International Textile Manufacturers Federation (ITMF)  
International Cotton Committee on Testing Methods (ICCTM)  
Stickiness session

International round-test on stickiness measuring methods: 
new results and proposal for an harmonization step forward

GOURLOT J.-P., LASSUS S. and GAWRYSIAK G.  
Bremen, March 2016
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 gossypii*  
Honeydew on open boll

*Bemisia tabaci*  
Honeydew in fibers

Problems  
Productivity, quality

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


Mandates

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.” [.../…]
Objectives of the international inter-laboratory round-test

- 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
Stickiness measurement (any time)

Chemical methods
- Simple
  - Perkins
  - Fehling
  - Color reaction
  - KOTITI
- Complex
  - HPLC
  - GC

Physical techniques
- Infra-red
- Mini-card

Mechanical
- ITMF Reference method
- ITMF Recommended method

Thermo-mechanical
- SCT
- H2SD
- FCT / FQT
- Quickspin
Stickiness measurements (in RT2013)

**Chemcare**
(spray)

**Caramelization**
(differences in +b measurements before and after a heating treatment (150°C, 25 minutes)

**KOTITI**
colour-reaction paper => incubation => comparison to five visual standards (ISO 12027-2012(E))

Not reported as still under data analysis

As reported by Perkins in ‘ITMF grades’

<table>
<thead>
<tr>
<th>Number</th>
<th>Description</th>
</tr>
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<tbody>
<tr>
<td>0</td>
<td>No stickiness</td>
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<tr>
<td>1</td>
<td>Light stickiness</td>
</tr>
<tr>
<td>2</td>
<td>Moderate stickiness</td>
</tr>
<tr>
<td>3</td>
<td>Heavy stickiness</td>
</tr>
</tbody>
</table>

**Number of sticky points**

**Chemical extraction**: mg/100g of fibers

Records of productivity and quality parameters
Not reported as still under data analysis
Within-technique, between laboratories
Within-technique, between laboratories

Evaluation by one single person while the sample preparation was made by two independent laboratories.
Within-technique, between laboratories

Correlations

<table>
<thead>
<tr>
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<th>H2SD-50</th>
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Within-technique, between laboratories

Correlations

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Scatterplot Matrix
Within-technique, between laboratories

Correlations

<table>
<thead>
<tr>
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</table>

Scatterplot Matrix

- Mini-card-5
- Mini-card-76
- Mini-card-81
Within-technique, between laboratories

<table>
<thead>
<tr>
<th>Correlations</th>
<th>SCT-17</th>
<th>SCT-23</th>
<th>SCT-37</th>
<th>SCT-51</th>
<th>SCT-53</th>
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<td>0.9253</td>
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</table>
## Participating techniques and laboratories

<table>
<thead>
<tr>
<th>Technique</th>
<th>Number of laboratories per stickiness technique</th>
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<tbody>
<tr>
<td>Caramelization</td>
<td>3</td>
</tr>
<tr>
<td>Chemcare</td>
<td>2</td>
</tr>
<tr>
<td>H2SD</td>
<td>3</td>
</tr>
<tr>
<td>KOTITI</td>
<td>4</td>
</tr>
<tr>
<td>Mini-card</td>
<td>3</td>
</tr>
<tr>
<td>SCT</td>
<td>9</td>
</tr>
<tr>
<td>Total sugar</td>
<td>1</td>
</tr>
<tr>
<td><strong>Total Nb</strong></td>
<td><strong>25</strong></td>
</tr>
</tbody>
</table>
Conclusions … before discussions

• The meanings of the results provided by the various measuring techniques are not equivalent, even though they intend to measure/predict the same phenomenon: stickiness
• Units are fully different (grades, numbers, masses…)
• Observed differences in readings, both within laboratories using the same technique, and between techniques
Conclusions … before discussions

- Need for a harmonization
- Which are the ways to achieve this harmonization?
- What to recommend?
- Requires policies and support tools to continue
Objectives of the international inter-laboratory round-test

• 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
• To check the level of ability of each measuring technique to predict stickiness as measured by the reference method and the recommended method
• To check the level of ability of each measuring technique to predict stickiness as measured by a micro-spinning test
Operating method used for producing yarn in a micro ring-spinning facility

ITMF-ICCTM inter-laboratory round test on stickiness, 2014

Lab opening machine
2 fleeces
(L=1.75 m each; tex=31000)

Mini-card
1 fleece
(L=1.75 m; tex=57200)

Drawing frame, pass 1
5 slivers
(L=3.35 m each; tex=5800)

Drawing frame, pass 2
10 slivers
(L=3.35 m each; tex=2900)

Drawing frame, pass 3
2 slivers
(L=37.40 m each; tex=2300)

Spinning frame
10 bobbins
(L=500 m each; tex=20)

Eveness Tester: 100 m * 2.5 min / bobin
Strength Tester: 100 breaks / bobin @ 0.3 second per break

11 cottons * 2 RH * 2 blocks

Observations made during spinning on the ring spinning frame

ITMF-ICCTM inter-laboratory round test on stickiness, 2014

Drawing frame
Spinning frame

Up to one revolution
Requires no human intervention
No machine stop
= Attachment (A)

or

Requires human intervention
to continue production of yarn
No machine stop
= Rolling-up (R)

+ breaks (B)
Machine stop
+ required cleanings (C)

After more than one revolution...
... it could be adding-up ...

Position 1
Position 2
Spinning conditions

Chosen RH conditions

1. Spinning tests: first RH conditions
2. Drying period for cottons (72 hours)
3. Spinning tests: second RH conditions

<table>
<thead>
<tr>
<th></th>
<th>T°C</th>
<th>RH%</th>
<th>g H₂O/g Gaz</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>24</td>
<td>60</td>
<td>11.18</td>
</tr>
<tr>
<td>2</td>
<td>23</td>
<td>35</td>
<td>6.09</td>
</tr>
<tr>
<td>3</td>
<td>24</td>
<td>45</td>
<td>8.35</td>
</tr>
</tbody>
</table>
T°C and RH% records during the spinning experiment

- Setting at 58%RH
- Drying + pre-conditioning
- Setting at 45%RH
### Recorded parameters for Yarn (Ys)

#### Quality (28)
- Um, CVm, CVm1
- Indice
- Tex
- Pil, Sh, Sh1
- Thin30, 40, 50, 60
- Thick35, 50, 70, 100
- Nep1s140, 200, 280, 400
- Fmax, CVFmax, Ten, WorkMax, N/texM1, N/texM2, All, CVAI1

#### Productivity (8)
- Soulèv. / attachments
- Enroul. / rolling-up
- Nettoy. / cleaning
- Casses / break
- Events
- Events/km
- L_fil_produite m
- Prod m/mn
## Recorded parameters

### For fiber (Xs)

<table>
<thead>
<tr>
<th></th>
<th>Number of Tech_LabID</th>
<th>Mean</th>
<th>CF</th>
<th>Ranks*</th>
<th>SO</th>
<th>PCA</th>
<th>Total</th>
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<tbody>
<tr>
<td>Caramelization</td>
<td>3</td>
<td>1</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>1</td>
<td>14</td>
</tr>
<tr>
<td>Chemcare</td>
<td>2</td>
<td>1</td>
<td>2</td>
<td>2</td>
<td>2</td>
<td>1</td>
<td>10</td>
</tr>
<tr>
<td>H2SD</td>
<td>3</td>
<td>1</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>1</td>
<td>14</td>
</tr>
<tr>
<td>KOTITI</td>
<td>4</td>
<td>1</td>
<td>4</td>
<td>4</td>
<td>4</td>
<td>1</td>
<td>18</td>
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<td>Mini-card</td>
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<td>1</td>
<td>3</td>
<td>3</td>
<td>3</td>
<td>1</td>
<td>14</td>
</tr>
<tr>
<td>SCT</td>
<td>9</td>
<td>1</td>
<td>9</td>
<td>9</td>
<td>9</td>
<td>1</td>
<td>38</td>
</tr>
<tr>
<td>Total sugar</td>
<td>1</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>1</td>
</tr>
<tr>
<td>Total Nb</td>
<td>25</td>
<td>6</td>
<td>24</td>
<td>24</td>
<td>24</td>
<td>6</td>
<td>121</td>
</tr>
</tbody>
</table>

*: Partly studied and shown in 2014
Strange #10 cotton…
(Not taken into account from now on)

RH: 58%

RH: 45%
**Percent of significant relationships (α=5%)**

**Yarn = f (Fiber)**

<table>
<thead>
<tr>
<th>Without cotton #10</th>
<th>Caram - 45%</th>
<th>Card - 45%</th>
<th>Chem - 45%</th>
<th>H2SD - 45%</th>
<th>Kotiti - 45%</th>
<th>SCT - 45%</th>
<th>Caram - 58%</th>
<th>Card - 58%</th>
<th>Chem - 58%</th>
<th>H2SD - 58%</th>
<th>Kotiti - 58%</th>
<th>SCT - 58%</th>
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</thead>
<tbody>
<tr>
<td><strong>Productivity (max=8)</strong></td>
<td>0</td>
<td>1</td>
<td>4</td>
<td>1</td>
<td>1</td>
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<td>6</td>
<td>7</td>
<td>5</td>
<td>6</td>
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<td>6</td>
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<tr>
<td><strong>Quality (max=28)</strong></td>
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<td>20</td>
<td>21</td>
<td>19</td>
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<td>16</td>
<td>18</td>
<td>22</td>
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<td>17</td>
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<td>29</td>
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<td>21</td>
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<td><strong>Nb total</strong></td>
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<td>36</td>
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<td>36</td>
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<td>36</td>
<td>36</td>
<td>36</td>
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<td>36</td>
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<tr>
<td><strong>Percent of significant relationships between fiber and yarn parameters</strong></td>
<td>28</td>
<td>58</td>
<td>69</td>
<td>56</td>
<td>50</td>
<td>47</td>
<td>67</td>
<td>81</td>
<td>61</td>
<td>78</td>
<td>58</td>
<td>78</td>
</tr>
</tbody>
</table>

Green : > 70% of significant cases  
Orange: between 60 and 70% of significant cases  
Violet: between 50 and 60% of significant cases  
No color below 50%
With/without #10 cotton: method sensitivity to outlier

<table>
<thead>
<tr>
<th>Method</th>
<th>Without #10</th>
<th>With #10</th>
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<tbody>
<tr>
<td>Caram</td>
<td>86%</td>
<td>69%</td>
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<tr>
<td>Card</td>
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<td>Card</td>
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<td>Chem</td>
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<td>Chem</td>
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<td>H2SD</td>
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<td>Kotti</td>
<td>69%</td>
<td>58%</td>
</tr>
<tr>
<td>Kotti</td>
<td>69%</td>
<td>50%</td>
</tr>
<tr>
<td>SCT</td>
<td>78%</td>
<td>61%</td>
</tr>
<tr>
<td>SCT</td>
<td>81%</td>
<td>47%</td>
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Pourcent de relations significatives entre collage et caractéristiques de fil (sans 10)
Pourcent de relations significatives entre collage et caractéristiques de fil (avec 10)
Acknowledgements for contributions in the RT

• Contributors: Dr. A. Prades, N. Talha, Dr. J. Rodgers, Dr. C. Delhom, Dr. C. Fortier, Dr. E. Gozé, Prof. N. Ali, Dr. A. H. Abdelatif, M. Giner, Dr. E. Gérarddeaux

• Participating laboratories

• Sponsors and technical partners: A. Macdonald (CSITC), Dr. T. Townsend (ICAC), Dr. J.-L. Chanselme (Cotimes)

• Funding:
  – Cirad
  – Participating laboratories for their own characterizations
What’s next concerning any harmonization process?

a path toward a joint project...
Prod. Seed-cotton in greenhouse or in field

Seed-cotton samples

Ginning

Fiber samples

Fine characterization
Textile characterization
Stickiness characterization

Potential Reference materials

Value establishing

Prod. Individual sugars by synthesis

+ Fiber and homogenization

Results / information

Materials

Interpretation / management

Reference materials
On sucking insects, **honeydew** and sugar synthesis

---

Production of seed-cotton in greenhouse or in field

On field observations

Seed-cotton samples

Ginning

Fiber samples

Fine characterization

Textile characterization

Stickiness characterization

Potential Reference materials

Value establishing

Reference materials

---

On methods

Study how to produce
Study homogenization/variability
Study impact of ageing
Study packaging vs ageing
Study storage / distribution
Study sugar synthesis

Study fine characterization
Study textile characterization
Study stickiness characterization

---

Results / information
Materials
Interpretation / management

---

Bibliography
On sucking insects, honeydew and sugar synthesis

**Context**
- Study value establishing
- Study fine characterization
- Study textile characterization
- Study stickiness characterization

**Procedure**
- Prod. Seed-cotton in greenhouse or in field
- On field observations
- Prod. Individual sugars by synthesis

**Database**
- Seed-cotton samples
- Ginning
- Fiber samples
- Fine characterization
- Textile characterization
- Stickiness characterization
- Potential Reference materials
- Value establishing

**Results / information**
- Study how to produce
- Study homogenization/variability
- Study impact of ageing
- Study packaging vs ageing
- Study storage / distribution
- Study sugar synthesis

**Materials**
- Prod.

**Interpretation / management**
- Study value establishing
- Study packaging vs ageing
- Study storage / distribution
- Study sugar synthesis
- Study fine characterization
- Study textile characterization
- Study stickiness characterization

**Bibliography**
On sucking insects, **honeydew** and sugar synthesis

**Context**

- Study how to produce
- Study homogenization/variability
- Study impact of ageing
- Study packaging vs ageing
- Study storage / distribution
- Study sugar synthesis

**Database**

- Seed-cotton samples
- Fiber samples
- Ginning
- Fine characterization
- Textile characterization
- Stickiness characterization

**Procedure**

- Product. Seed-cotton in greenhouse or in field
- Prod. Aida-Carabe on field observations
- Prod. Individual sugars by synthesis
- Fiber and homogenization

**Results / information**

- Value establishing
- Potential Reference materials
- All, under control of ITMF, CSITC, managed by LTC(?)

**Materials**

- All, under ITMF, CSITC control, managed by LTC(?)

**Bibliography**

- LTC, LPMT, USDA
- Study how to produce
- Study homogenization/variability
- Study impact of ageing
- Study packaging vs ageing
- Study storage / distribution
- Study sugar synthesis

- LPMT, LTC, UMR95 (USDA in it)
- Study fine characterization
- Study textile characterization
- Study stickiness characterization

- LTC, USDA, ITMF
- ICAC, CSITC
- Study value establishing

**Potential partners...**
Under construction
Proposed activities

1 Project management
   1.1 Create and apply data management plan
   1.2 Organize initial workshop
   1.3 Organize intermediate workshops
   1.4 Prepare and distribute periodic scientific, technical, accounting and progress reports
   1.5 Organize diffusion of information between Project Members
   1.6 Organize diffusion of information outside the Project Members
   1.7 Participate to international seminars for diffusion of results
   1.8 Organize final workshop
   1.9 Manage data and information after the end of the Project (provision)

2 Work in entomology to know how and where to produce sticky cotton from known origins
   2.1 Study the insects populations and their dynamics under climate change pressure
   2.2 Study the insect's impact on the quantity and on the types of produced honeydews

3 Produce/collection of sticky fibers from various insects populations, alone or in various repartition ratios
   3.1 Organize the packaging, marking/labelling (in relation with database) and plan conservation of collected samples
   3.2 Study the possibilities of producing/collection sticky seed-cotton from various insects populations, alone or in various repartition ratios
   3.3 Produce seed-cotton contaminated by honeydew from known insects in greenhouse
   3.4 Produce seed-cotton contaminated by honeydew from known insects in Ecotron (not in this project)
   3.5 Produce seed-cotton contaminated by honeydew from known insects in cotton fields in producing countries (according to findings in literature)
   3.6 Attempt to synthesize individual sugars in quantities big enough to calibrate and check measuring devices
   3.7 Collect, homogenize, and gin seed-cotton into fibers per level in the range in standardized conditions

4 Study the characteristics of produced honeydew and individual sugars (as such and/or in fiber samples)
   4.1 Study available techniques for fine charaterizations of insect honeydew
   4.2 Study the impact of a 'calibration' or a 'leveling' of stickiness results from commercial instruments using reference materials
   4.3 Study homogenizing methods/techniques to insure a high uniformity of honeydew distribution and not disturbing honeydew deposits
   4.4 Analyze and quantify individual sugars in honeydew
   4.5 Study the behavior of individual identified sugars at temperature, at moisture change and at processing
   4.6 Study the impact of ageing on honeydew characteristics and design storage methods accordingly
   4.7 Plan budget for PhD, MS, ... students
   4.8 Study, plan and use preservation medias for long storage of reference materials
   4.9 Study the long-term stability of the reference stickiness materials set(s)

5 Establish a first reference materials set of sticky fibers
   5.1 Apply an homogenization if required
   5.2 Realize fine charaterizations on investigated potential future reference materials used for the round test
   5.3 Organize discussion within CSITC-TF participants
   5.4 Organize discussion within ITMF-ICCTM working groups participants

5.5 Organize international inter-laboratory round-tests using major fine and 'commercial' measuring techniques
5.6 Prepare report on the results and conclusions based on characterization results
5.7 Update an operating method on the way to produce, prepare, characterize, establish reference materials to be released
5.8 Constitute a first set of reference material
5.9 Inform users about the availability of the operating method and of the reference material set
5.10 Feed data management plan
5.11 Distribute report
Proposed activities

1. Project management
   1.1 Create and apply data management plan
   1.2 Organize initial workshop
   1.3 Organize intermediate workshops
   1.4 Prepare and distribute periodic scientific, technical, accounting and progress reports
   1.5 Organize diffusion of information between Project Members
   1.6 Organize diffusion of information outside the Project Members
   1.7 Participate to international seminars for diffusion of results
   1.8 Organize final workshop
   1.9 Manage data and information after the end of the Project (provision)

2. Work in entomology to know how and where to produce sticky cotton from known origins
   2.1 Study the insect’s populations and their dynamics under climate change pressure
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Thank you for your attention