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

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Bremen, March 2016

CIRAD UPR115 / Gesc
Laboratoire de Technologie et de Caractérisation des fibres naturelles

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…)

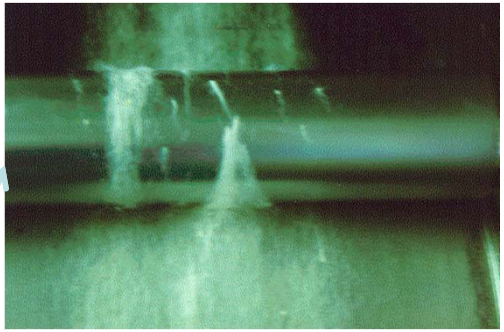
Aphis gossipii

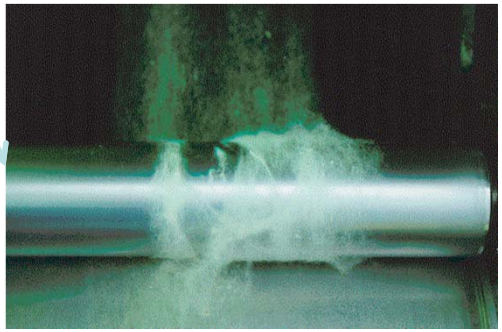
Honeydew on open boll

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**.”** […/…]

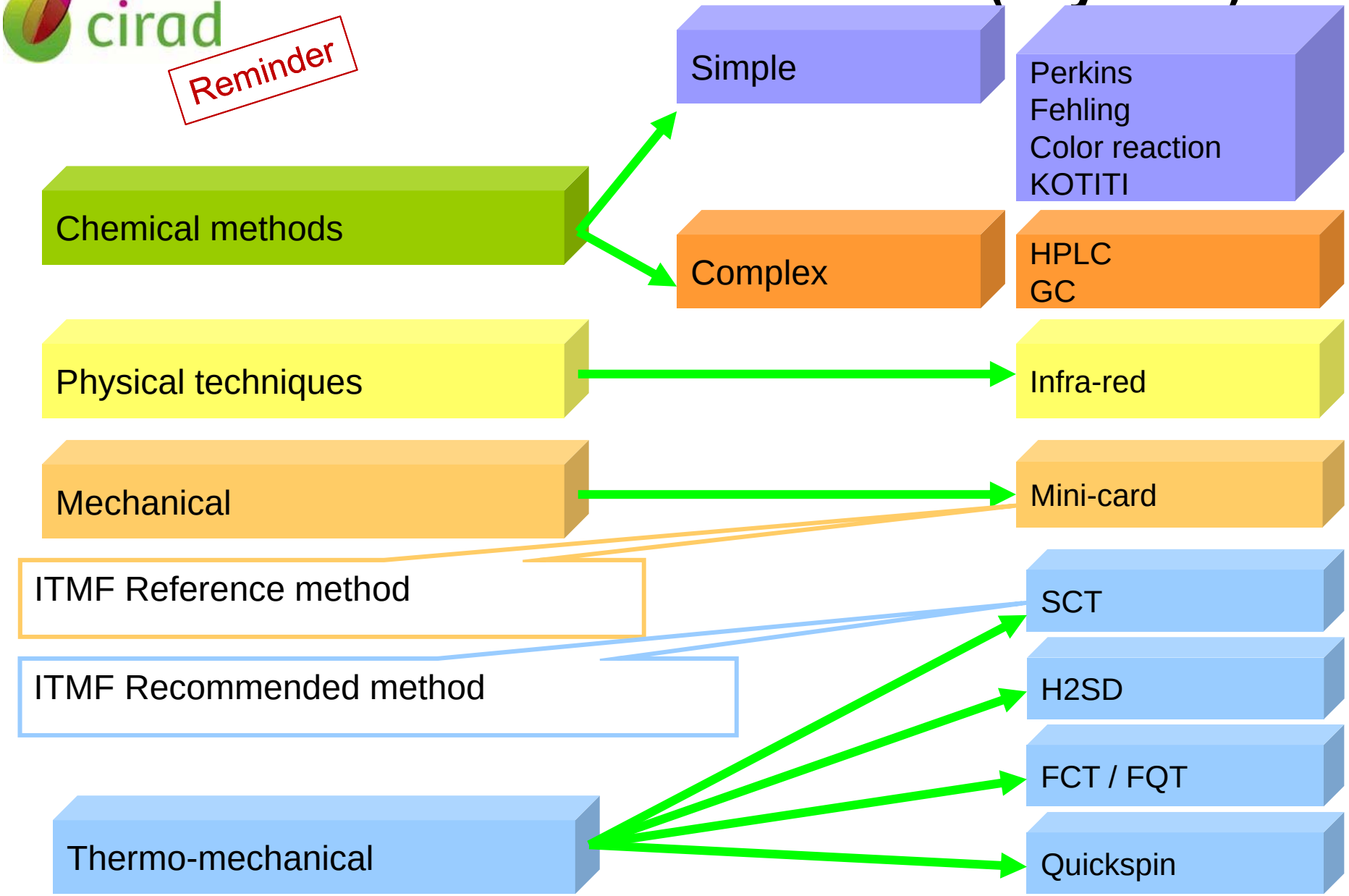
Reminder

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)

Reminder



Stickiness measurements (in RT2013)

Reminder

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

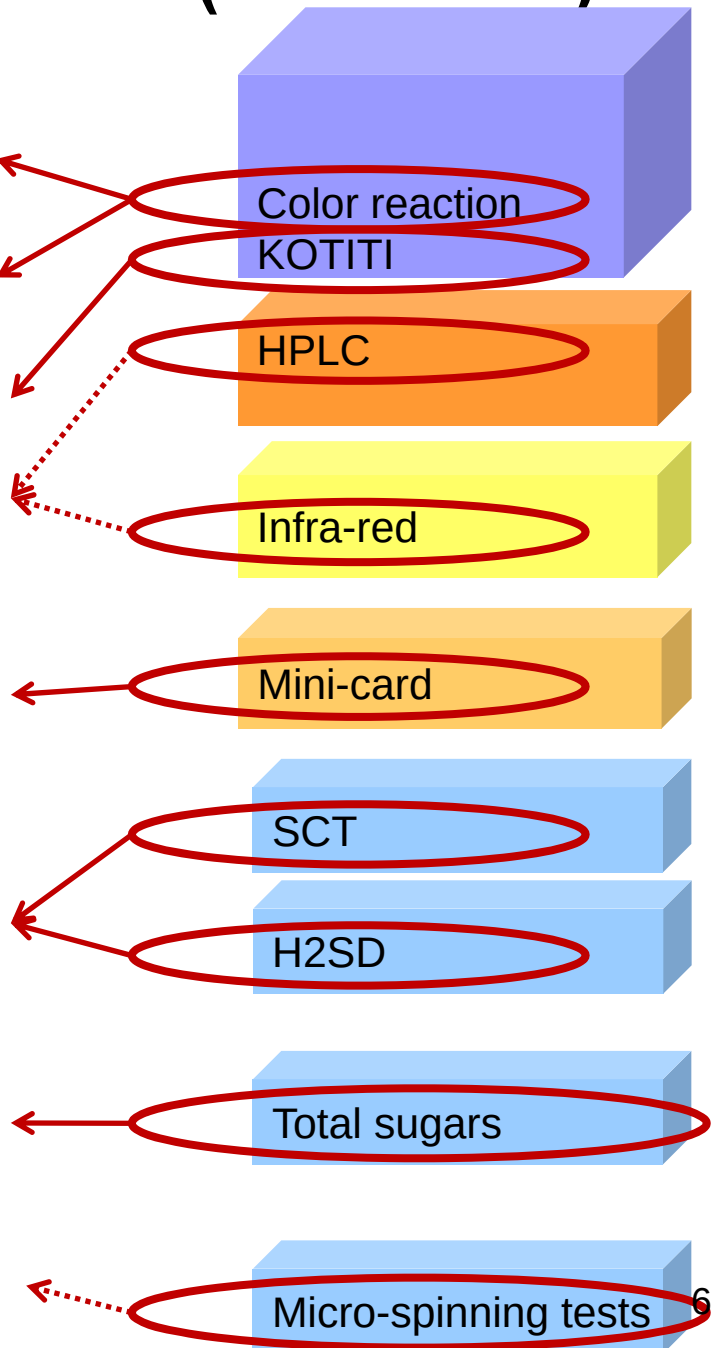
As reported by Perkins in 'ITMF grades'

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

Number of sticky points CEN_NF_14278-1&2 (2004)

Chemical extraction: mg/100g of fibers

Records of productivity and quality parameters
Not reported as still under data analysis



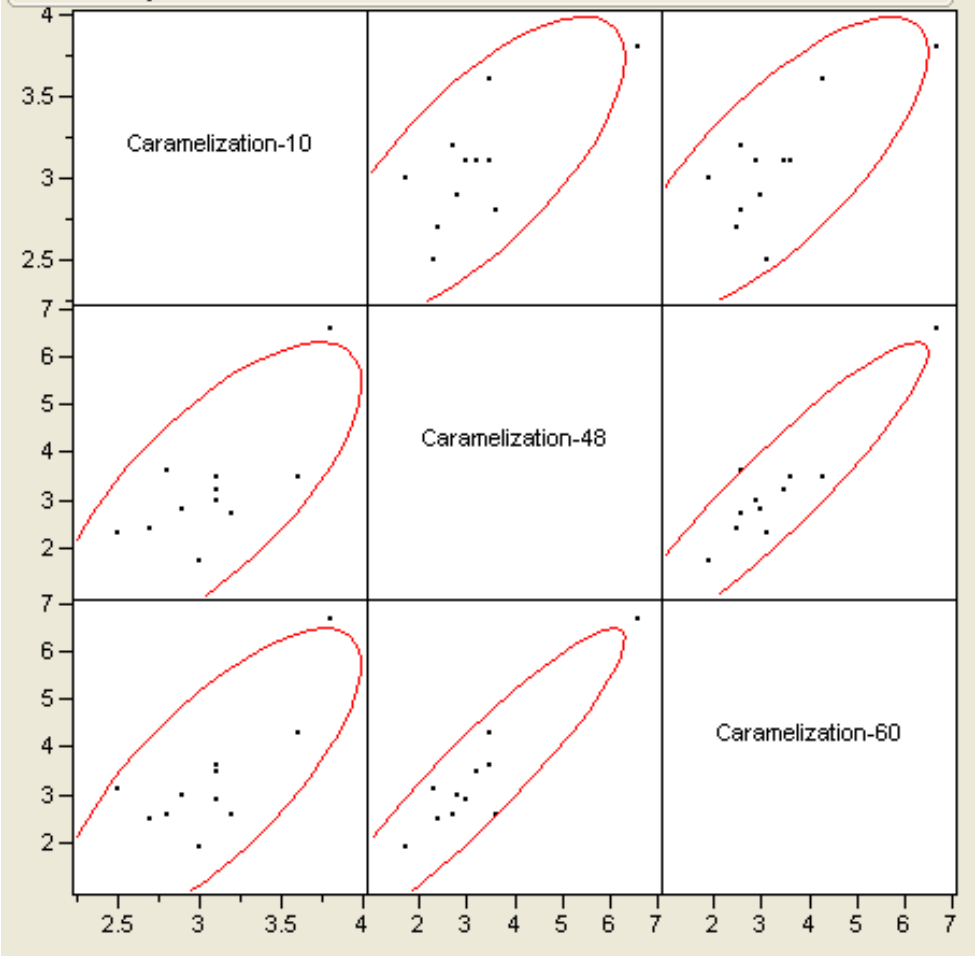
Within-technique, between laboratories

Reminder

Correlations

	Caramelization-10	Caramelization-48	Caramelization-60
Caramelization-10	1.0000	0.7279	0.7622
Caramelization-48	0.7279	1.0000	0.9289
Caramelization-60	0.7622	0.9289	1.0000

Scatterplot Matrix

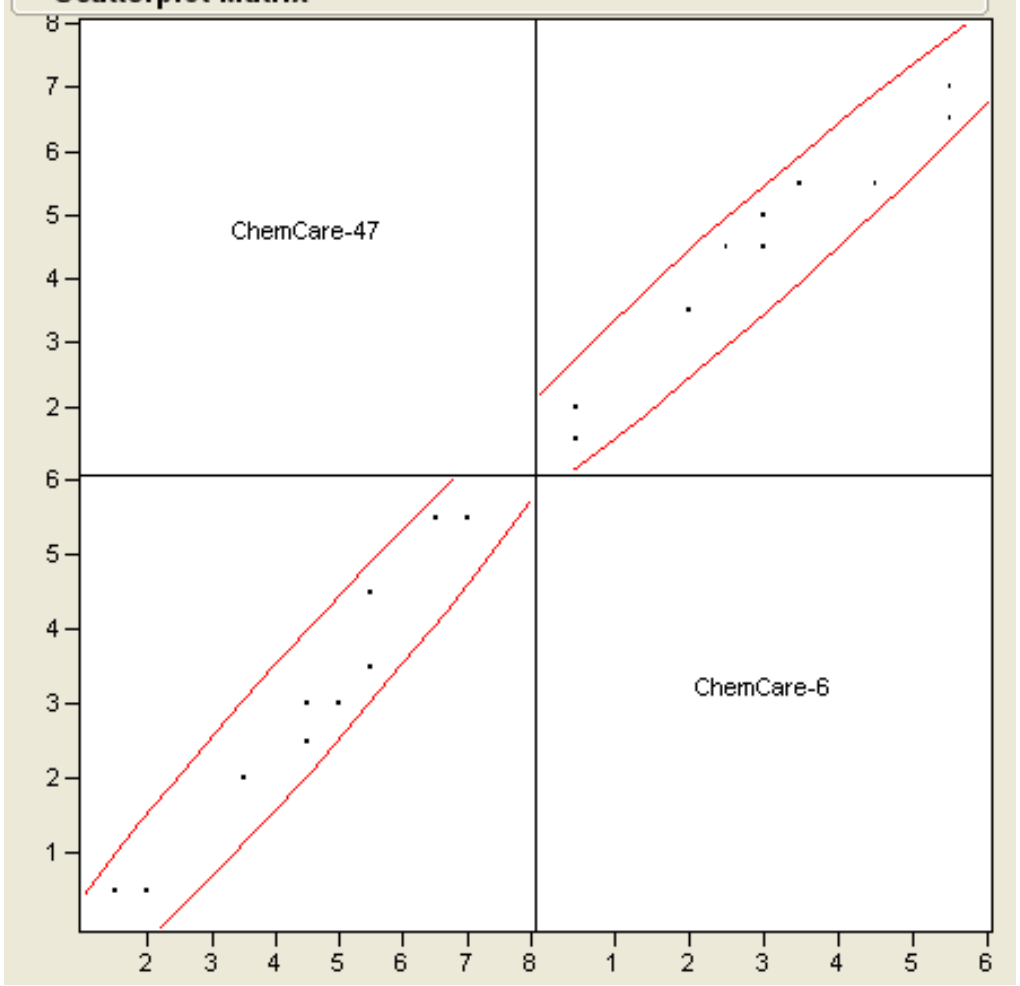


Within-technique, between laboratories

Reminder

Correlations

	ChemCare-47	ChemCare-6
ChemCare-47	1.0000	0.9766
ChemCare-6	0.9766	1.0000



Evaluation by one single person while the sample preparation was made by two independent laboratories

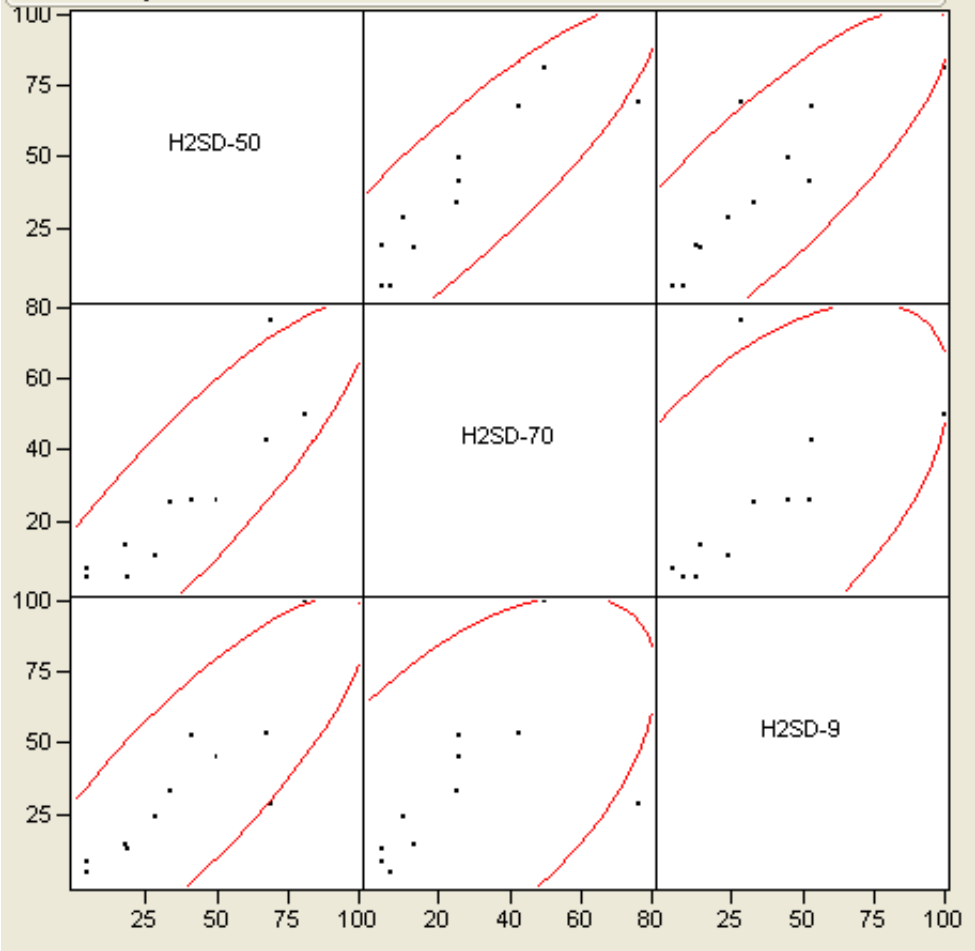
Within-technique, between laboratories

Reminder

Correlations

	H2SD-50	H2SD-70	H2SD-9
H2SD-50	1.0000	0.8870	0.8471
H2SD-70	0.8870	1.0000	0.5804
H2SD-9	0.8471	0.5804	1.0000

Scatterplot Matrix



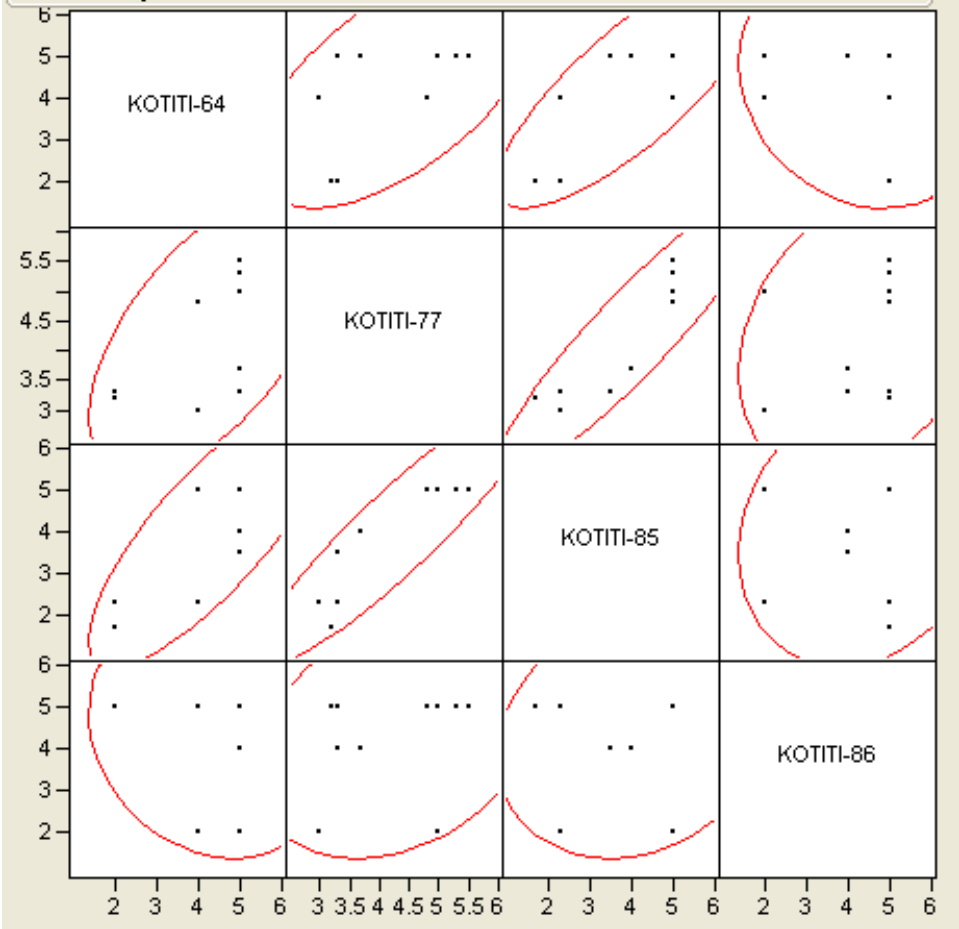
Within-technique, between laboratories

Reminder

Correlations

	KOTITI-64	KOTITI-77	KOTITI-85	KOTITI-86
KOTITI-64	1.0000	0.6018	0.8011	-0.1987
KOTITI-77	0.6018	1.0000	0.9191	0.2816
KOTITI-85	0.8011	0.9191	1.0000	0.1566
KOTITI-86	-0.1987	0.2816	0.1566	1.0000

Scatterplot Matrix



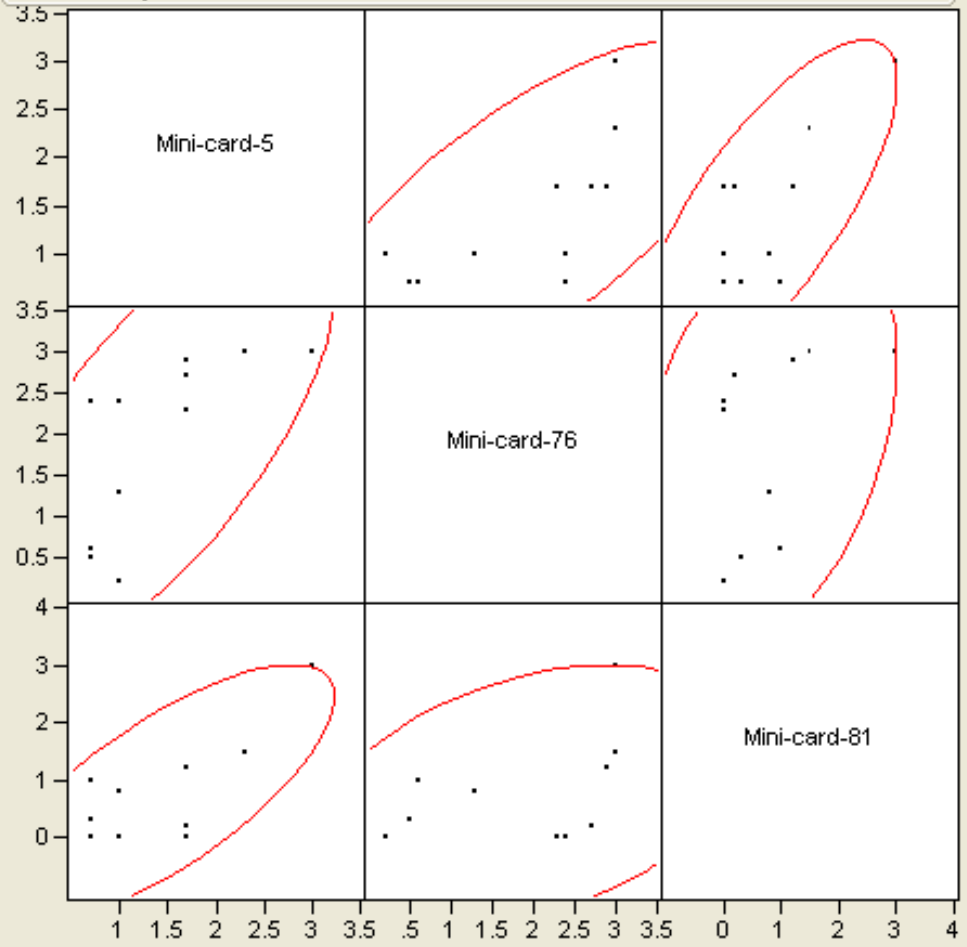
Within-technique, between laboratories

Reminder

Correlations

	Mini-card-5	Mini-card-76	Mini-card-81
Mini-card-5	1.0000	0.7036	0.7579
Mini-card-76	0.7036	1.0000	0.3814
Mini-card-81	0.7579	0.3814	1.0000

Scatterplot Matrix



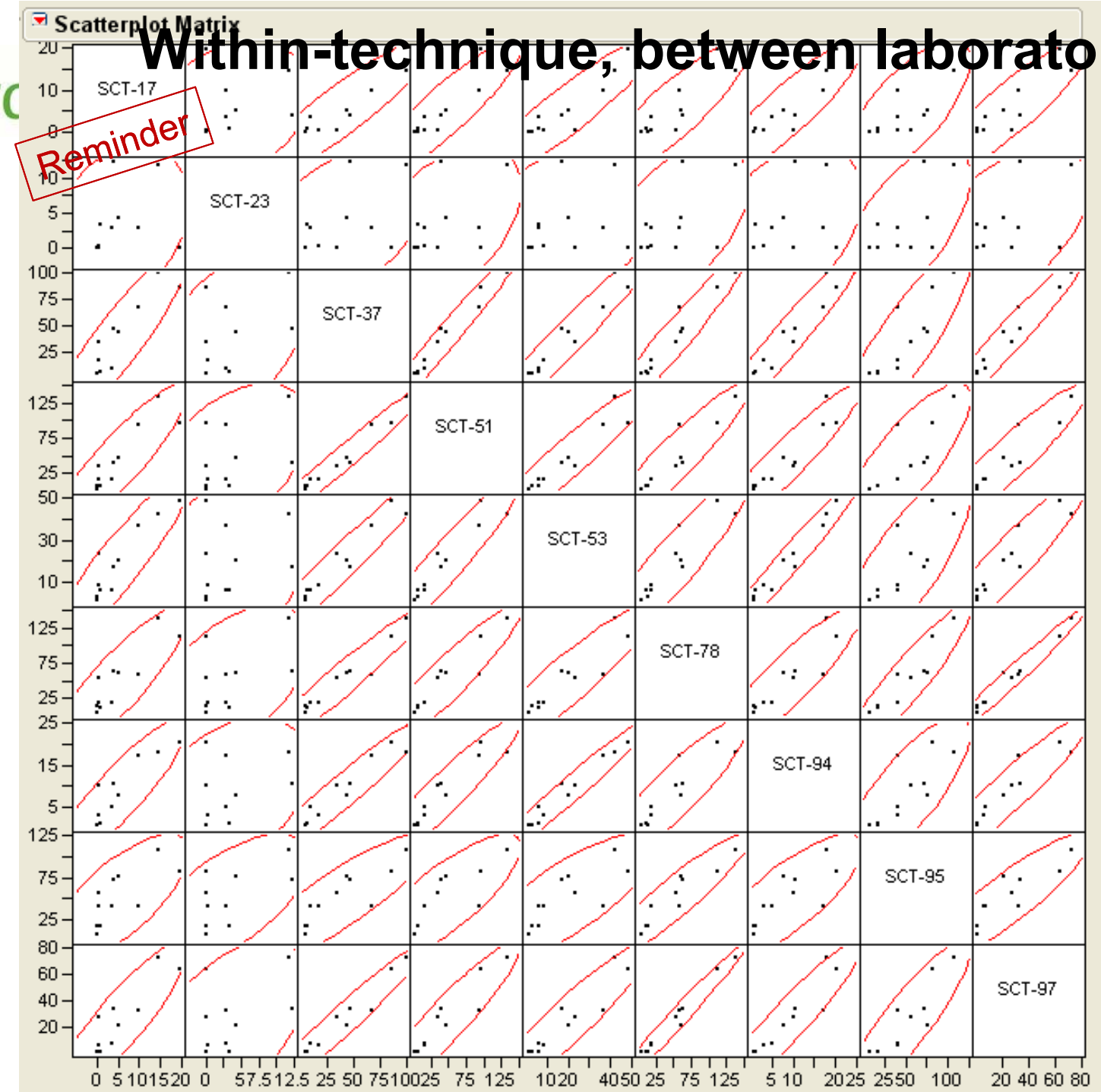
Within-technique, between laboratories

Reminder

Correlations

	SCT-17	SCT-23	SCT-37	SCT-51	SCT-53	SCT-78	SCT-94	SCT-95	SCT-97
SCT-17	1.0000	0.2812	0.9039	0.8978	0.9137	0.8713	0.8974	0.7195	0.8967
SCT-23	0.2812	1.0000	0.4816	0.4582	0.2735	0.5146	0.3663	0.6076	0.4890
SCT-37	0.9039	0.4816	1.0000	0.9804	0.9665	0.9664	0.9626	0.8571	0.9723
SCT-51	0.8978	0.4582	0.9804	1.0000	0.9479	0.9335	0.9397	0.8001	0.9369
SCT-53	0.9137	0.2735	0.9665	0.9479	1.0000	0.9207	0.9781	0.7761	0.9390
SCT-78	0.8713	0.5146	0.9664	0.9335	0.9207	1.0000	0.9064	0.9253	0.9871
SCT-94	0.8974	0.3663	0.9626	0.9397	0.9781	0.9064	1.0000	0.7904	0.9414
SCT-95	0.7195	0.6076	0.8571	0.8001	0.7761	0.9253	0.7904	1.0000	0.8879
SCT-97	0.8967	0.4890	0.9723	0.9369	0.9390	0.9871	0.9414	0.8879	1.0000

Within-technique, between laboratories



Reminder

Reminder

Participating techniques and laboratories

	Number of laboratories per stickiness technique
Caramelization	3
Chemcare	2
H2SD	3
KOTITI	4
Mini-card	3
SCT	9
Total sugar	1
Total Nb	**25**

Conclusions … before discussions

Reminder

- 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

Reminder

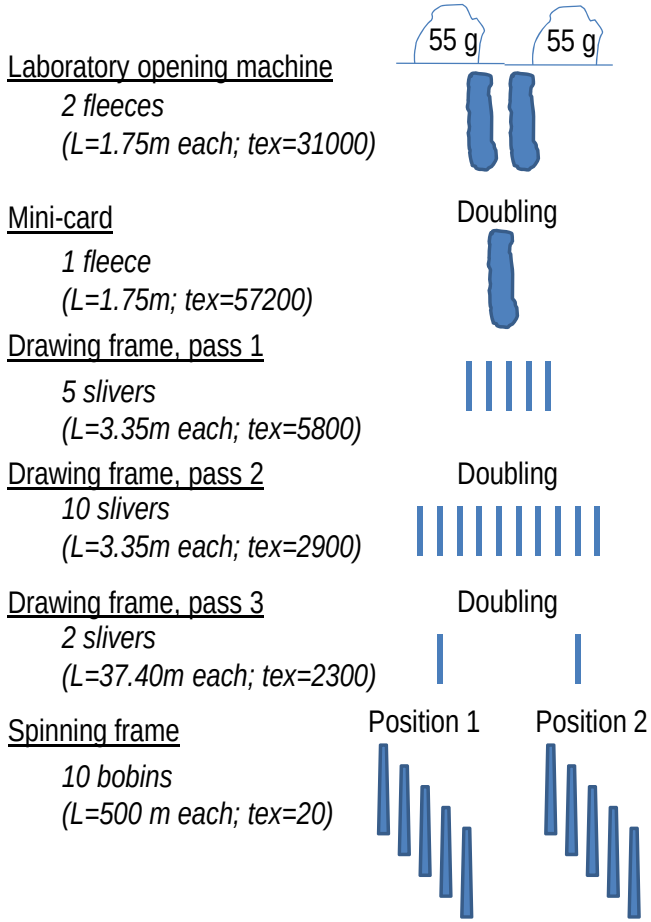
- Need for a harmonization
- Which are the ways to achieve this harmonization?
- What to recommend?
- Requires policies and support tools to continue

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

Spinning protocole

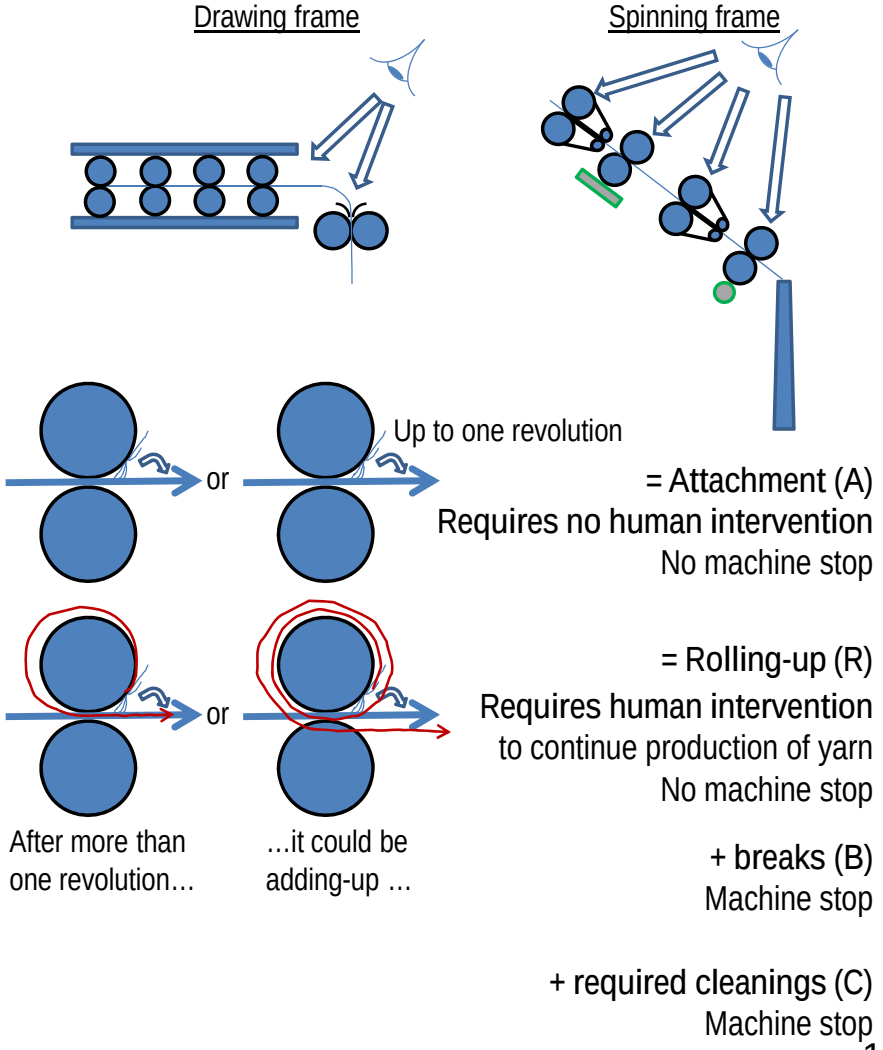
Operating method used for producing yarn
in a micro ring-spinning facility
ITMF-ICCTM inter-laboratory round test on stickiness, 2014



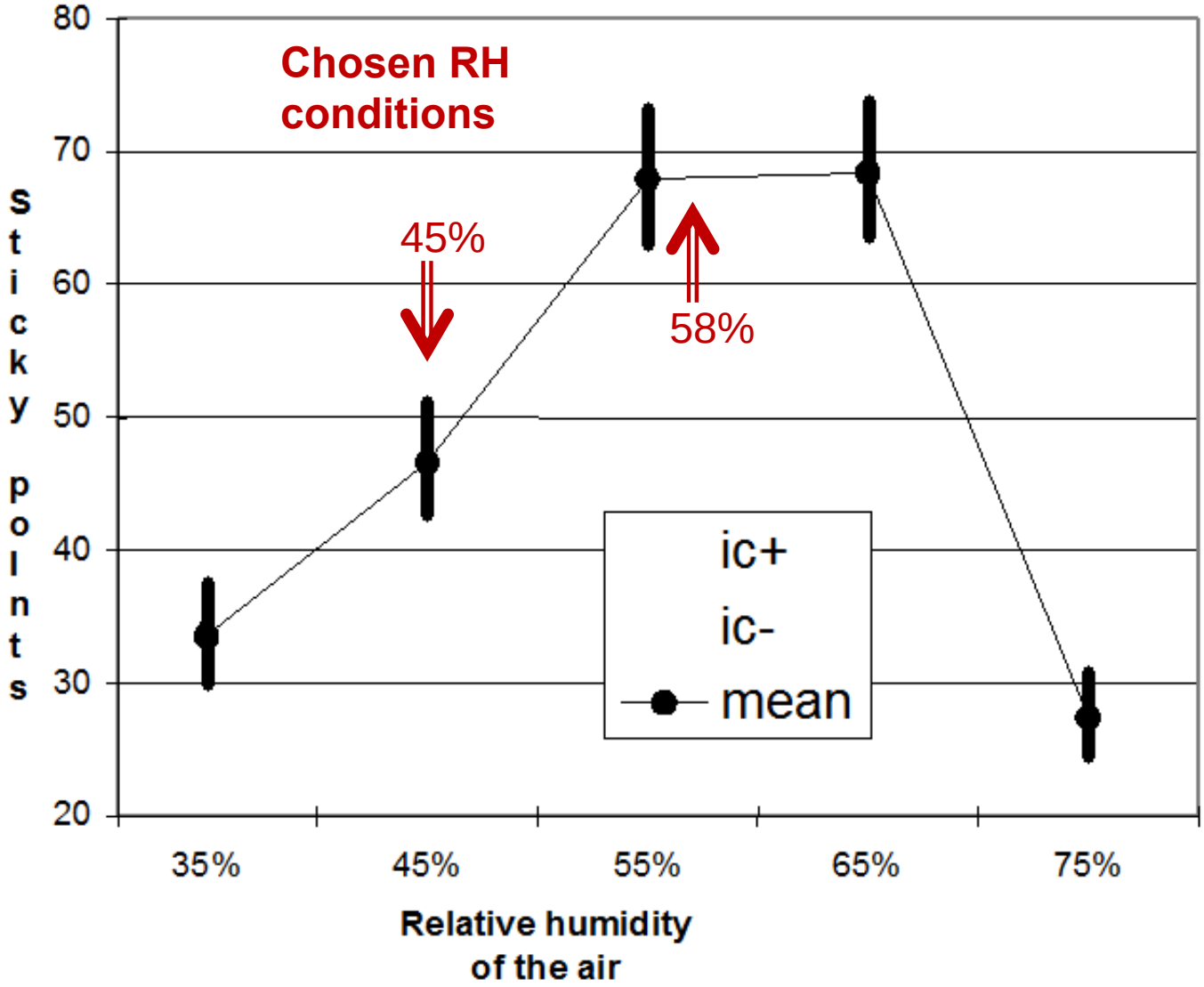
Eveness Tester: 100 m * 2.5 min / bobin
Strength Tester: 100 breaks / bobin @ 0.3 second per break

11 cottons * 2 RH * 2 blocks

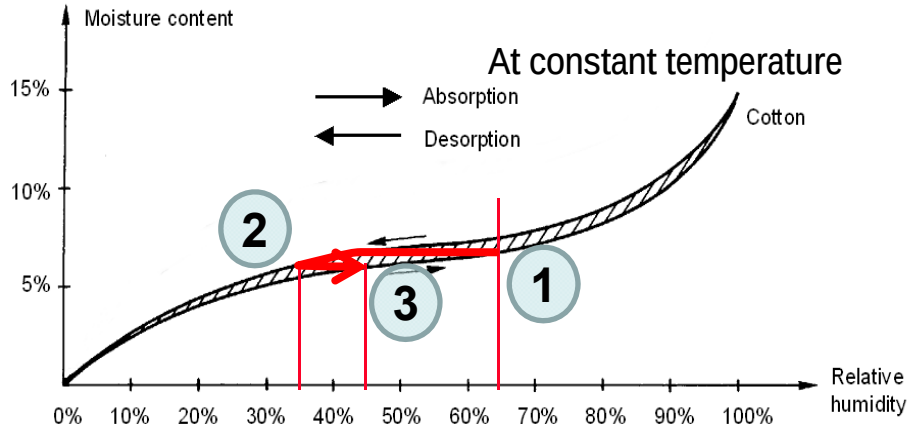
Observations made during spinning
on the ring spinning frame
ITMF-ICCTM inter-laboratory round test on stickiness, 2014



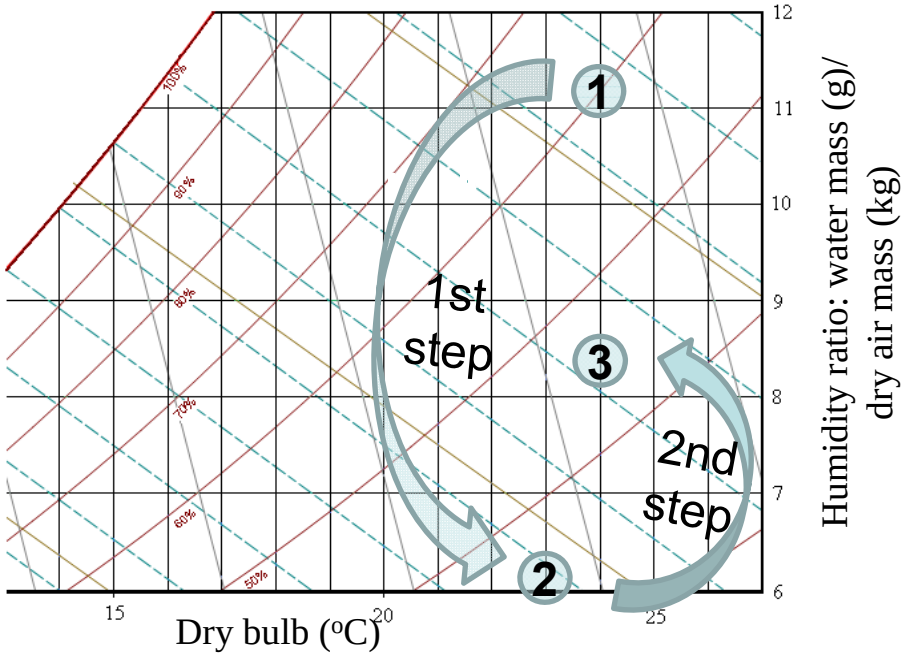
Spinning conditions



Source: Frydrych R., 2003, Habilitation à Diriger des Recherches « Les polluants du coton : cas du collage et des débris de coque », Université de Haute Alsace, 202 p.



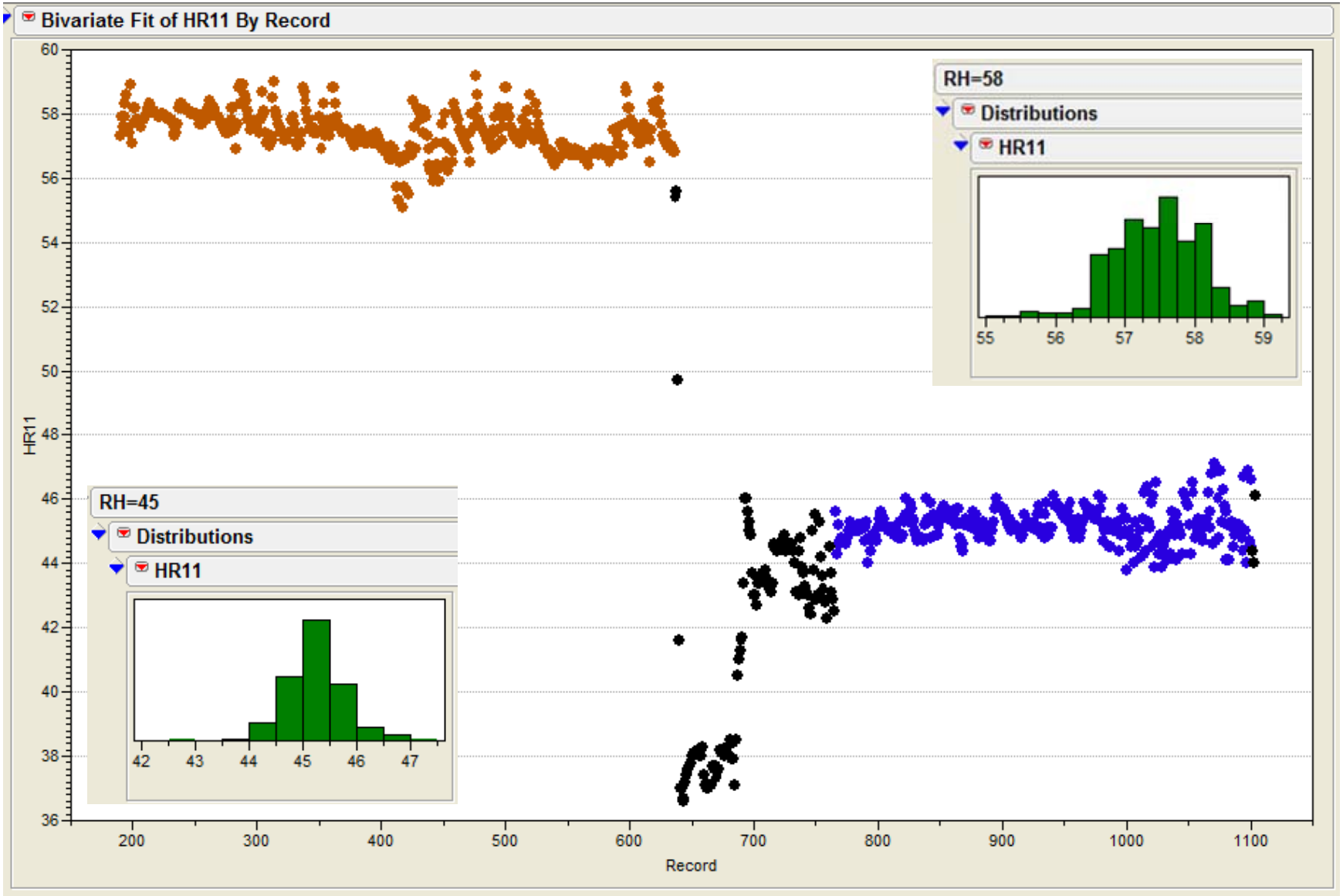
1. **Spinning tests: first RH conditions**
2. **Drying period for cottons (72 hours)**
3. **Spinning tests: second RH conditions**



Nominal values

	T°C	RH%	g H_2O/g Gaz
1	24	60	11.18
2	23	35	6.09
3	24	45	8.35

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
- Neps140, 200, 280, 400
- Fmax, CVFmax, Ten, WorkMax, N/texM1, N/texM2, All, CVAll

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)

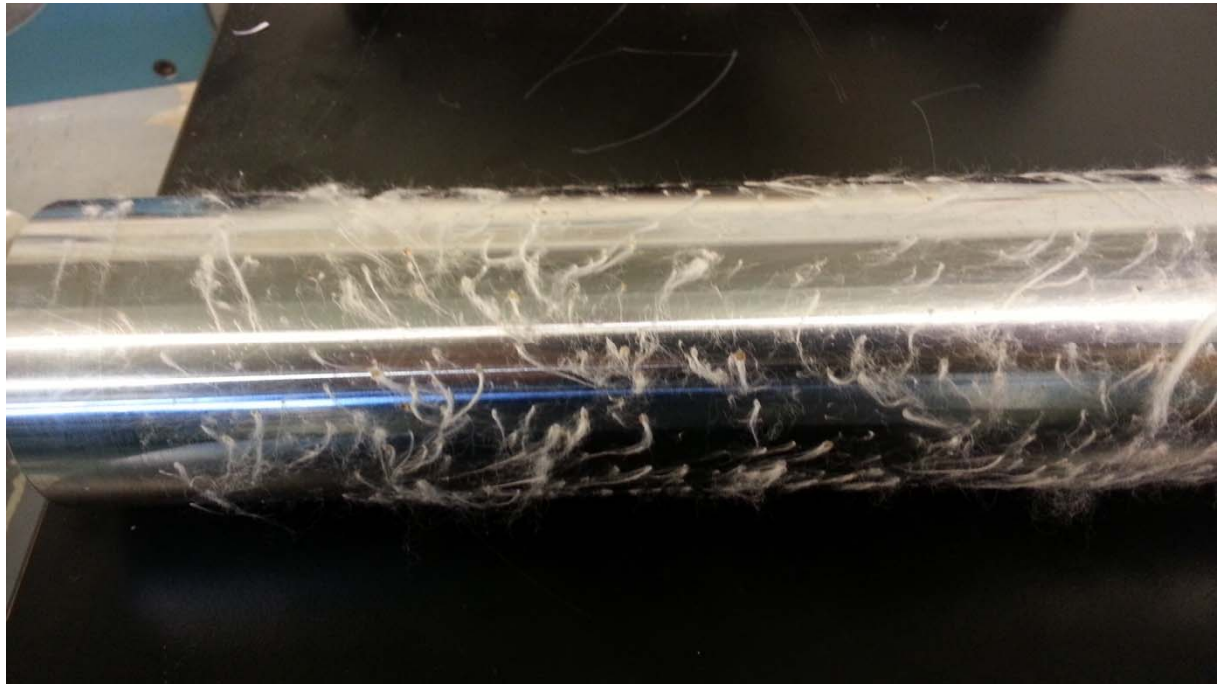
	Number of Tech LabID	Mean	CF	Ranks*	SO	PCA	Total
Caramelization	3	1	3	3	3	1	14
Chemcare	2	1	2	2	2	1	10
H2SD	3	1	3	3	3	1	14
KOTITI	4	1	4	4	4	1	18
Mini-card	3	1	3	3	3	1	14
SCT	9	1	9	9	9	1	38
Total sugar	1						1
Total Nb	**25**	**6 +6 HPLC**	**24**	**24 +6 HPLC**	**24**	**6**	**121**

*: Partly studied and shown in 2014

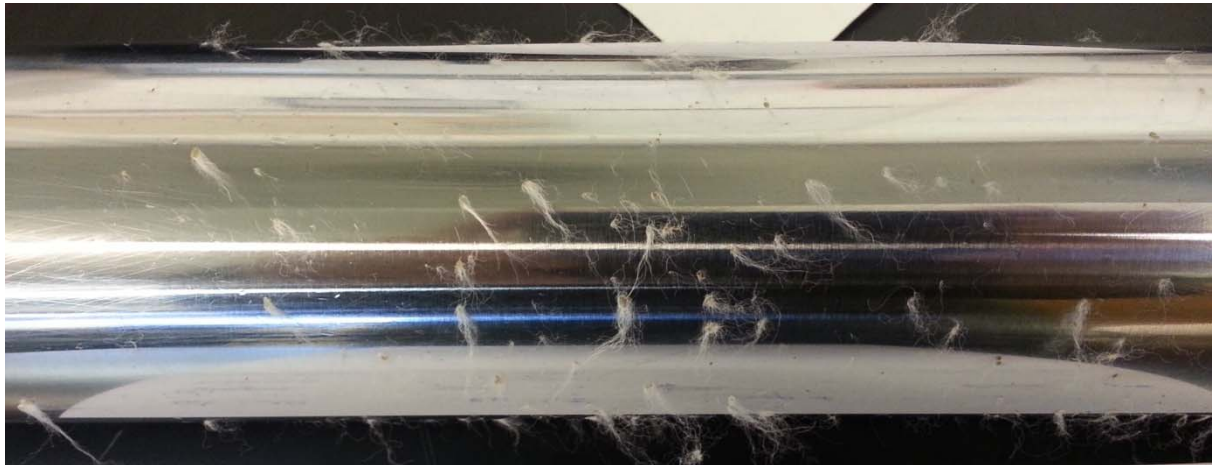
Strange #10 cotton…

(Not taken into account from now on)

RH: 58%



RH: 45%

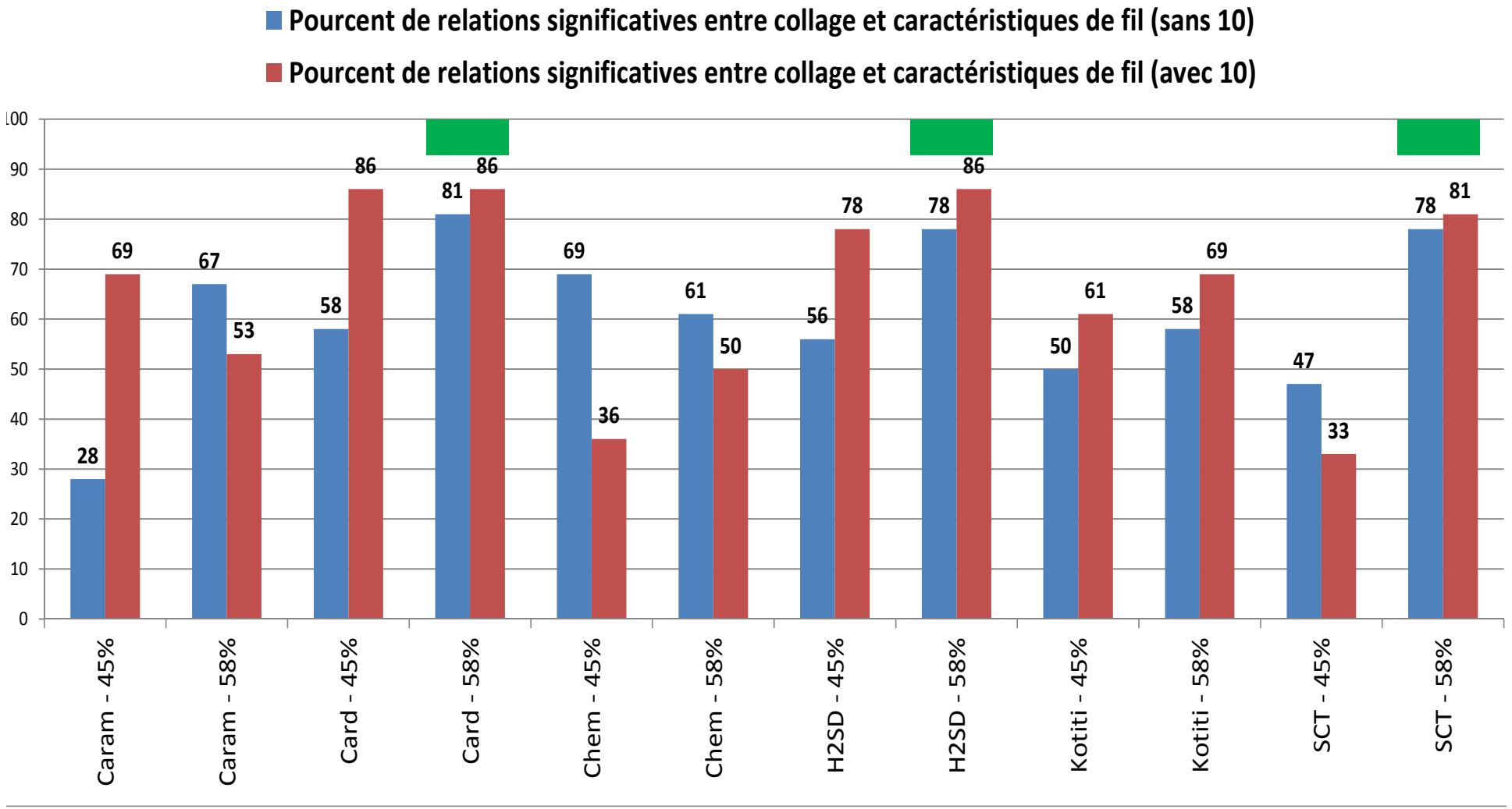


Percent of significant relationships (α=5%) Yarn = f (Fiber)

Without cotton #10	Caram - 45%	Card - 45%	Chem - 45%	H2SD - 45%	Kotiti - 45%	SCT - 45%	Caram - 58%	Card - 58%	Chem - 58%	H2SD - 58%	Kotiti - 58%	SCT - 58%
Productivity (max=8)	0	1	4	1	1	1	6	7	5	6	2	6
Quality (max=28)	10	20	21	19	17	16	18	22	17	22	19	22
Nb significant	**10**	**21**	**25**	**20**	**18**	**17**	**24**	**29**	**22**	**28**	**21**	**28**
Nb total	**36**	**36**	**36**	**36**	**36**	**36**	**36**	**36**	**36**	**36**	**36**	**36**
Percent of significant relationships between fiber and yarn parameters	**28**	**58**	**69**	**56**	**50**	**47**	**67**	**81**	**61**	**78**	**58**	**78**

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



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- 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. Gérardeaux

- 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

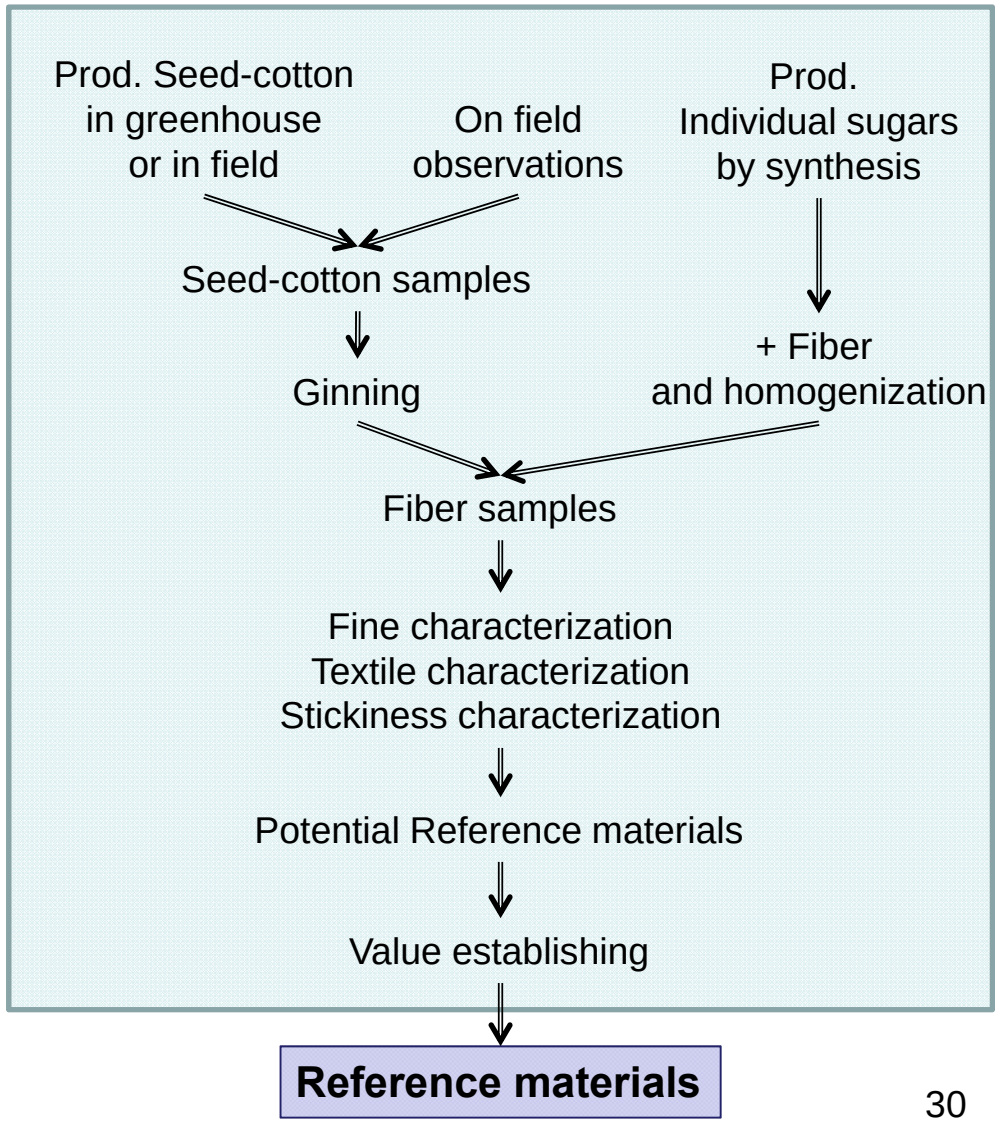
What's next concerning any harmonization process?

a path toward a joint project…

CIRAD UPR115 / Gesc
Laboratoire de Technologie et de Caractérisation
des fibres naturelles

cirad

Reference materials



Results / information
Materials
Interpretation / management

Bibliography

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

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

Prod. Seed-cotton in greenhouse or in field

On field observations

Prod. Individual sugars by synthesis

Seed-cotton samples

Ginning

+ Fiber and homogenization

Fiber samples

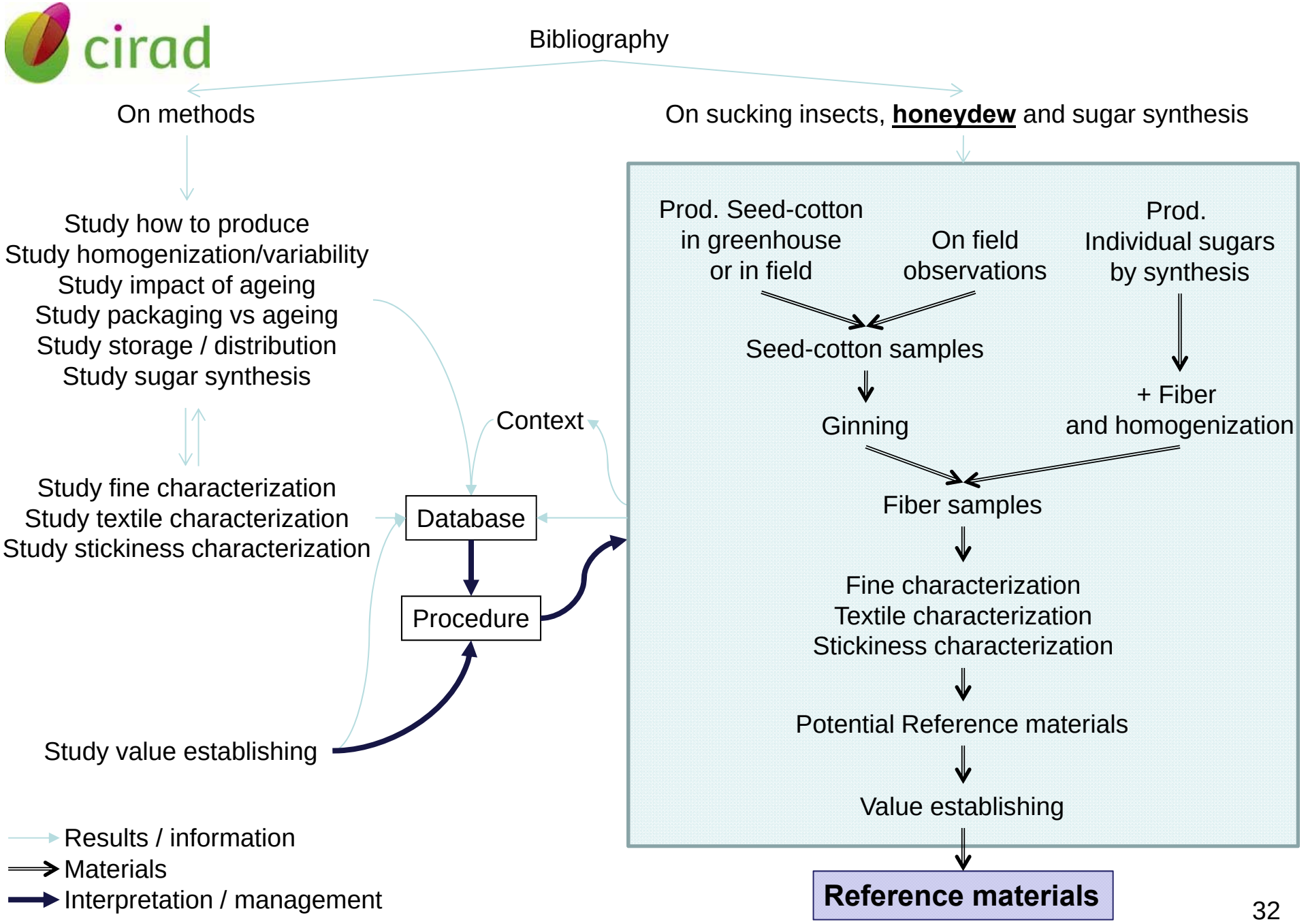
Fine characterization
Textile characterization
Stickiness characterization

Potential Reference materials

Value establishing

Reference materials

Results / information
Materials
Interpretation / management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Study textile characterization
Study stickiness characterization
Context
Database
Procedure
Study value establishing
Prod. Seed-cotton in greenhouse or in field
On field observations
Prod. Individual sugars by synthesis
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Ginning
+ Fiber and homogenization
Fiber samples
Fine characterization
Textile characterization
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Potential Reference materials
Value establishing
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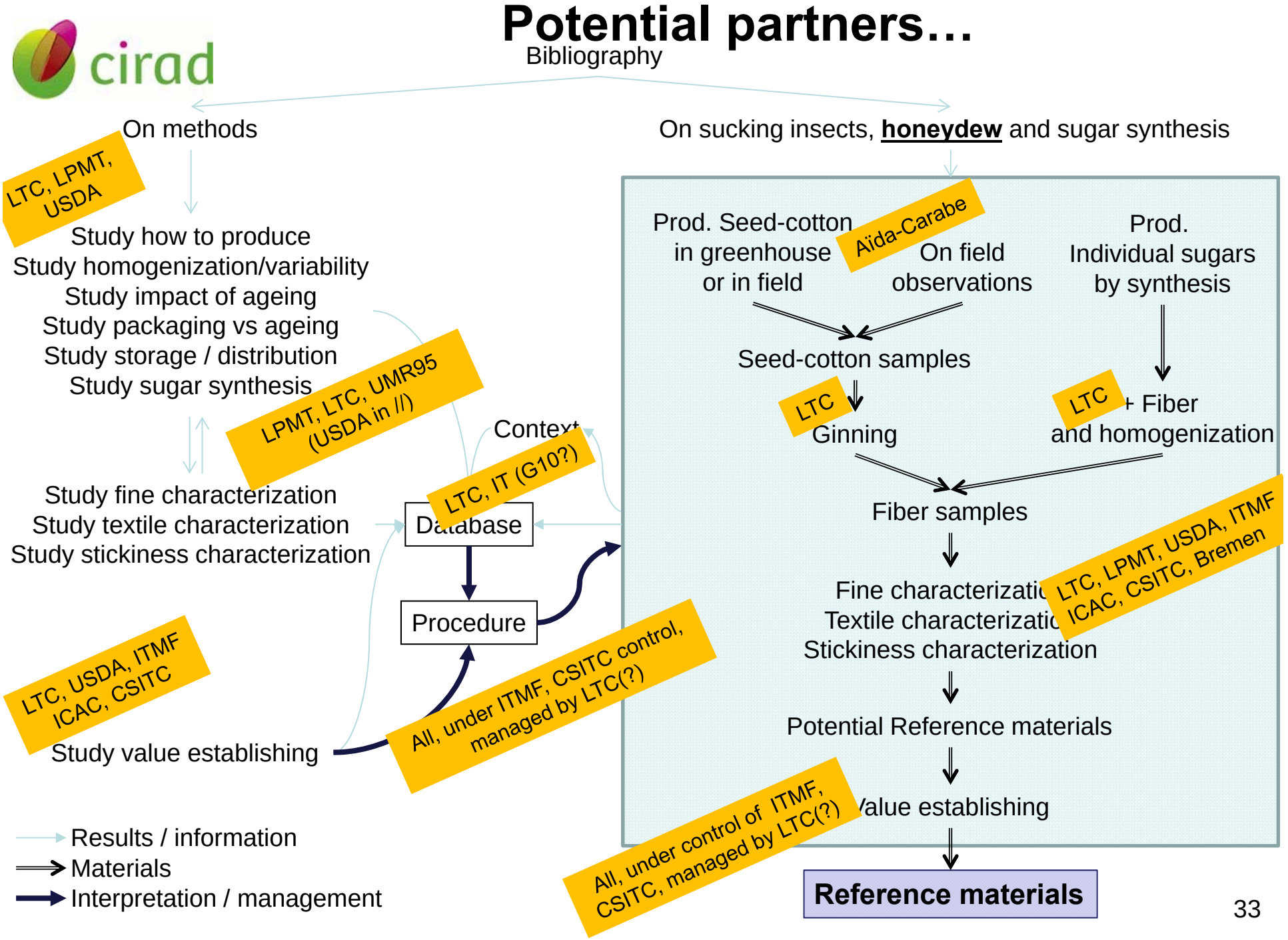
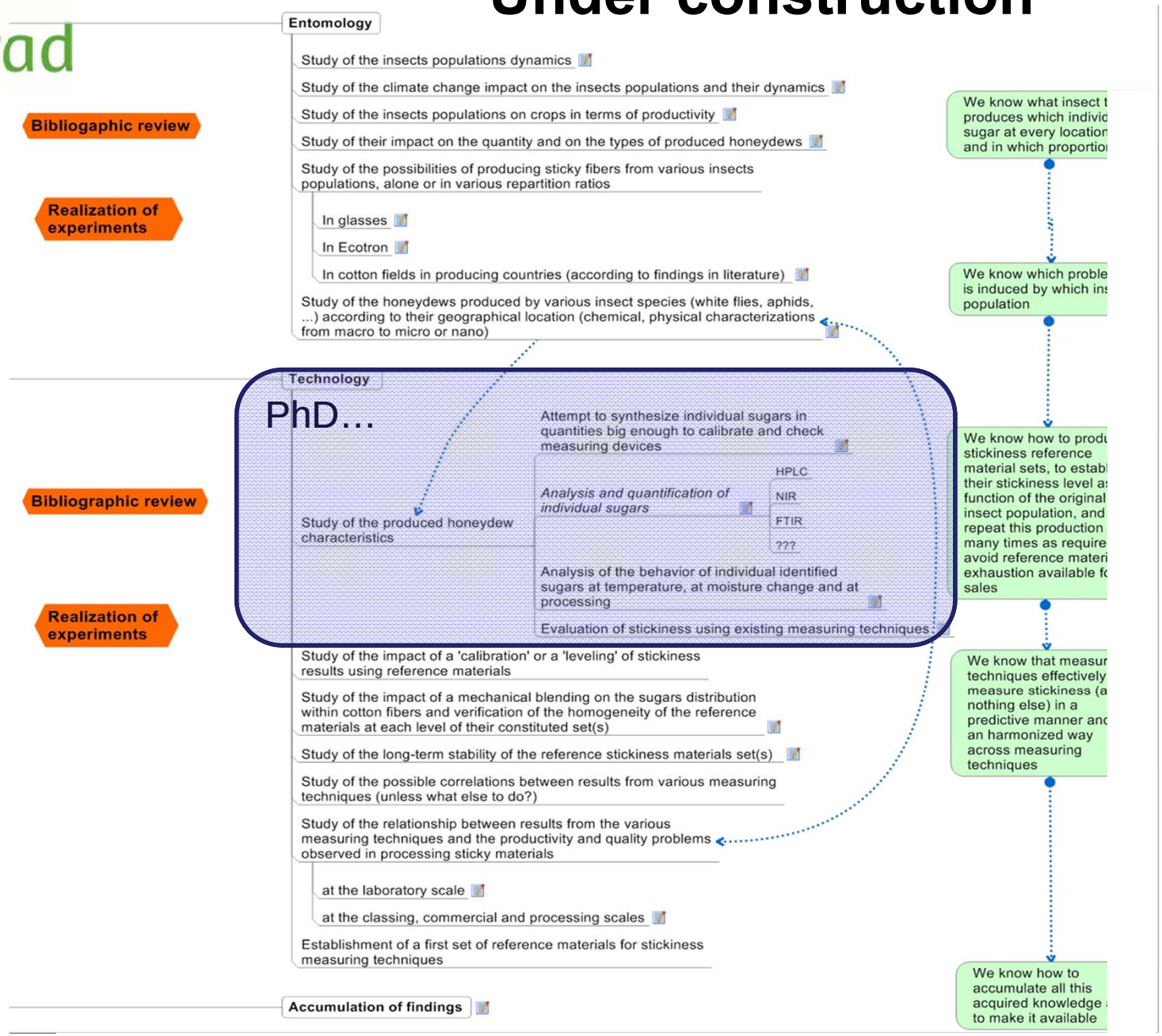
Potential partners…



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/collect 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/collecting 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 characterizations 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 honewdew 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 characterizations 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 characterizion results
  - 5.7 Update an operating method on the way to produce, prepare, caracterize, 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

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  - 4.3 Study homogenizing methods/techniques to insure a high uniformity of honewdew 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)

PhD…

- 5 Establish a first reference materials set of sticky fibers
  - 5.1 Apply an homogenization if required
  - 5.2 Realize fine characterizations 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 characterizion results
  - 5.7 Update an operating method on the way to produce, prepare, caracterize, 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

Thank you

for

your attention

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des fibres naturelles