What did we learn from the eight first occurrences of the ITMF-ICCTM international round-trials comparing stickiness measuring methods (2017-2020)?

Jean-Paul GOURLOT
Axel DRIELING
Karsten FROESE
Serge LASSUS

Periodic international inter-laboratories round-tests on stickiness

- RTStick initiated in 2017
  - Planned for 2 times / year
  - Possibility of having up to 6 measurements / cotton
  - 2017-1 and 2017-2 with 3 cottons
  - 2018-1 and followings with 5 cottons
  - Anonym participation (one LabID* / test)
- Cottons covering a range per test (at least expected)
- Organized and funded by CIRAD, FIBRE and BBB
- Methods
  - No micro-spinning
  - Used in laboratories

*: one instrument of one method in one laboratory

Technical objectives of RTStick

Measure within-methods variations

Measure between-instruments within-methods variations

Criteria

- Evolution of variances over time
  - Within-lab / cotton / method / test
  - Between-lab / cotton / method / test (CommonScale included since 2018-1)
- Based on raw and CommonScale(Max)

Participation in 8 ITMF-ICCTM RTs

[RT 2017-1 - RT2020-2]

Methods	2017-1	2017-2	2018-1	2018-2	2019-1	2019-2	2020-1	2020-2
Benedict	1	-	-	-	-	-	-	-
Caramelization	4	4	5	4	5	4	3	3
Clinitest	1	1	1	1	1	1	-	-
Contest-S	5	6	5	6	4	3	3	4
GB/T13785-1992	-	1	1	1	-	-	-	-
H2SD	4	3	4	4	4	2	3	4
HSI-NIR	-	1	1	1	1	1	1	-
KOTITI	1	1	1	1	1	1	1	1
Minicard	4	4	3	2	3	3	1	1
Qualitative meth.	-	-	1	1	1	1	1	1
Quantitative meth.	1	1	1	1	1	1	2	1
Reactive spray/heat	1	1	-	1	1	1	-	-
SCT	13	11	12	9	9	11	7	6
TDM-A	-	-	1	1	-	-	-	-
Total	**35**	**34**	**36**	**33**	**31**	**29**	**22**	**21**

Participation in 8 ITMF-ICCTM RTs

[RT 2017-1 - RT2019-2]

Method	Sample	Principle	Observation	Unit
Benedict	Washed sample	Benedict reagent	Grading	Grade
Caramelization	Fiber sample	'+b', oven, '+b'	Colorimeter	Color degree
Clinitest	Washed sample	Reducing sugars, medical test	Colormeter	Color chart
CommonScale		Proportion to MaxEver	Proportion	Percent
Contest-Fibermap	Fiber web	Drum + tangent laser	Counting	Grade
H2SD	Fiber pad	Hot and cold pressure, cleaning	Counting	Sticky points
HSI-NIR	Fiber web	Calibrated to H2SD	Counting	Percent
KOTITI	Fiber web	Reactive agent	Grading	Kotiti grade

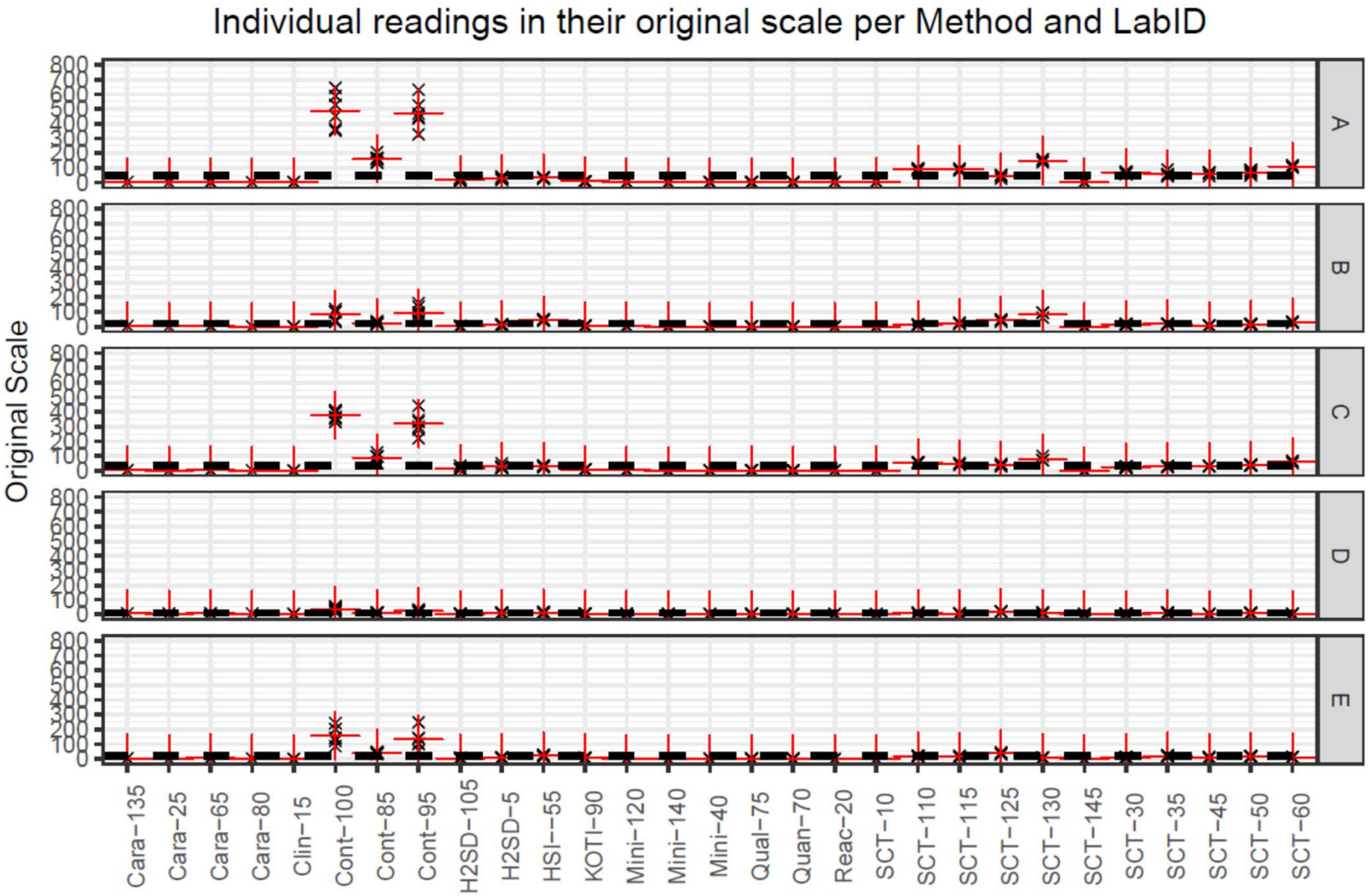
.../...

Participation in 8 ITMF-ICCTM RTs

[RT 2017-1 - RT2019-2]

Method	Sample	Principle	Observation	Unit
Minicard	Fiber web	Sample carding	Grading	ITMF grade
Qualitative method	Washed sample	Fiber+reagent => boiling	Grading	Grade
Quantitative method	Washed sample	Fehling solution	Spectrophoto-colorimeter	Percent
Reactive Spray	Washed sample	Clinitest type test + heat	Grading	Spray grade
SCT	Fiber web	Hot and cold pressure, cleaning	Counting	Sticky points
TDM-A	Fiber web	Hot and cold pressure, cleaning	Grading	Sticky grade

RT2019-2 report



RT2019-2 report

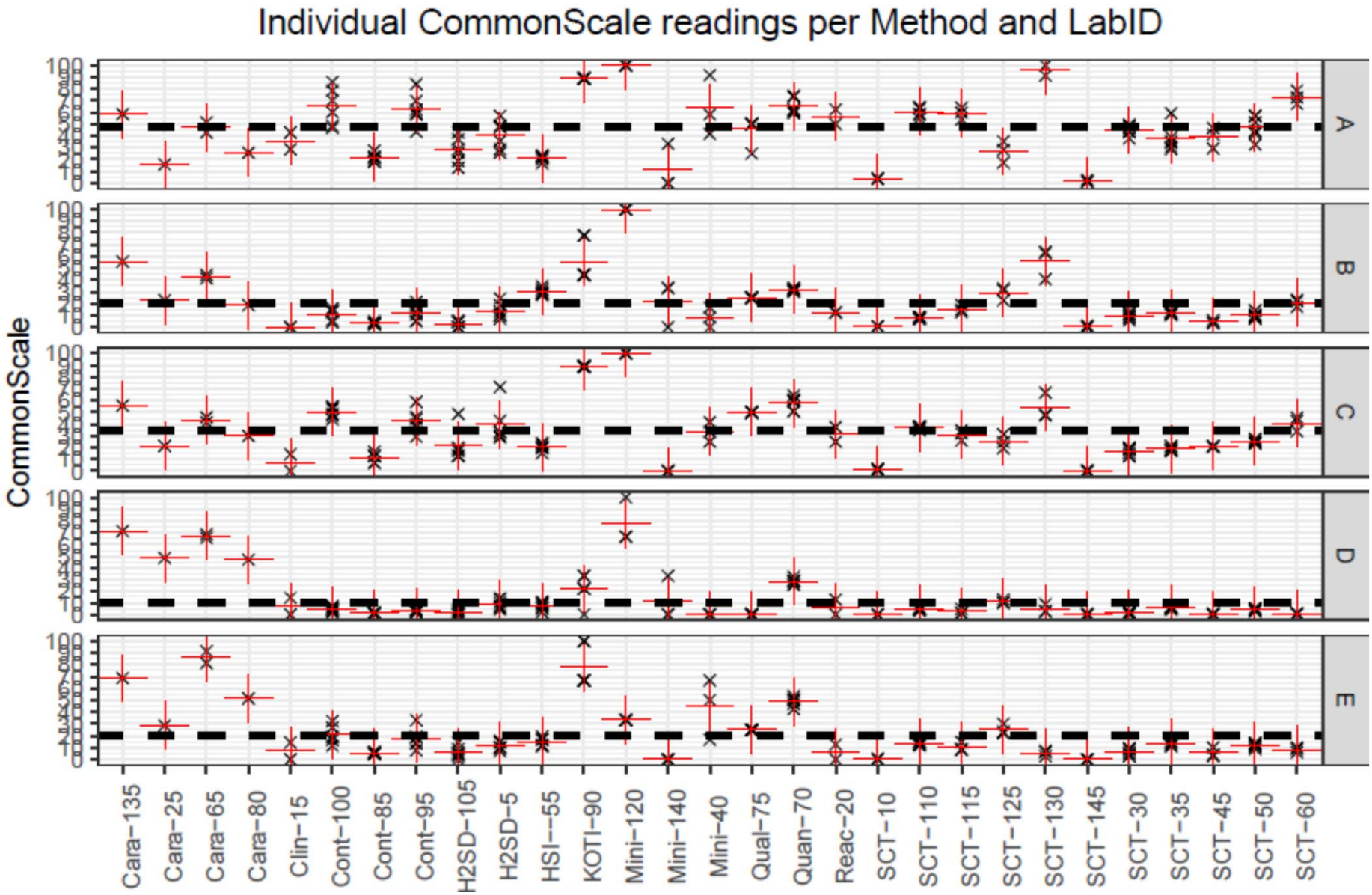
- CommonScale Approach
  - Aim: Ease the comparison between methods

=>CommonScale(Max) has been developed as

$$\frac{\text{Measured Stickiness Raw" (MSR)} * 100}{\text{MaxEver}} = \text{Measured Stickiness Scaled (MSS)}$$

Methods	Unit	MaxEver
Minicard	ITMF grade	3
SCT	Sticky points	150
H2SD	Sticky points	70
Contest-S	Grade	750

RT2019-2 report

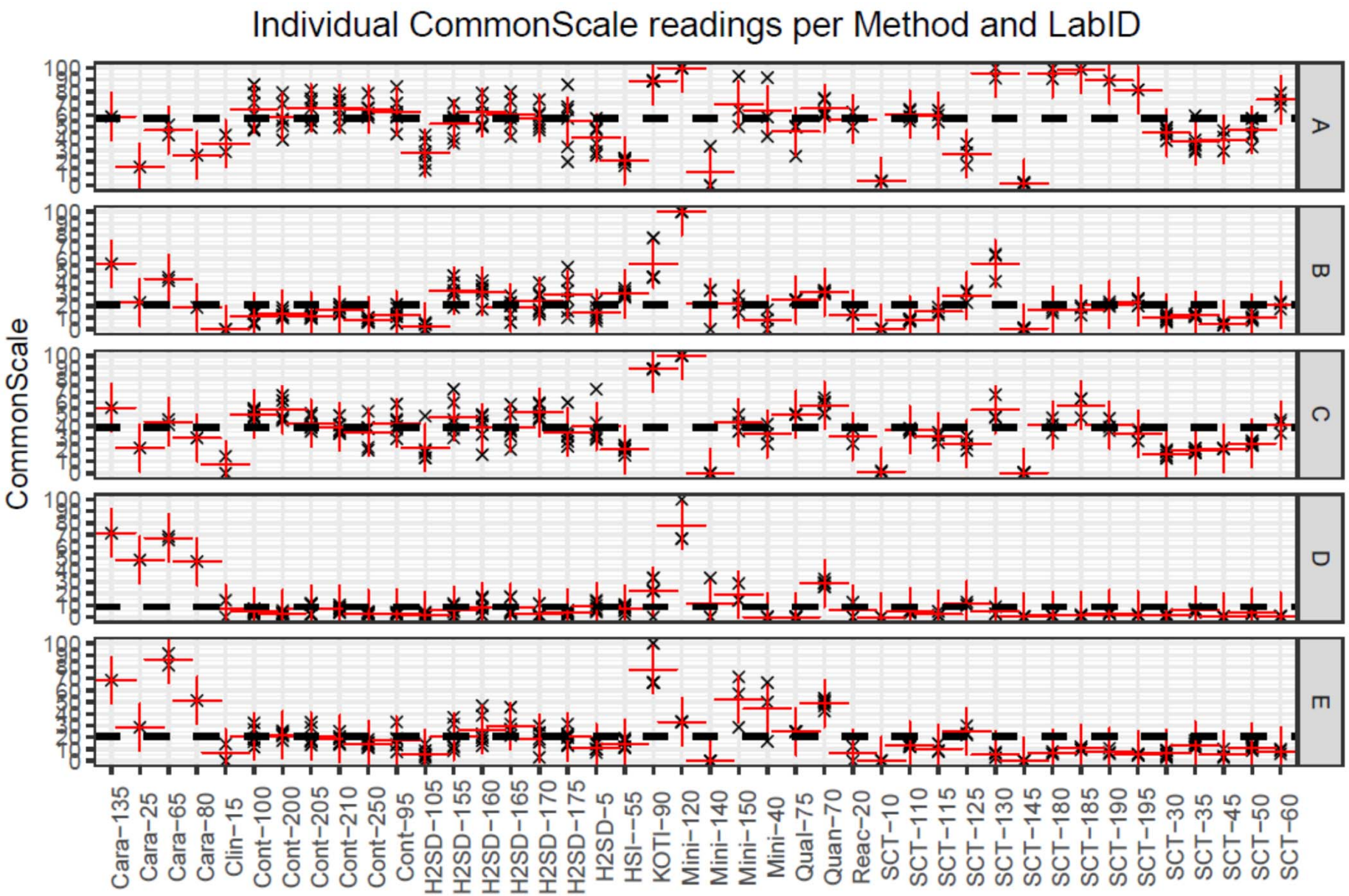


Participation in 8 ITMF-ICCTM RTs with variab.

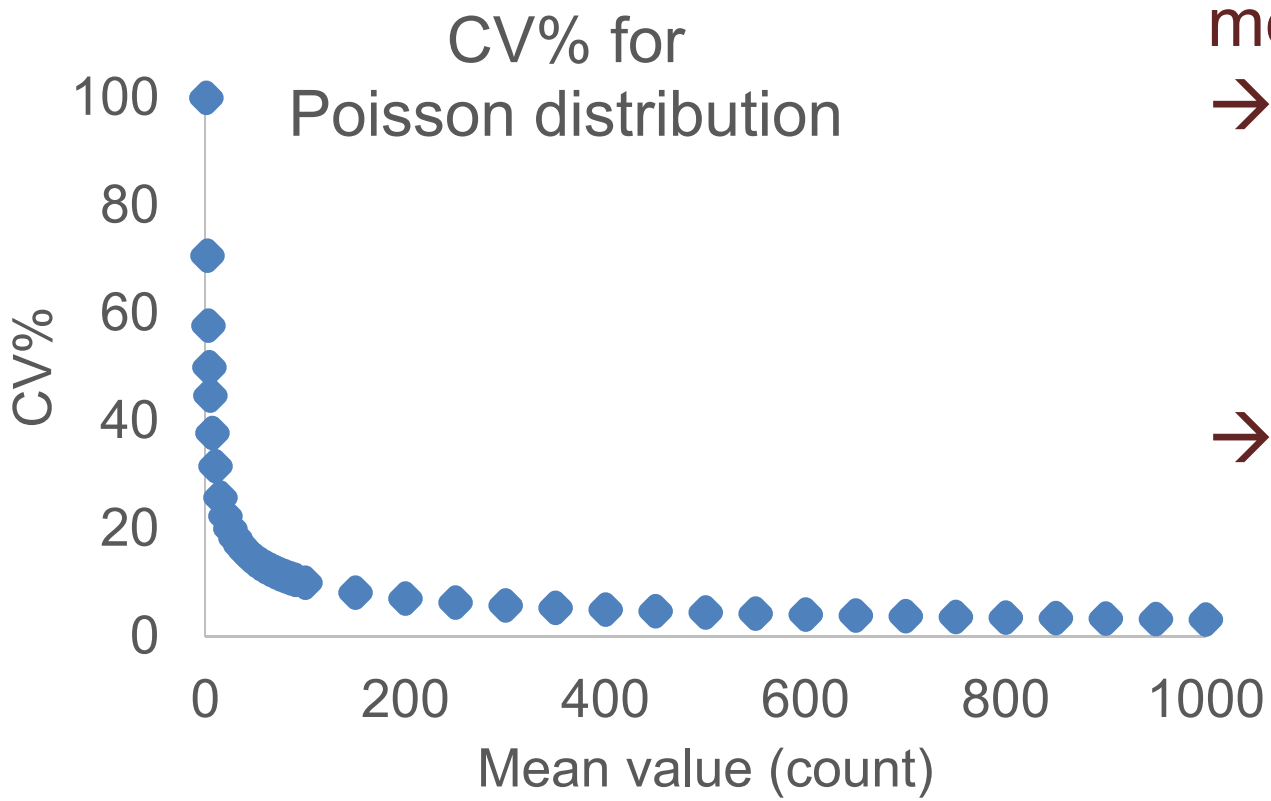
[RT 2017-1 - RT2020-2]

Methods	2017-1	2017-2	2018-1	2018-2	2019-1	2019-2	2020-1	2020-2
Benedict	1	-	-	-	-	-	-	-
Caramelization	4	4	5	4	5	4	3	3
Clinitest	1	1	1	1	1	1	-	-
Contest-S	5	6	5 (+1*)	6 (+4*)	4 (+3*)	3 (+3*)	3 (+4*)	3 (+4*)
GB/T13785-1992	-	1	1	1	-	-	-	-
H2SD	4	3	4 (+5*)	4 (+4*)	4 (+5*)	2 (+5*)	2 (+5*)	4 (+4*)
HSI-NIR	-	1	1	1	1	1	1	1
KOTITI	1	1	1	1	1	1	1	1
Minicard	4	4	3	4	3	3	1	1
Qualitative meth.	-	-	1	1	1	1	1	1
Quantitative meth.	1	1	1	1	1	1	2	1
Reactive spray/heat	1	1	-	1	1	1	-	-
SCT	13	11	12 (+4*)	9 (+6*)	9 (+4*)	11 (+4*)	7 (+4*)	6 (+4*)
TDM-A	-	-	1	1	-	-	-	-
Total	**35**	**34**	**36 (+10*)**	**33 (+16)**	**31 (+12)**	**29 (+12*)**	**22 (+13*)**	**21 (+12*)**
Total	**35**	**34**	**46**	**49**	**43**	**41**	**35**	**33**

RT2019-2 report (including variab. study)



Problem of CV% for comparing methods for raw counts (valid for CS?)



CV% changes with mean value

→ CV% not a good indicator for a fair comparison of methods

→ Need to look for better indicator

Evolution of variances [RT 2017-1 - RT2020-2]
Within-method and Between-LabIDs

Means of the CVs% of raw data* (all labs/method, all cottons)

Methods	2017-1	2017-2	2018-1	2018-2	2019-1	2019-2	2020-1	2020-2
Benedict	0	-	-	-	-	-	-	-
Caramelization	12	8	16	11	13	9	5	14
Clinitest	0	0	8	16	14	71	-	-
Contest-Fibermap	13	15	17	14	23	22	25	20
GB/T13785-1992	-	28	26	21	-	-	-	-
H2SD	18	28	28	27	25	40	30	24
HSI-NIR	-	12	10	19	9	16	20	9
KOTITI	0	16	4	5	3	14	6	4
Minicard	18	34	31	39	22	24	35	44
Qualitative meth.	-	-	0	58	0	7	0	31
Quantitative meth.	16	12	28	15	9	9	12	12
Reactive spray/heat	0	0	-	21	17	31	-	-
SCT	16	17	22	17	20	17	14	14
TDM-A	-	-	46	32	-	-	-	-

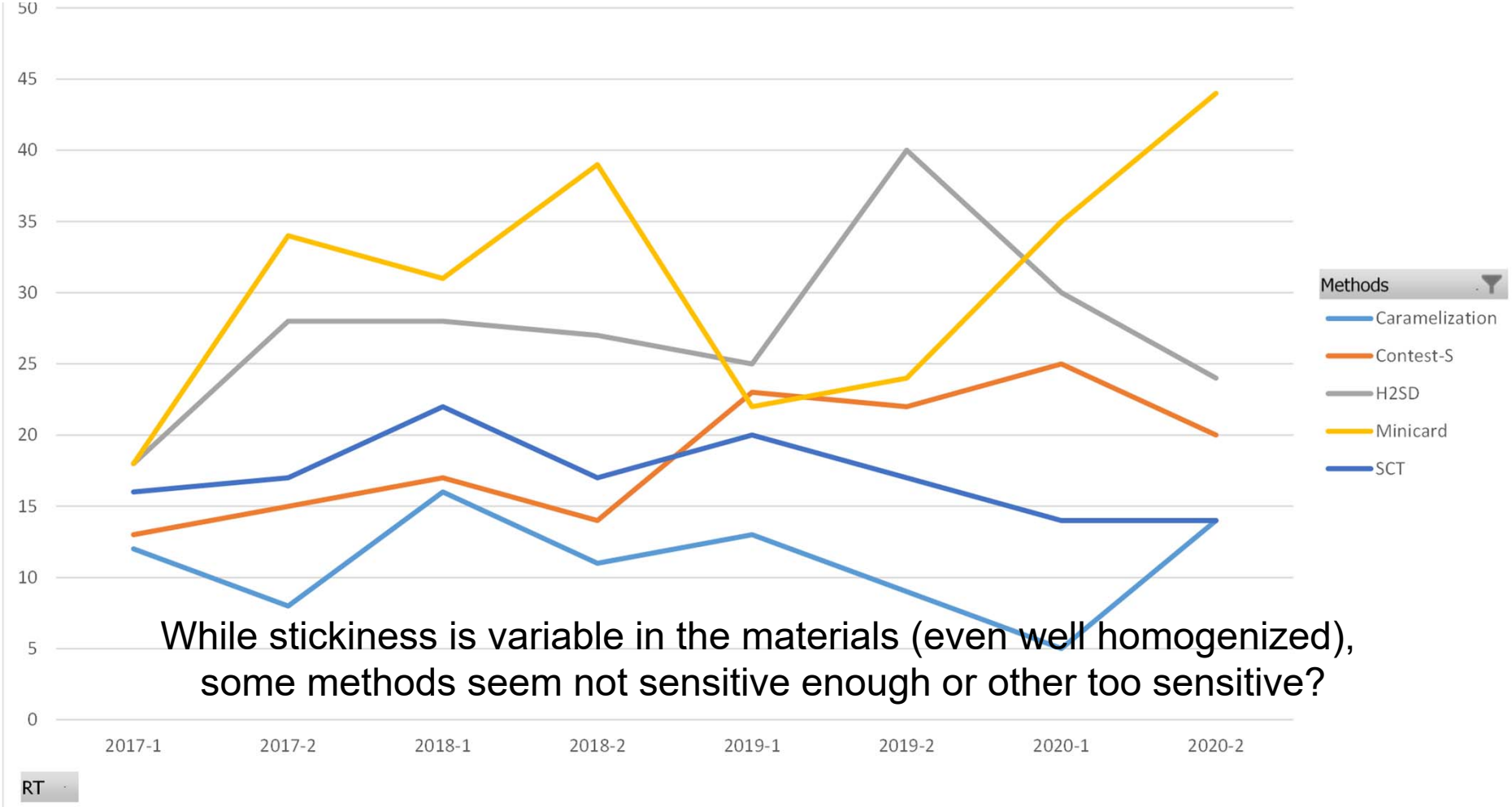
- = No participation or only one reading per cotton

0 = All readings the same=Strange

* Includes data 'raw', 'variab' and 'late'

Evolution of variances [RT 2017-1 - RT2019-2]
Within-method and Between-LabIDs

Means of the CVs% of raw data* (all labs/method, all cottons)

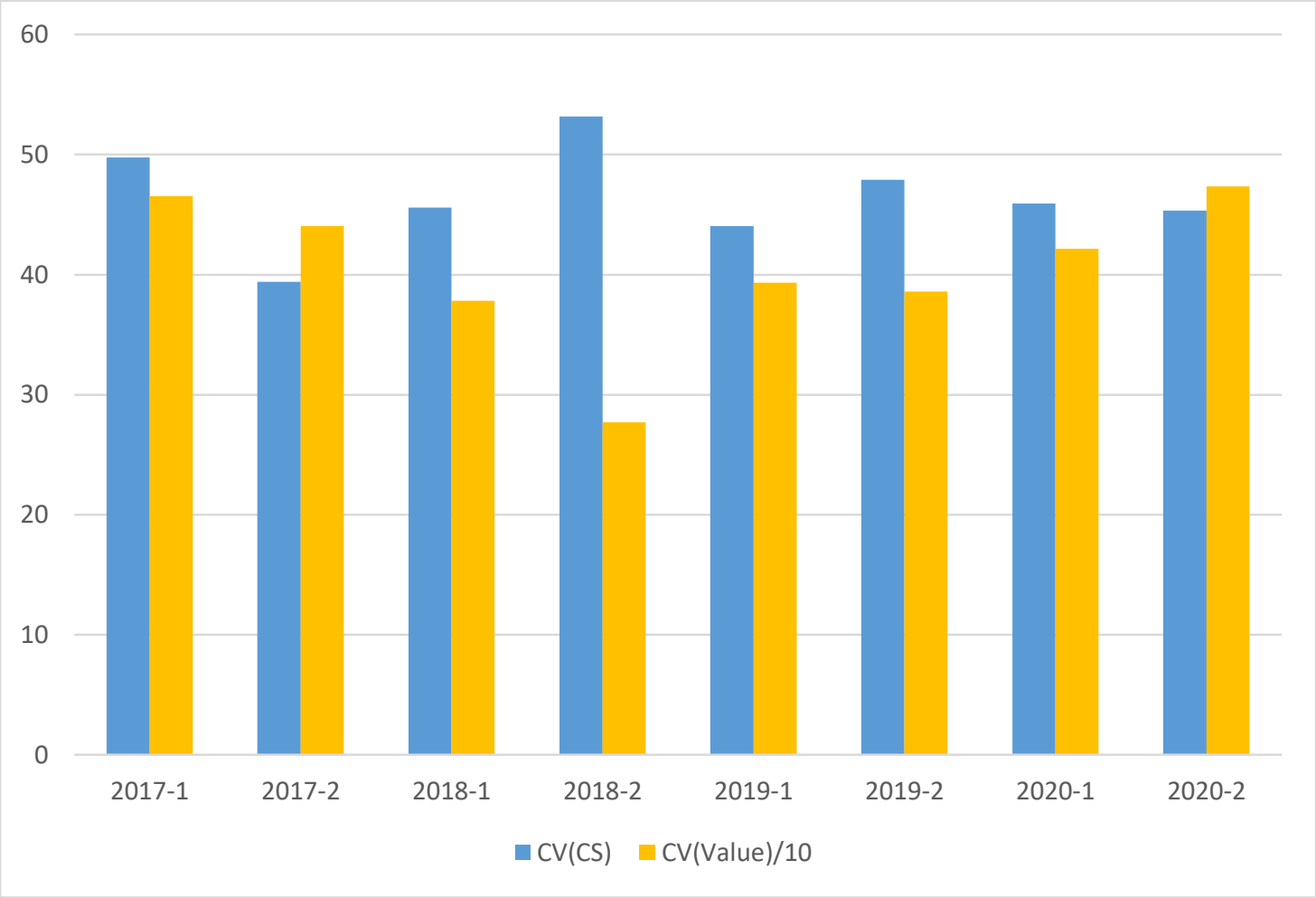


While stickiness is variable in the materials (even well homogenized), some methods seem not sensitive enough or other too sensitive?

* Includes data 'raw', 'variab' and 'late'

Evolution of variances [RT 2017-1 - RT2019-2]
Between methods

Means of the CVs% of raw data* (all labs/method, all cottons)



* Includes data 'raw', 'variab' and 'late'

Example of categorization
Non-sticky material (RT2019-1)

MaxEver

Example of categorization
Non-sticky material (RT2019-1)

Method	Unit	0_Lower reading_stickiness	1_No_stickiness	2_Light_stickiness	3_Medium_stickiness	4_High_stickiness	5_Very High_stickiness	6-Extreme_stickiness
Benedict	Grade	0	1.99	2.99	3.99	4.99	5.49	6
Caramelization	Color degree	0	1.99	3.99	5.99	6.33	6.66	7
Clinitest	Color chart	0	1.2	2.3	3.5	4.7	5.8	7
CommonScale	%	0	16.59	33.19	49.79	66.39	82.99	100
Contest	Grade	0	50.99	100.99	160.99	250.99	500.99	750
Contest-S	Grade	0	50.99	100.99	160.99	250.99	500.99	750
Fibermap	Grade	0	50.99	100.99	160.99	250.99	500.99	750
GB/T13785-1992	Color degree	0	0.7	1.3	2	2.7	3.3	4
H2SD	Sticky points	0	4.99	12.99	22.99	33.99	51.3	70
HSI-NIR	Sticky points	0	25	50	75	100	125	150
KOTITI	Kotiti grade	0	0.99	1.99	3.99	5.99	7.99	9
Minicard	ITMF grade	0	0.49	0.99	1.49	1.99	2.49	3
MinicardC	Cirad grade	1	1.59	3.59	4.59	5.59	6.3	7
Qualitative method	Grade	0	0.7	1.3	2	2.7	3.3	4
Quantitative method	Percent	0	0.19	0.39	0.59	0.79	0.99	1.2
Reactive Spray	Spray grade	0	1.3	2.7	4	5.3	6.7	8
SCT	Sticky points	0	5.99	16.99	32.99	53.99	101.99	150
TDM-A	Sticky grade	0	4.99	6.49	7.99	8.66	9.33	10

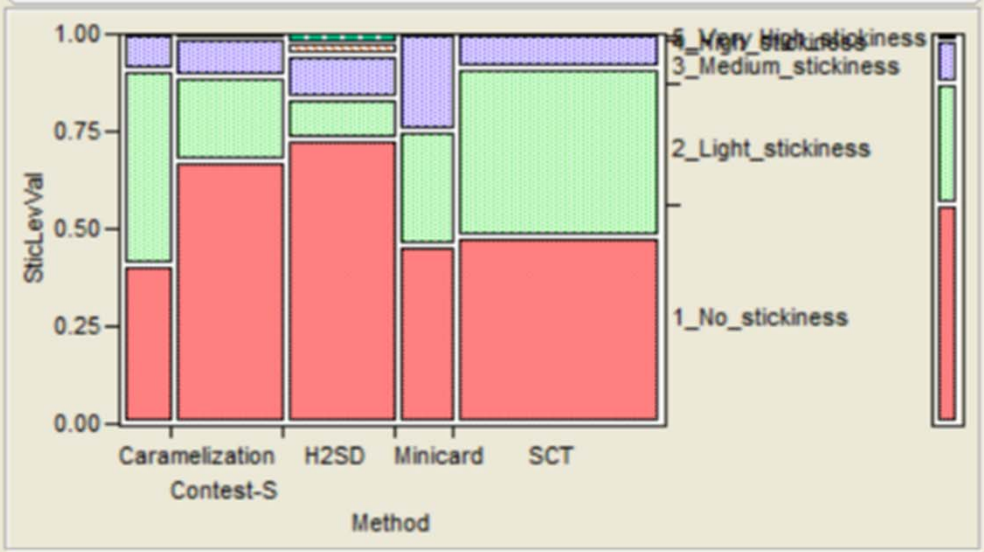
MaxEver

Example of categorization
Non-sticky material (RT2019-1)

Cotton=C05

Contingency Analysis of SticLevVal By Method

Mosaic Plot



Contingency Table

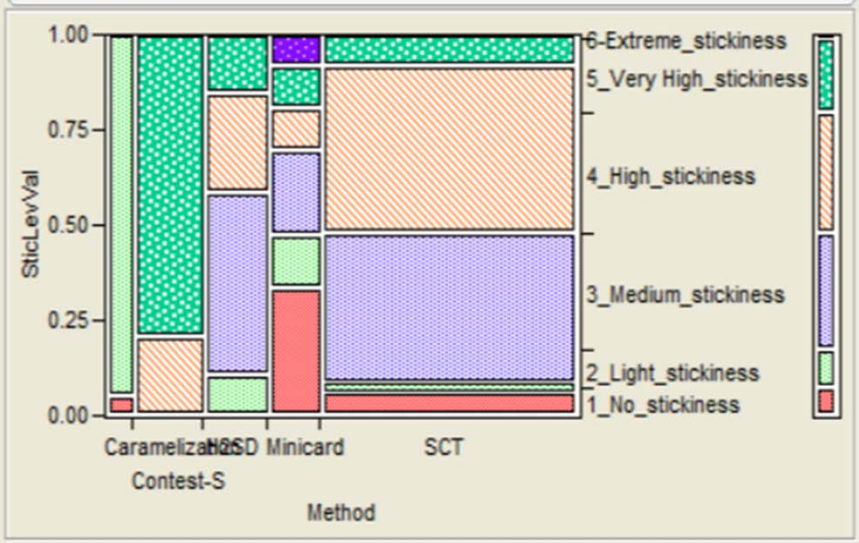
Method	Count	1_No_stickiness	2_Light_stickiness	3_Medium_stickiness	4_High_stickiness	5_Very High_stickine	
	Caramelization	18	22	4	0	0	44
	Contest-S	62	20	9	1	0	92
	H2SD	67	10	10	3	2	92
	Minicard	22	14	12	0	0	48
	SCT	81	73	14	0	0	168
		250	139	49	4	2	444

Example of categorization
Sticky material (RT2019-2)

Cotton=C06

Contingency Analysis of SticLevVal By Method

Mosaic Plot



Contingency Table

SticLevVal

Method / Count	1_No_stickiness	2_Light_stickiness	3_Medium_stickiness	4_High_stickiness	5_Very High_stickine	6-Extreme_stickiness	
Caramelization	1	19	0	0	0	0	20
Contest-S	0	0	0	10	38	0	48
H2SD	0	5	22	12	7	0	46
Minicard	12	5	8	4	4	3	36
SCT	11	4	70	77	14	0	176
	24	33	100	103	63	3	326

Technical objectives of RTStick
What did we learn yet?

Lessons

- Variety of units, of methods, of approaches
- Measure within-methods variations → possible
- Measure between-instruments within-methods variations → need indicator
- Proposal of CommonScale unit → check for best
- Proposal to work on some methods only (stickiness) related to stickiness in practice → see plenary talk
- Necessity of reference materials → is it workable and efficient? Experiments are ongoing
- But many questions on how to proceed (// trash?)

Technical objectives of RTStick
What did we learn yet?

The more labs participate:

- the more we learn!
- the more we can share that knowledge!
- the more we can measure progress!

Please accompany us in RTs
And/or provide materials to feed the RTs

Contacts: jean-paul.gourlot@cirad.fr
Drieling@Faserinstitut.de