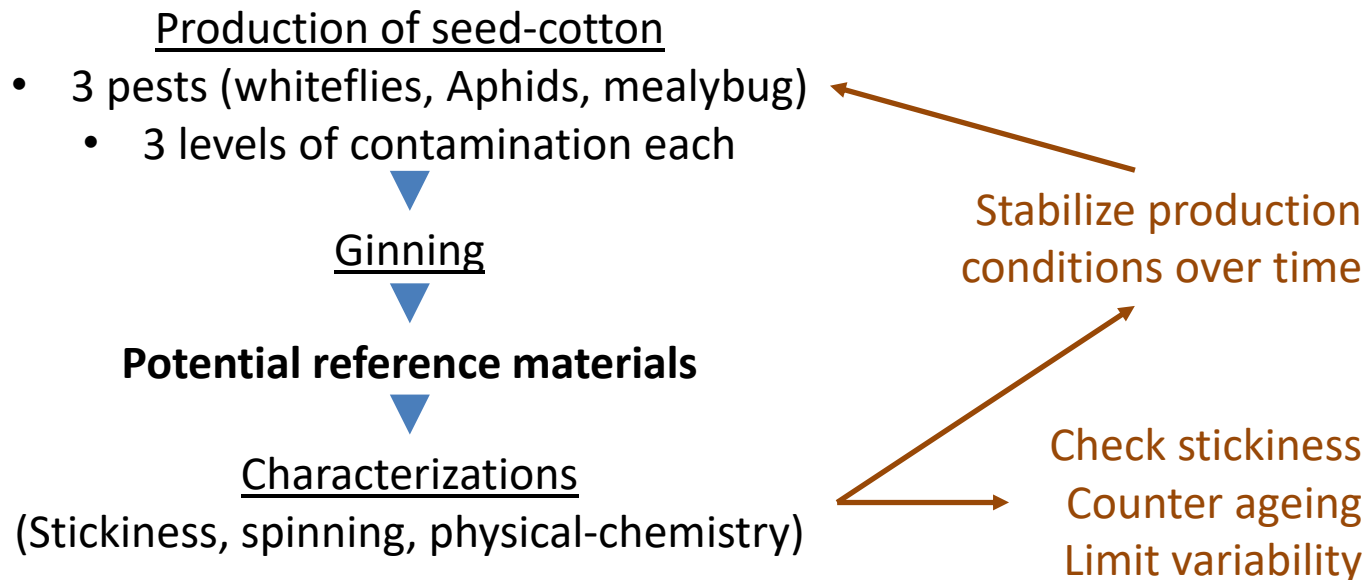
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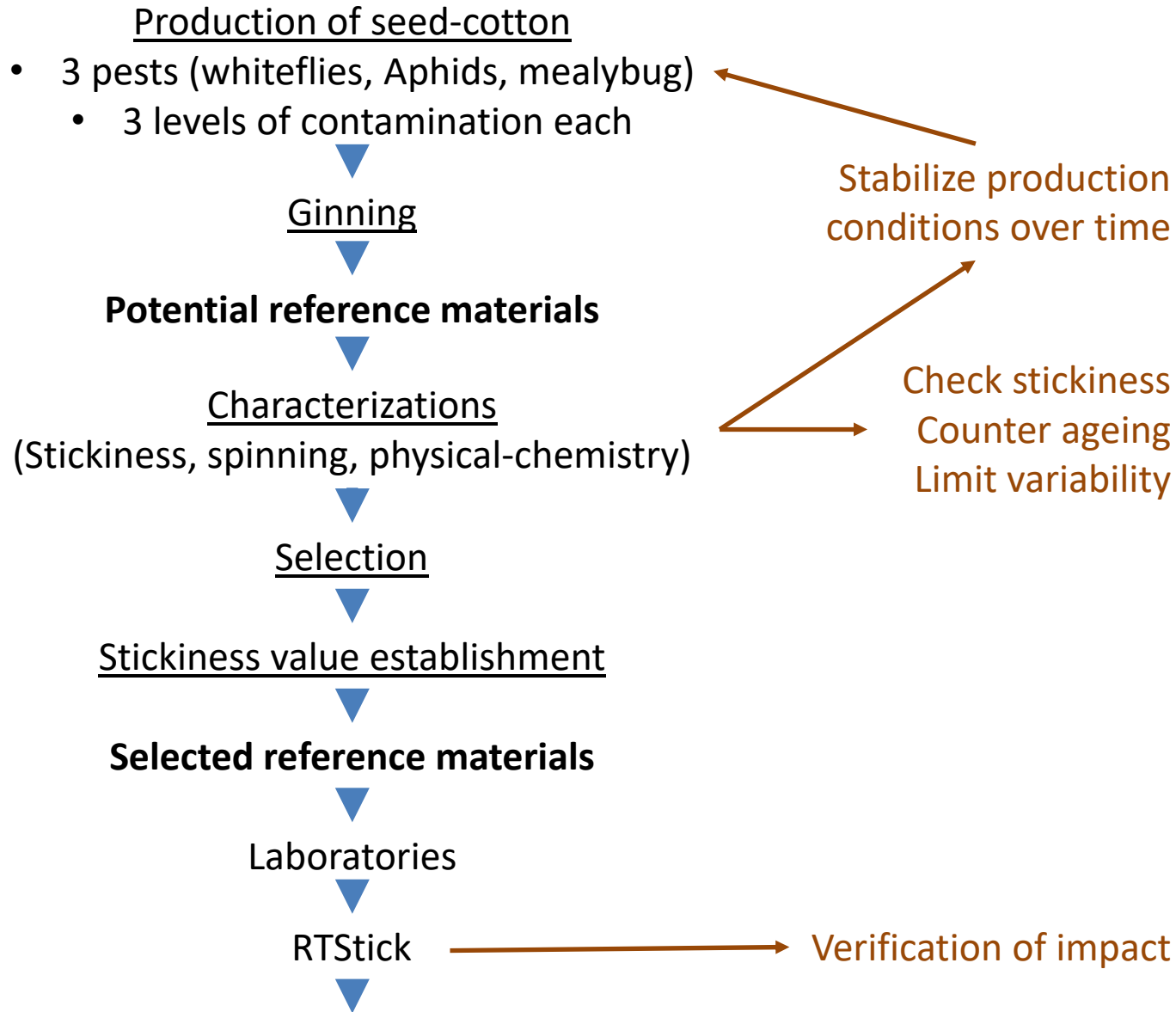➔need creation of reference materials
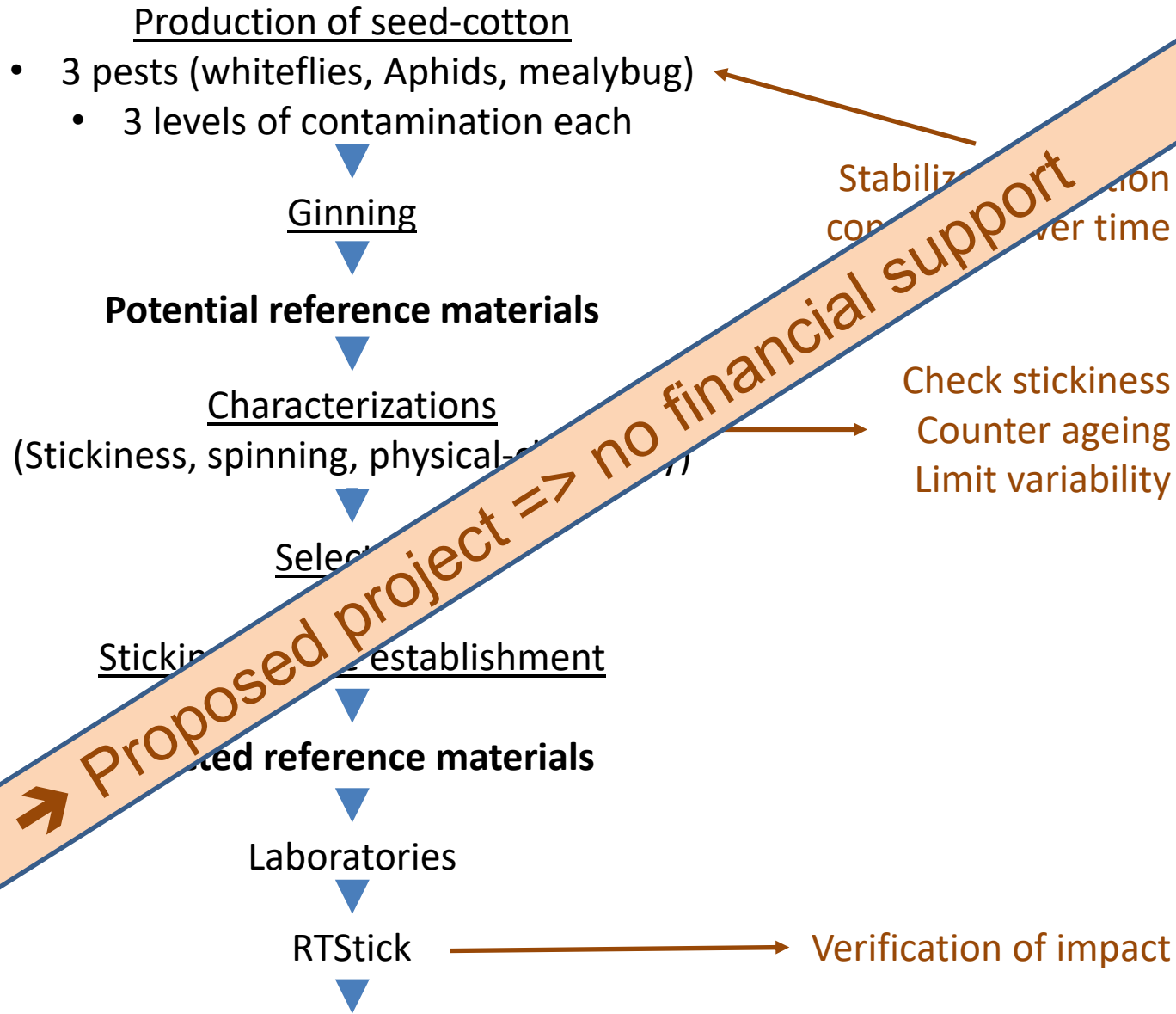➔proposed project

Proposed project HarCoStiC

Production of seed-cotton
- 3 pests (whiteflies, Aphids, mealybug)
  - 3 levels of contamination each

▼

Ginning

▼

Potential reference materials

▼

Characterizations
(Stickiness, spinning, physical-chemistry)

Stabilize production
conditions over time

Check stickiness
Counter ageing
Limit variability

Harmonization of Cotton Stickiness Characterization

Proposed project HarCoStiC

Production of seed-cotton
- 3 pests (whiteflies, Aphids, mealybug)
  - 3 levels of contamination each

▼

Ginning

▼

Potential reference materials

▼

Characterizations
(Stickiness, spinning, physical-chemistry)

▼

Selection

▼

Stickiness value establishment

▼

Selected reference materials

▼

Laboratories

▼

RTStick

▼

Harmonization of Cotton Stickiness Characterization

Stabilize production conditions over time

Check stickiness
Counter ageing
Limit variability

Verification of impact

Stepwise improved procedure

Proposed project HarCoStiC

Production of seed-cotton
- 3 pests (whiteflies, Aphids, mealybug)
  - 3 levels of contamination each

Stabilization conover time

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Potential reference materials

Characterizations
(Stickiness, spinning, physical-)

Check stickiness
Counter ageing
Limit variability

Selec

Stickie establishment

ed reference materials

Laboratories

RTStick → Verification of impact

Harmonization of Cotton Stickiness Characterization

➔ Proposed project => no financial support

Stepwise improved procedure

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Proposed project HarCoStiC

Production of seed-cotton
- 3 pests (whiteflies, Aphids, mealybug)
  - 3 levels of contamination each

Ginning

Potential reference materials

Characterizations
(Stickiness, spinning, physical-c...)

Selec...

Sticki... establishment

...ted reference materials

Laboratories

RTStick

Stabiliz... ...tion
con... ...er time

Check stickiness
Counter ageing
Limit variability

Verification of impact

Stepwise improved procedure

→ Proposed project => no financial support

Harmonization of Cotton Stickiness Characterization

Periodic international
inter-laboratories round-tests on stickiness

- One RTStick initiated in 2017
  - Planned for 2 times / year
  - Possibility of having up to 6 measurements / cotton
  - Anonym participation (one LabID / test)
  - 2017-1 and 2017-2 conducted

- Organized and funded by CIRAD, BBB and FIBRE

- Methods
  - No micro-spinning
  - Used in laboratories

- Three cottons (various origins) per test covering a range

Periodic international inter-laboratories round-tests

- Please fill-in the following information table :

Item / question	Answers	
Lab name / company (will stay confidential)		
Lab code number		
Name of the Person in charge		
Name of the Technician in charge of the tests		
Date of the tests (DD/MM/YYYY)		
Temperature of the lab when testing		°C
Relative humidity in the lab when testing		%
Duration of the sample conditioning prior testing (hours)		hours
Name of the testing technique		
Identification of the instrument (necessary at least in case of more than one instrument participating)		
Unit used for reporting results in the following table (sticky points, grade, percent, or else)		
If else, please explain the scale used		

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Periodic international inter-laboratories round-tests

- Please fill-in this table with your results:

Sample	Sample A	Sample B	Sample C
Result 1			
Result 2			
Result 3			
Result 4			
Result 5			
Result 6			
Comment for sample			

Participation in 2 RTs

Methods	RT 2017-1	RT 2017-2*	RT 2018-1
Benedict	1	0	
Caramelization	4	4	
Clinitest	1	1	
Contest	3	3	
Fibermap	2	2	
H2SD	4	3	New labs welcomed
KOTITI	1	1	
Minicard	4	4	
Quantitative meth.	1	1	
Reactive spray/heat	1	1	
SCT	13	11	
GB/T13785-1992	(1)	1	
Total	**35(+1)**	**32***	**(40)**

*: Interim counting due to late delivery of samples in laboratories.

Report format

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Sticky Cottons in RTs

- ## RT_2017-1:

  Check 1
  - A (blended using CSITC blending machine)
  - B (same cotton as A, blended using mini-card with smooth flats)
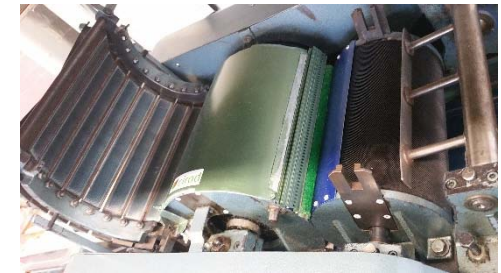 - C (blended using CSITC blending machine)

- ## RT_2017-2:

  - A, B, C, blended using CSITC blending machine
  - Check 2 A = C of 2017-1

- ## RT_2018-x:

  - 10 kg/cotton required,
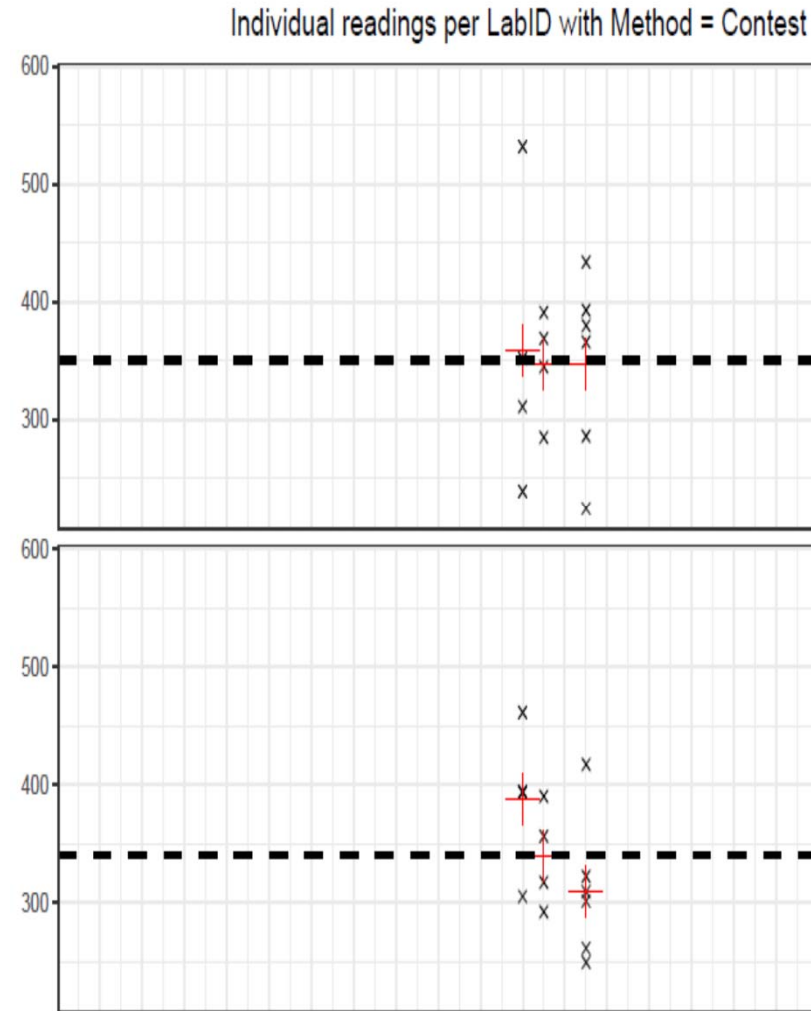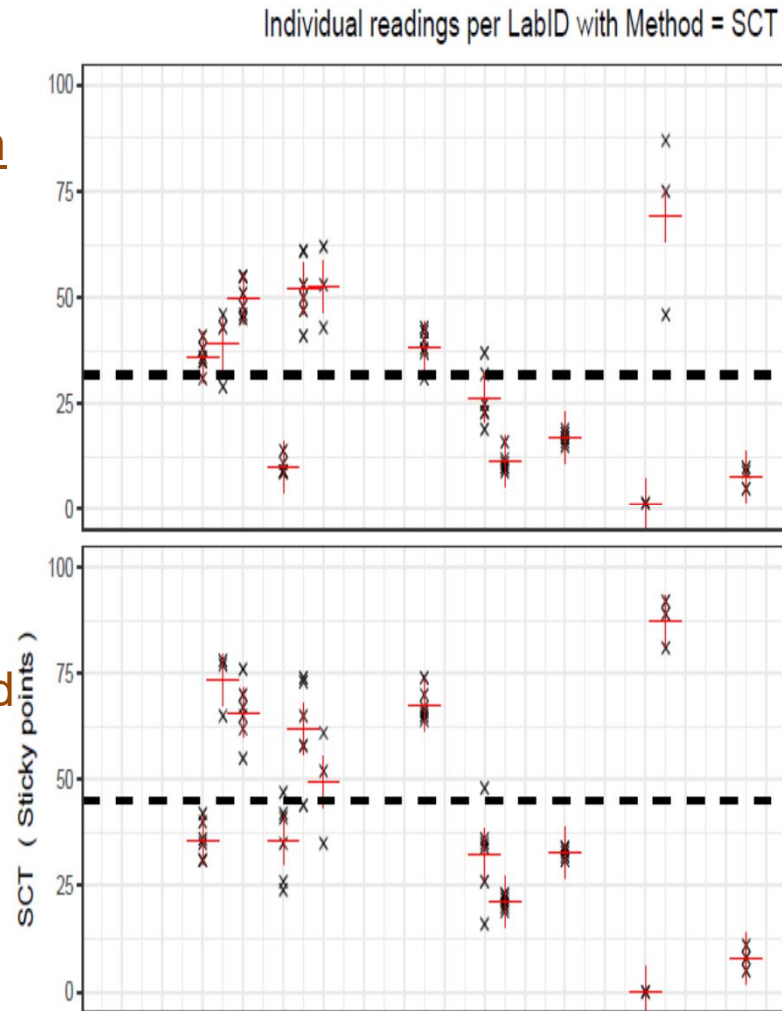 - call for anonym provision of sticky cottons for feeding RTs

Check 1: effect of preparation
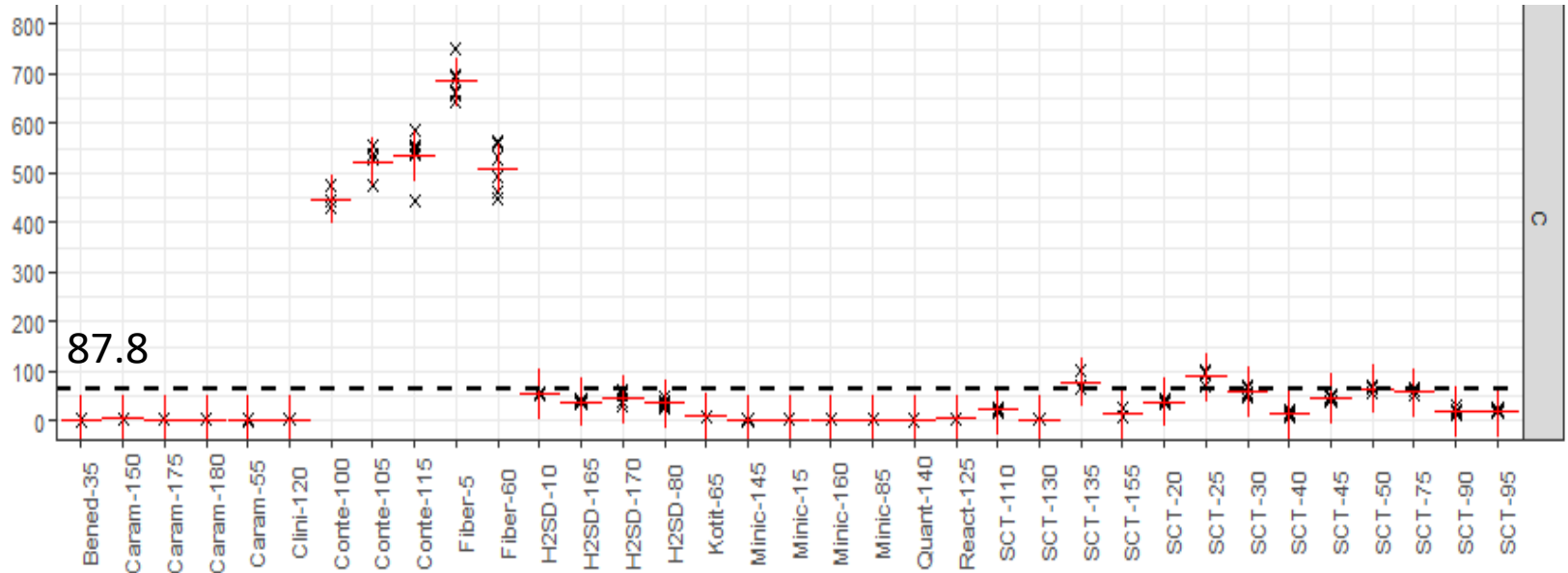

Preparation

CSITC blending machine

Mini-card with smooth flats

Individual readings per LabID with Method = SCT

Individual readings per LabID with Method = Contest

SCT (Sticky points)

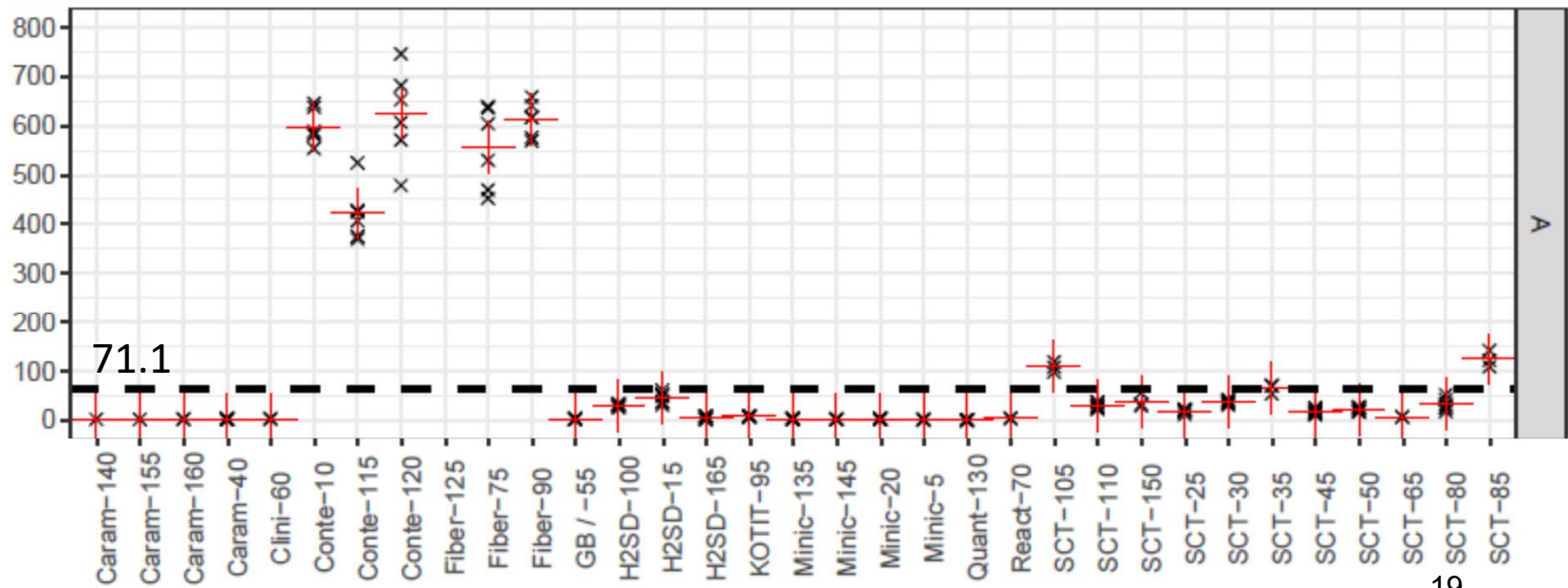| Cotton	SCT			Contest		
	Mean	Mean SDs	CV%	Mean	Mean SDs	CV%
A	32	5.8	18	350	82.6	24
B	45	6.0	13	340	55.6	16

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Check 2: one cotton in 2 RTStick

RTStick 2017-1 C

RTStick 2017-2 A*

*: Interim Report due to late delivery of samples in laboratories.

Units and scales for stickiness results

Methods	Unit	Scale RT 2017-1
Benedict	grade	0 - 2
Caramelization	+b	0 - 3.5
Clinitest	+b	0 - 2.5
Contest	grade	0 - 585
Fibermap	grade	0 - 750
H2SD	points	0 - 60
Kotiti	grade	0 - 9
Minicard	ITMF grade	0 - 3
Quantitative meth.	%	0 - 1.2
Reactive spray/heat	grade	0 - 4.5
SCT	points	0 - 100

Units and scales for stickiness results
RTStick 2017-1

Individual readings per Method and LabID

+: Lab mean   x: Lab ind. results   - - - -: Overall mean / cotton
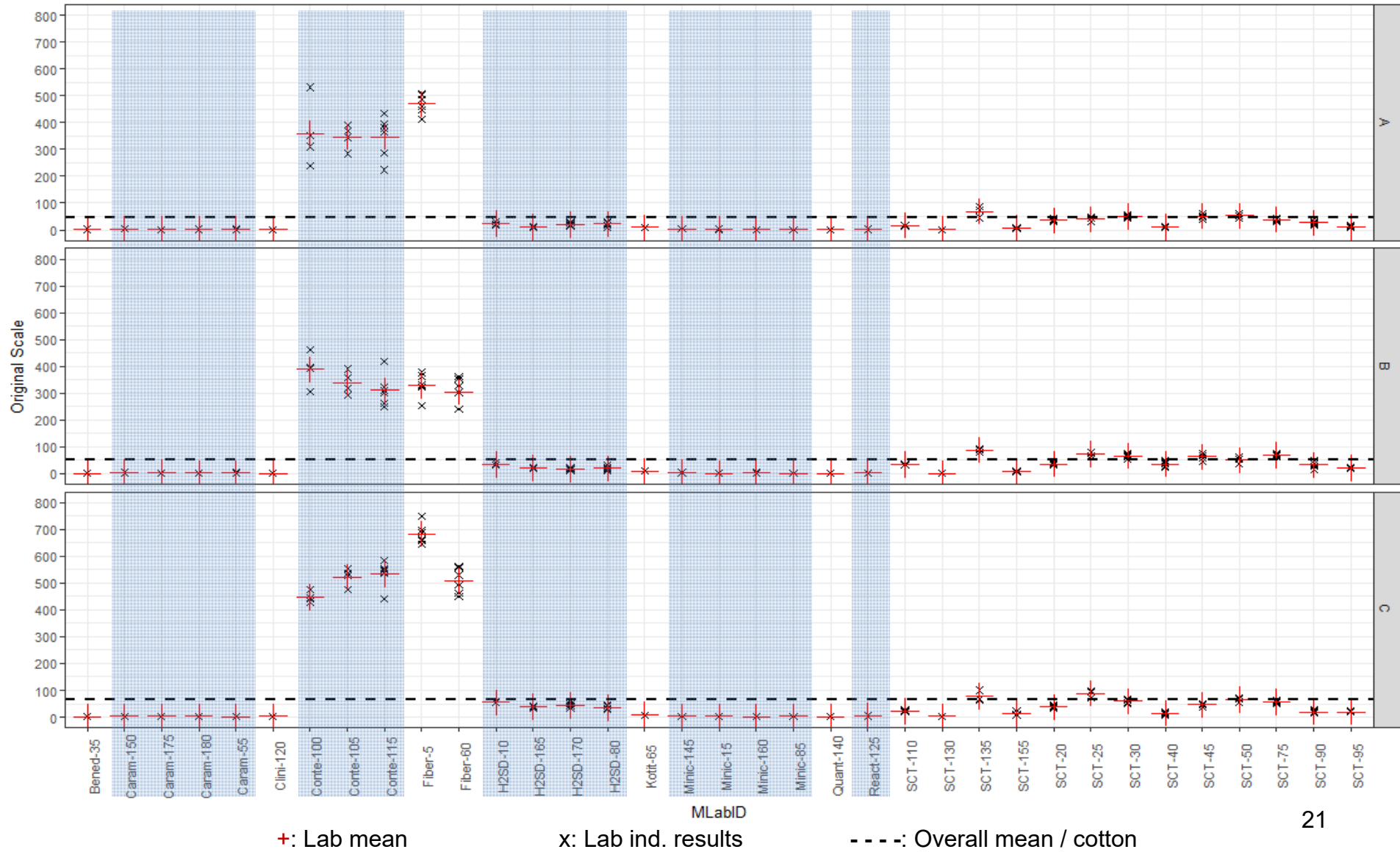
Units and scales
Could we express results in a common scale?

What for?

- Compare methods
  - Sensitivity, Resolution, Precision, Accuracy,
  - Reproducibility
  - Repeatability

- Improve
  - Correlation within and between methods
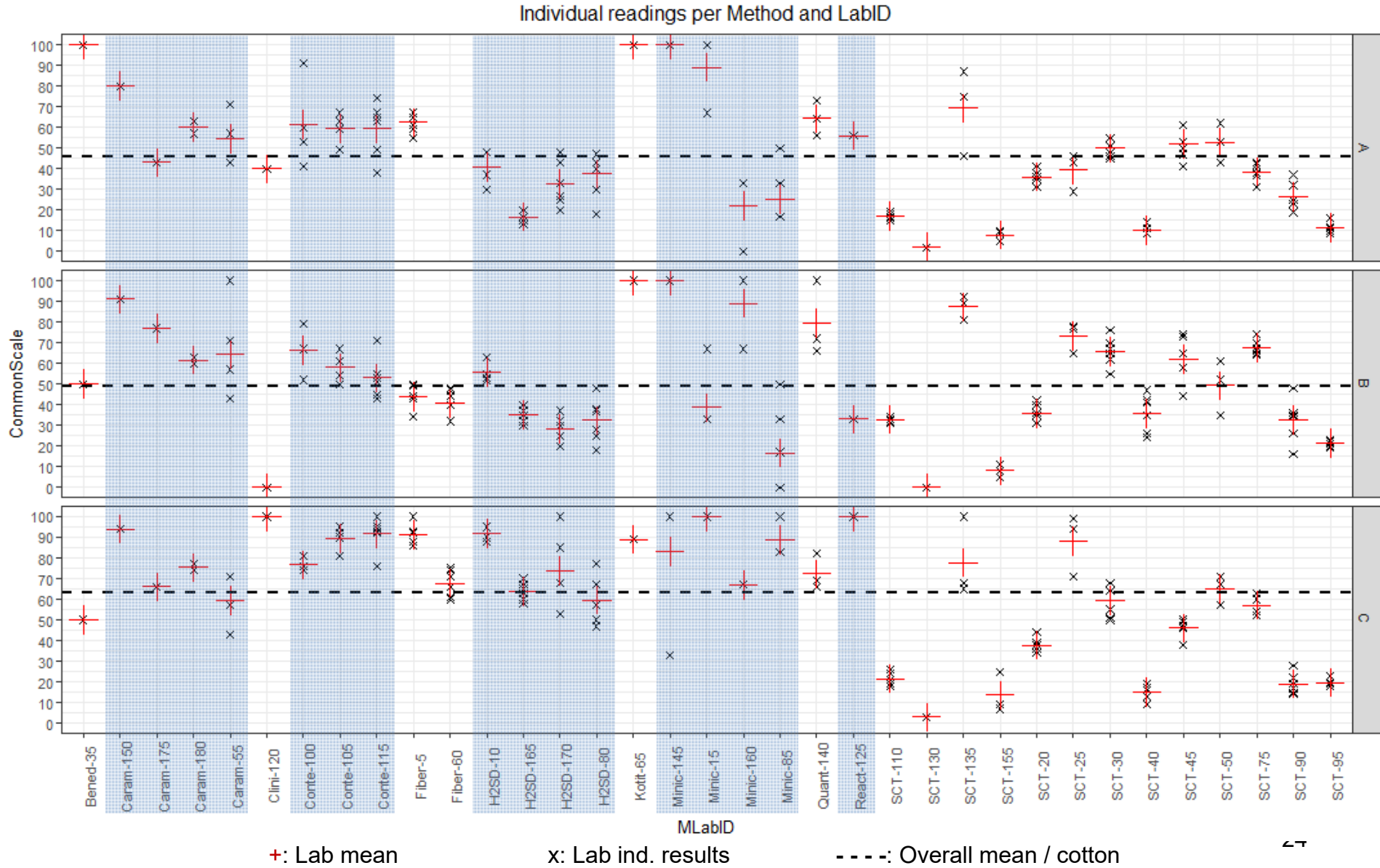
Units and scales
Example: xMax / RT 2017-1

Method	Result	xMax test	CommonScale
Minicard (grades)	3	3	**100**
	2		**67**
	1		**33**
	0		**0**
SCT (nb sticky points)	100	100	**100**
	66		**66**
	63		**63**
	0		**0**
A (any unit)	555	600	**93**
	200		**33**
	190		**32**
	0		**0**

Rounded(100*Result/xMax;0)

'Common' scale for stickiness measurements? Application xMax to RTStick 2017-1

Individual readings per Method and LabID

+: Lab mean   x: Lab ind. results   - - - -: Overall mean / cotton

'Common' scale for stickiness measurements?
Example : xMaxEver

Method	Result	xMaxEver	CommonScale
Minicard (grades)	3	3	**100**
	2		**67**
	1		**33**
	0		**0**
SCT (nb sticky points)	100	100 => 200	**50**
	66		**33**
	63		**32**
	0		**0**
A (any unit)	555	600 => 1000	**55**
	200		**20**
	190		**19**
	0		**0**

Rounded(100*Result/xMaxEver;0)

Units and scales
Could we express results in a common scale?

Interesting and usefull only if <u>really</u> the same indicator is measured

⇒ **<u>S</u>tickiness or any indicator proven closely related to stickiness**

"the propensity of fibers to stick to spinning parts during their processing"

=> Any method subject to recognition should demonstrate that point

Subject to discussion…

Additional notes: respect of testing conditions

- Minicard test should be performed at 55%RH according to 1988 ICCTM proceedings, page 12:

Atmosphere - $24^{O}C \pm 1^{O}C$, $55\% \pm 2\%$ RH

Sample size - 10g, 11 x 25cm batt

Passage time - 3 to 5 min

Stickiness ratings:

       0 - No stickiness
       1 - Light stickiness
       2 - Moderate stickiness
       3 - Heavy stickiness

- Other methods: Subject to discussion…

Estimated costs of each RTStick occurrence

- Organization
  - 5 days   5000 €
- Stock management
  - ?
- Cotton selection and preparation
  - 500 € / kg, 3 cottons * 5 kg   7500 €
- Expedition
  - 20 € / lab, 35 labs =>   700 €
- Data analysis and report preparation
  - 5 days   5000 €

> 18000 €

Organization of future RTStick?
Subject to discussion…

- **The effect of ETO fumigation on stickiness**
  René van der Sluijs

- **Contest-Fibermap**: The first year of field testing on stickiness detection
  Gabrielle Salvinelli

- Embrapa: Developing a Portable Device Using NIR Image for Easy Detection of Stickiness in Cotton
  Liv Severino

- KOTITI

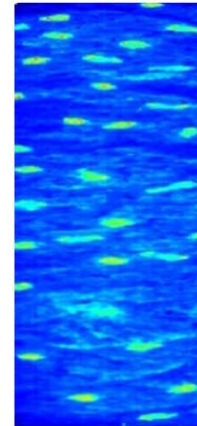- Prodev

- CIRAD

- And a surprise …

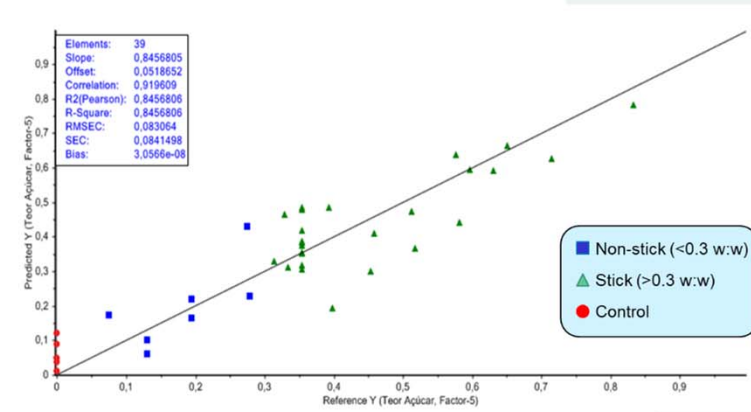# Using NIR Image for Detection of Stickiness in Cotton

Cotton samples: sugar spots are invisible

High resolution image acquired in NIR-HYS

After filtering sugar spots become visible

Sugar content is estimated in a cheap, fast, reliable and non-destructive method.

This method is under development. The proof of concept was successful and test are initiating in a textile industry.

More information:
liv.severino@embrapa.br
everaldo.medeiros@embrapa.br
Brazil

Full presentation: Session IX (Friday, 9-11:30, Room 506)

ISO 12027:2012 Textiles—Cotton-fibre Stickiness
## — Detection of sugar by color reaction

- New ISO standard **visualizes the honeydew droplets in cotton** and evaluates the degree of honeydew contamination.
- Honeydew droplets that may occur in a specific cotton web with its fixed surface area and mass are transferred to color reaction paper by applying pressure.
- The transferred honeydew is developed on the color reaction paper by using forced convection oven at 120℃ for 5min.
  → According to HPLC results of color reaction points, **trehalulose and melezitose could be detected.**
- The field test on this new test method was done and the results showed very good relationship with actual stickiness behavior
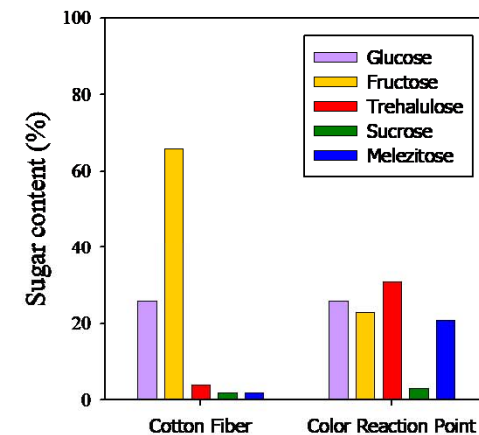

Sticky cotton
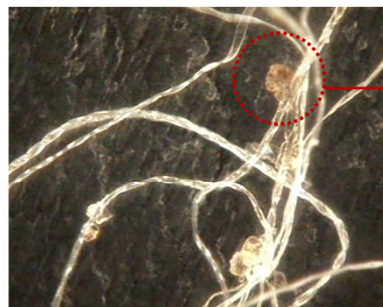
Honeydew droplets
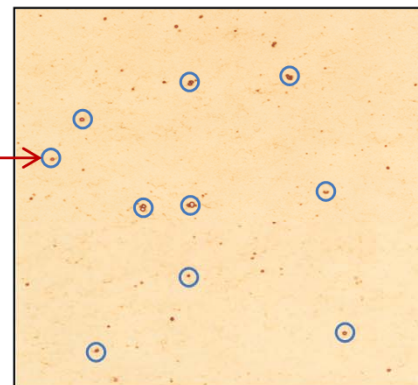
Color reaction paper

HPLC results of color spots

Test procedure

STEP 1
Preparation

- **Preparation of sample web**
  - A web (10 × 10cm) is prepared after impurities are removed by using the instruments such as MDTA 3.
- **Preparation of color reaction paper**
  - By wetting paper having a certain degree of pore size with a solution for color reaction

STEP 2
Color reaction

(1) contacting sample web with color reaction paper

(2) Development of color reaction paper

- **Transfer of honeydew from sample web to color reaction paper by contacting together**
  - Pressing condition
    - *weight : 4 kg / (10 × 10 cm), time : 1 min*
- **Development of color (by using instrument such as forced convection oven)**
  - Development condition of color reaction paper
    - *120 ℃ × 5 min*

STEP 3
Grading of cotton stickiness

- Grading by comparing with standard replicas under visual evaluation
- Grading by image analysis program

KOTITI
Testing & Research Institute

Visual assessment using cotton fibre stickiness replicas

- Stickiness grade is evaluated by comparing the appearance of **the developed brown spots(honeydew reaction)** with a series of five visual replicas

Manufacture of SCT and of H2SD by

Prodev System

Zac de la Louvade

221 rue des Aramons

34130 Mauguio

France

Email: contact@prodev-system.fr

Tel: +33 (0)4 67 12 12 42

Fax: +33 (0)4 34 43 72 00

Cirad

- Produces a small quantity of reference materials for calibrating SCT and H2SD

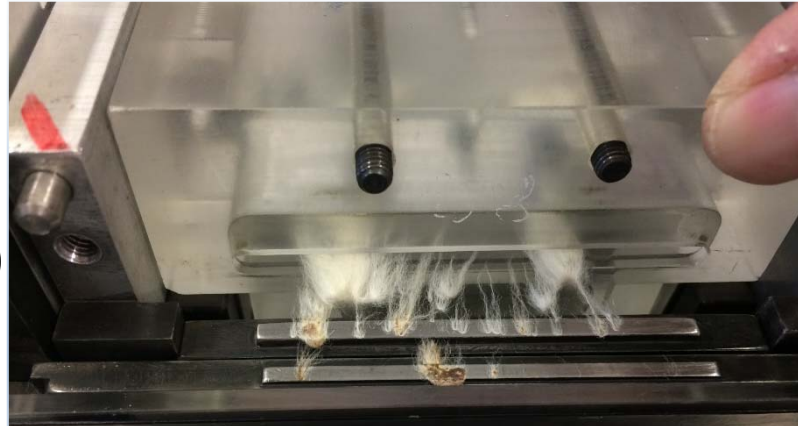- Can check SCT instruments using a 30 years old 'standard' routine

Email: serge.lassus@cirad.fr
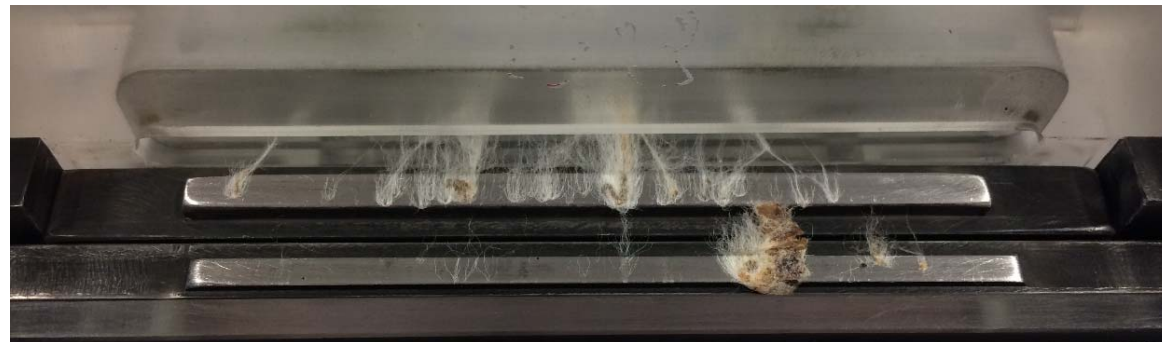technologie.coton@cirad.fr
coton@cirad.fr

And… what is the surprise?

Stickiness…*
Also look into your instrument!

After 1 800 combs
(300 samples * 6 meas.)

**After 1 620 combs
(270 samples * 6 meas.)**

*: Experimentation results with intentional use of sticky to very sticky cottons

Stickiness…*
Also look into your instrument!

Cleaning at the beginning

After 480 combs

After 960 combs

After 1440 combs

Photos Ocana and Gourlot   *: Experimentation results with intentional use of sticky to very sticky cottons

Discussion conclusion
ICCTM-Stickiness TF 2018

- RTStick 2017: Conclusions: similar to RT2013-14
    - Still remain differences between labs within each method
    - Still remain large differences between methods
- RTStick: Organizational matters
    - Sample dispatch has been problematic
    - Feeding the RT with cotton: call for proposals
    - RTStick will continue
- Proposal of a method to compare results
- Proposal of HarCoStiC* project

    Reference materials + a common scale + RT Stick
    = basis of harmonized measurements

- Progress report for Contest and Fibermap
- NIR technology in Embrapa
- Prodev continues producing SCT and H2SD
- Production of some reference materials / check of SCTs

*: Harmonization of Cotton Stickiness Characterization