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Steps towards suitable stickiness test results for trading and processing

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STEPS TOWARDS SUITABLE STICKINESS TEST RESULTS FOR TRADING AND PROCESSING

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Abstract

Stickiness has been defined as “the propensity of honeydew contaminated fibres to stick to spinning parts during their processing”. Several measuring methods have been designed to measure that type of stickiness using various measuring principles, but still their results are not converging for each individual contaminated cotton. Based on accumulated results within the periodical ITMF-ICCTM round-test for stickiness methods, this paper focusses on the major constraints that have to be levelled-out with the next steps of the international harmonization work for this property. The final aim is to allow the use of stickiness results for trading when required.

Introduction and context

Stickiness originates from various sources: Insect sugars as well as plant sugars and other sources as vegetal parts, oil traces or wax. The most important and problematic cause of stickiness has been attributed to the entomological sugars from insect (mainly whiteflies and aphids) secretions forming the insect honeydew. Even though this honeydew may first be limited in a part of a field and therefore in a limited amount of seed-cotton, seed-cotton picking, collection, and ginning operations usually disseminate this honeydew into a larger portion of the production. Therefore, an initially localized problem of stickiness, more or less intense at the origin, is then disseminated within several bales in a lesser degree of intensity but in a higher variability level within the fibrous material.

Stickiness induces productivity and quality losses as sticky points remain in the material, from fibres in the field to some semi-transformed products. The behaviour of contaminated fibres during processing is highly dependent upon the quantity of the sugars present, the types of the main complex sugars (melezitose and trehalulose mainly) present in fibres and their proportions in honeydew. This behaviour also depends on transformation machine types and settings, as well as on processing conditions (temperature and relative humidity in spinning rooms as well as temperature of processing parts in machines of transformation). In general, honeydew deposits stick onto machine parts taking with them fibres out of the fibre flow, thus generating a defect that could expend from some fibres only to the whole material flow rolling up onto the cylinders (Figure 1, (a)), up to a point the processing becomes impossible

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(Figure 1, (b)).

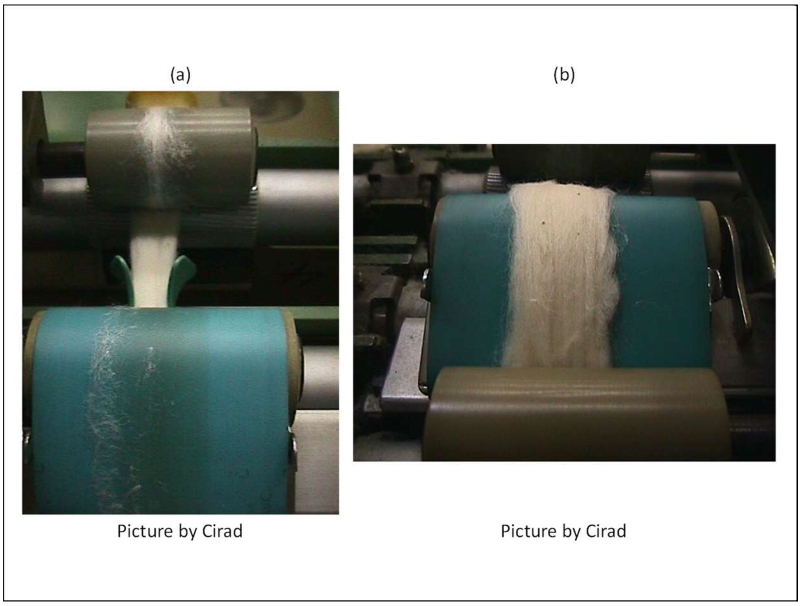

Figure 1: Sticky material on a cylinder probably affecting material evenness (a) and stopping the production (b) (Picture by Cirad ®).

Because stickiness is a contamination that is potentially causing quality and productivity problems in processing, surveys and tools have been designed to quantify its importance. Of course, this collected information serves the purpose of marketing fibres with incidences on both the reputation and the premium/discount received by producers.

On one side, ITMF has conducted periodical surveys (ITMF 2016) for quantifying stickiness importance based on records of perception by spinners processing various cotton origins. But unfortunately, the information is only of limited value without quantifying the number/share of bales and the level of stickiness. When available, ITMF divided up these perceptions into two categories “Least affected” (green) and “Most affected” (orange) origins in the reports, whose data is displayed in Figure 2. Along the period from 1989 to 2016 and depending on the processed origin, several situations have occurred: stickiness may have been never perceived, or from time to time, or permanently, or periodically, and in various intensities.

On the other side, several instrument manufacturers or method inventors have designed characterization methods to estimate the importance of a possible contamination of cotton fibres by insect honeydew. These characterization methods are performed on cotton fibre samples. The main goal of these measuring techniques should be to ‘predict’ processing and quality problems that could occur in the industry, with its main target being the spinning industry. However, it is not always the case (ITMF-ICCTM 2016, 2018).

Indeed, as soon as characterization methods appeared, the spinning industry has tried to use them in order to organize both 1) their bale laydowns to limit further potential processing problems and 2) their cotton purchases accordingly. At that moment, it appeared that these methods are more or less predictive of the stickiness potential of the fibres during their processing on one hand, and more or less sensitive to various stickiness contamination levels or origins on the other hand.

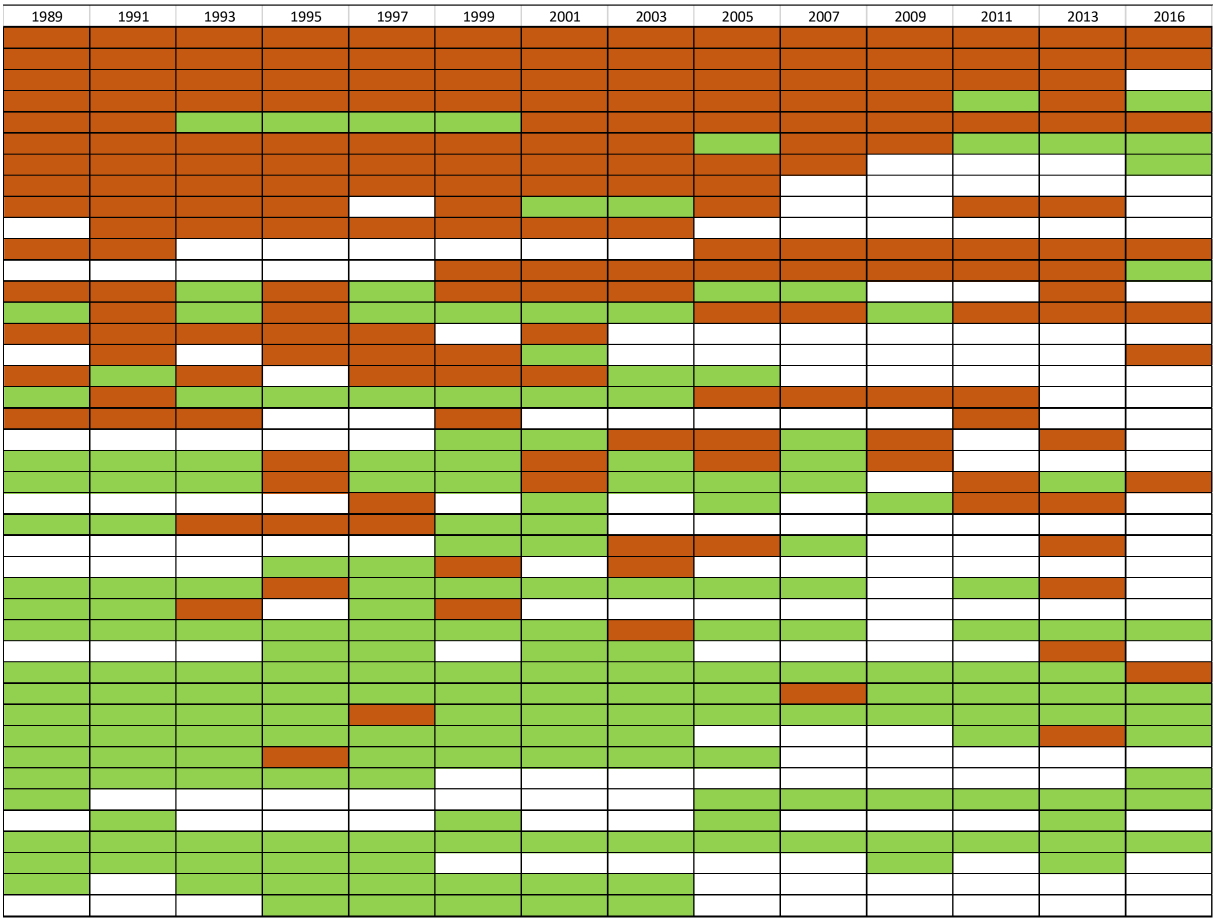

Figure 2: Extract of available ITMF contamination surveys data (one line = one production area, ordered from Most to Least affected origin). According to ITMF categorization: cells in green as 'Least affected'; cells in orange as 'Most affected'; cells without colour when no available data.

Research results indicated several reasons for these lacks of ability in predicting spin-ability. In general,

- There are, depending on the testing method, differences between the detection of different sources for stickiness and their effects in processing.
- The presence of sticky material is very variable within and between cotton fields, within and between origins, within and between lots, within and between bales, …
- Testing methods (discussed later and shown in Figure 3) may be more or less sensitive to the high variability of stickiness observed at all levels, starting from within-sample, within-bale, within-lot, …, to any level of observation.
- Machines and processing conditions (like e.g. humidity) may interact with stickiness when it is present and could increase or in the contrary decrease its incidence.

Due to all of these at least, no harmonization has been successful yet for getting reliable and trustable stickiness results able to serve the objective of trading cotton bales on large scales or to predict process-ability suitably.

Our aim

Based on the analysis of stickiness round-test results from various methods with their variations and influences, and with a view to processing, we intend to strengthen stickiness testing by:

- First harmonize results between instruments within each testing method,
- Then harmonize between the different testing methods based on their ability to predict processing.

This should allow the industry to use stickiness measurements to fix problems and ultimately the traders to market cotton based on measurement results.

Measurement related to practice

How stickiness affects processing

Any measurement should be used to predict the processability and/or the intermediate or end product properties. In our case, stickiness mostly affects transformation processes when honeydew droplets adhere to machine parts when they transport thinner layers of fibres, whose fibres then remain stuck together with the honeydew onto machine parts. Then, these aggregates may accumulate additional fibres thus creating at best a default in the evenness of the processed material, or at worst, an accumulation such as the transformation process cannot continue (Fonteneau-Tamime, Frydrych, and Drean 2001; Fonteneau-Tamime, Gourlot, and Gozé 2001).

The steps in the spinning process where thinner layers are transported, are at the card and then at each drafting zone of the spinning process, at the drawing frame, at the flyer and at the spinning frame in ring-spinning operations. In open-end machine, stickiness deposits can at least clog the rotor and cause quality and or productivity problems. All these steps are more or less sensitive to the presence of honeydew depending on several processing factors alone or in interaction:

- Temperature and the relative humidity of the air surrounding the fibres;
- Temperature of the machine parts;
- Duration of the contact between the honeydew and the warm machine parts;
- Pressure with which honeydew is pressed against fibres and machine parts;
- Type of sugar in the given insect honeydew, depending on the types of insect present in the cotton fields.

It is important to mention here that stickiness also could induce problems in weaving and knitting machines as honeydew deposits usually remain in the yarn packages. In these machines, stickiness can accumulate and clog the parts guiding yarn threads, thus possibly causing yarn breakages. Indeed, the accumulated honeydew remains in the materials during the preparation to weaving step or after weaving / knitting steps, until their first washing that takes place at the dyeing and finishing steps in the textile process.

Based on the above, several manufacturers have designed tools, devices or methods (hereafter called ‘methods’) to directly measure or infer stickiness based on the measurement of other components or properties. For the time being, stickiness is define as “the propensity of insect honeydew contaminated fibres to stick to spinning parts during their processing”, and our aim is to harmonize its measurement.

Stickiness testing methods and their ability to predict impacts on spinning

The existing measuring methods may be categorized into four groups (Figure 3): chemical and physical methods for measuring sugar contents and ingredients, mechanical and thermo-mechanical techniques (mostly measuring actual stickiness behaviour). The problem is that despite the clearly different methods and their specific sensitivity and influences, the users expect all methods to be perfectly predicting stickiness in their specific processing stages.

Individual sugars (melezitose, trehalulose, …) contained in insect honeydew are various and in various contents on one side, and each of these individual sugars have hugely varying sticking potentials on the other side. Therefore, measuring total sugar contents cannot be highly predictive of stickiness manifestations during processing. It has also been found that stickiness due to whitefly honeydew is different from aphid honeydew due to their respective individual sugar contents (trehalulose and melezitose mainly), while each of these individual sugars have various physical and chemical behaviours during the spinning operations (Hequet 2003).

In the 1980s, ITMF-ICCTM has designated the mini-card as the reference method for measuring stickiness, even though results are very sensitive to the operator and operating conditions. In addition, this instrument is not produced anymore. The Sticky Cotton Thermodetector (SCT) has been recommended by ITMF in 1994 for daily/practical operation in laboratories for spinning mill / trading purposes based on its thermo-mechanical principle, as it has been shown predictive of stickiness as measured by the mini-card and in spinning facilities. Several methods that are more recent have been developed such as the High Speed Stickiness Detector (H2SD) and Contest-S, which are able to provide results in a quite rapid manner.

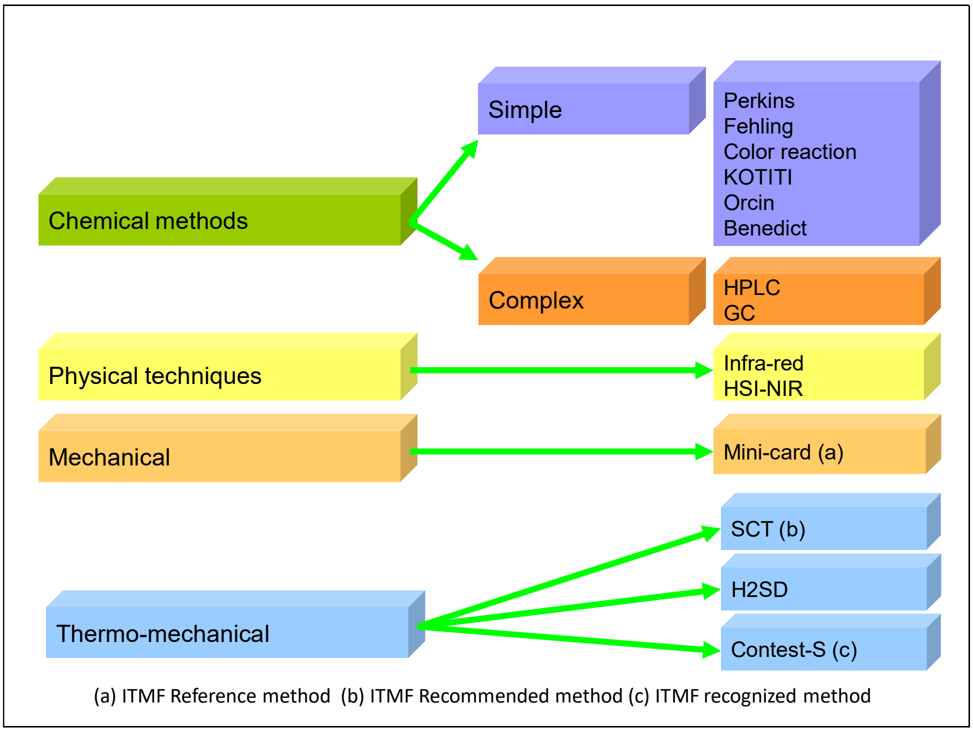

Figure 3: Possible methods to estimate stickiness, but not all are currently measuring stickiness.

To experiment all above processing factors in interaction in one single trial, a first, intense inter-laboratory test was conducted in 2013/14 (report in ITMF-ICCTM 2014). It included most of these methods in various worldwide laboratories and allowed to compare to results of micro-spinning tests measuring yarn productivity and yarn quality evaluations. In this experiment, 20 tex yarns (Ne 30 or Nm 50) were produced for a range of 11 cottons in two ambient conditions (report in ITMF-ICCTM 2016). The conclusions were " […/…] 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. At the same time, units in which stickiness potential is expressed also are fully different. Additionally, differences were observed in levels readings, both within laboratories using the same technique and between techniques. […/…] Some stickiness measuring techniques may predict more than others the potential spinning problems at 58% RH and potential losses in quality (in decreasing ranking order of prediction ability of the 36 yarn criteria[b]:

- Mini-card: 29/36 criteria or 81%,
- SCT: 78%,
- H2SD: 78%,
- Caramelization: 67%,
- Chemcare: 61%.

[…/…]. Strong attention is to be given to erroneous predictions from some stickiness measuring techniques (Chemcare), as they may correlate more to other characteristics than to stickiness expression during spinning in 45% RH conditions. […/…] »(Gourlot, Lassus, and Gawrysiak 2016). Certainly, different methods and instruments settings will result in changes in observed correlation levels, too.

In other words, a path toward harmonized results is to be found, and it has to be made with the understanding of all the causes that makes this stickiness measurement quite difficult (Gourlot, Lassus, et al. 2018).

However, for the time being, before developing further this harmonization part, here are some definitions later used in the text:

- "Stickiness in practice" (SIP) found in spinning (under usual conditions) that one would like to evaluate from measuring methods results,
- "Measured Stickiness Raw" (MSR) as the results in a counting or continuous scale provided by measuring methods,
- "Measured Stickiness Scaled" (MSS) as the results in a counting or continuous scale after scaling results to match another method or a consensus level/scale,
- "Measured Stickiness Categorization" (MSC) as the results of a categorization (e.g. not sticky, slightly sticky, sticky, highly sticky, …) given by measuring methods either directly or converted from MSR or MSS.

For the SIP property that the industry would like to evaluate, the measured characteristics (MSR) are various, going from sugar contents down to sticky points counting various ways, all assumed to properly estimate or predict SIP but via one or more of the MSR, MSS or MSC. Some of the source of variability in stickiness measurement will be explored for isolating ways for harmonizing measuring methods results.

<u>Naturally given variation of stickiness in the bales and impact on measurement results</u>

As an example, Frydrych (Frydrych et al. 2004) experimented several bales from four

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[b] Recorded yarn quality characteristics: 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. Recorded productivity characteristics: nb. of attachments, of rolling-up, of cleanings, of breaks, total nb of events, nb of events/km, length oy yarn produced with a given initial mass and productivity in m/mn.

various cotton origins (altogether twenty-four bales) to cover a range of stickiness. Four samples (100 grams) were independently taken from each of eight layers from the selected bales. Stickiness testing was made using H2SD (two measurements per sample). Figure 4 shows that variations in the numbers of sticky points could be very large within some bales based on 32 samples per bale even though the mean value per bale could be quite low.

This analysis is one way to show the existing variation within bales, based on one method, one instrument and one operator. Therefore, any variation from different methods, instruments, operators, testing conditions was excluded. Nevertheless, it is difficult to quantify the variation with a specific variation parameter, as it is largely depending on the actual variation within each bale and also depending on the average level (Figure 4). In general words, the given range was and is at least doubling the mean test value for the majority of the bales. Hence it will always be difficult to define valuable trade limits for stickiness, although the graphs show that different levels of stickiness in different bales can be distinguished from each other. And certainly, as always, the number of tests per sample and the number of samples may allow reducing the uncertainty of the results.

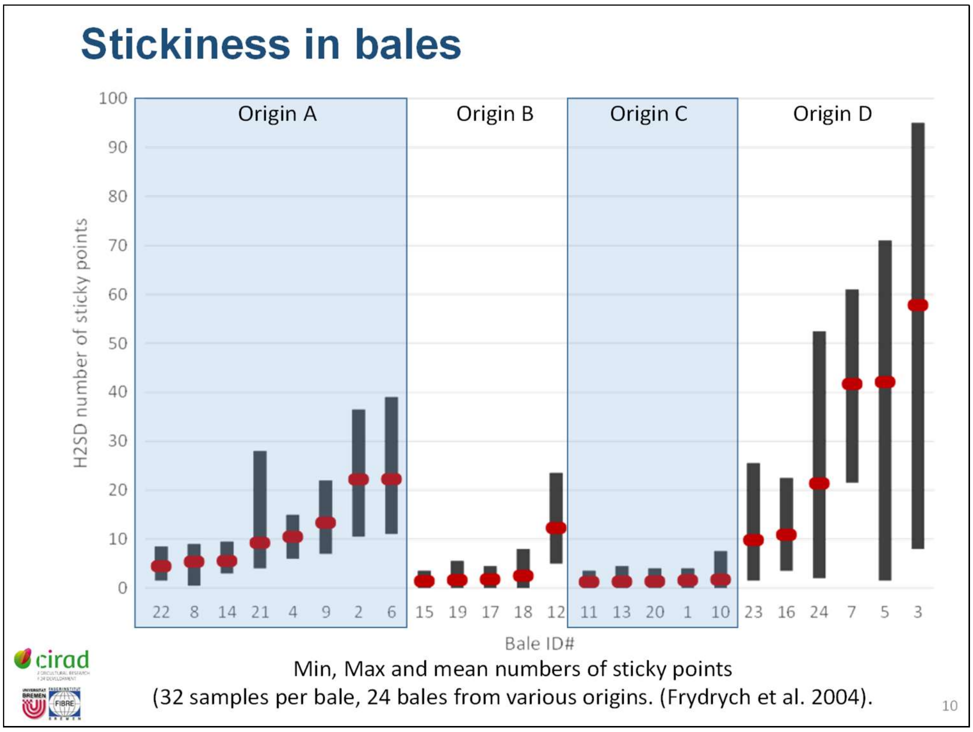

Figure 4: Min, Max and mean numbers of sticky points as measured by H2SD on 32 samples per bale from 24 bales from various origins. Bales ordered by increasing Mean value of the samples taken in the bales layers (top chart) or using the cutter sampling system only (bottom chart) (Frydrych et al. 2004).

The variations come from the fact that insects make their honeydew deposits in both a random (where they are at that time) and patchy manner (depending on the number of insects present in that location). In addition, the type of cotton plant architecture and of crop protection has an impact on both the number and the types of insects in the cotton field at any moment of the crop.

Then, cotton picking practices (manual, picker, stripper and the way they are performed) induce splitting or mixing or fragmenting the sticking materials among the seed-cotton collected, at two levels: within and between modules. These modules are then processed at the ginning mill where this splitting / mixing / fragmenting process are even more emphasized by ginning and (seed-cotton and lint) cleaning operations, before splitting the whole material between bales and lots of bales.

When insects are homogeneously spread in the fields, there is a large probability that stickiness is also spread homogeneously in the seed-cotton from that field and therefore in the corresponding bales. When insects are not homogeneously spread in the fields, a situation as described by Figure 4 may happen in some extent. If those twenty-four bales would be part of a delivery lot to a spinning mill, then half of the bales would not be problematic, while the rest of them would probably be partially (those with low mean and high variation) or very problematic (those with both a high mean stickiness and a high variation of stickiness within each bale).

Some observations and discussions on observed variations in round-tests results

Considering the existing variability of stickiness and the past efforts toward harmonization, the authors decided to run periodic inter-laboratory round-tests (hereafter named ‘RT’) with as many laboratories and methods included as possible. Two tests per year since 2017 have taken place. In this document, in sum six Round Tests (2017-2019) with 26 samples were tested by around 20-35 laboratories. The individual test results are anonymous to public, but every participating laboratory (coded with a Lab-ID) knows its own results in comparison to the average and the anonymized distribution of all labs. More or less eleven measuring methods have been used during these round-tests.

The observed variability in RTs results and in the real life may come from various sources; only the following ones are explored hereafter in order to deduce their effect on results and also the potential next steps toward the harmonization of this measurement:

- Effect of the reading levels for each testing method,
- Effect of the natural variability of stickiness,
- Effect of the material preparation,
- Effect of the sampling of any given material into several samples,
- Effect of the measurement result levels on the level of variability in measurements,
- Variability in stickiness characterization results for single instruments using one common material along time,
- Correlations between methods,
- Finding a common scale to report results.

<u>Effect of the reading levels for each testing method</u>

As seen in Figure 5 and Figure 6, several units are used to report results about stickiness (MSR). In these figures, results for five cotton samples in one RT are shown. Each lab is given with its Lab-ID on the x-axis. The variation of results within each lab can be seen with the small black crosses for each lab. The variation between the laboratories can be seen by comparing averages for each laboratory on each cotton sample (shown with a red cross). Each laboratory can be compared to the average of all laboratories, which is given as a dotted horizontal line for each sample. The variation will be discussed later in this document.

Displaying all available data from all methods per RT would require one page for each of the eleven methods involved and each of the RTs. Reading the charts for comparing results from various instrument within each testing method remain quite easy, while comparing methods is more difficult.

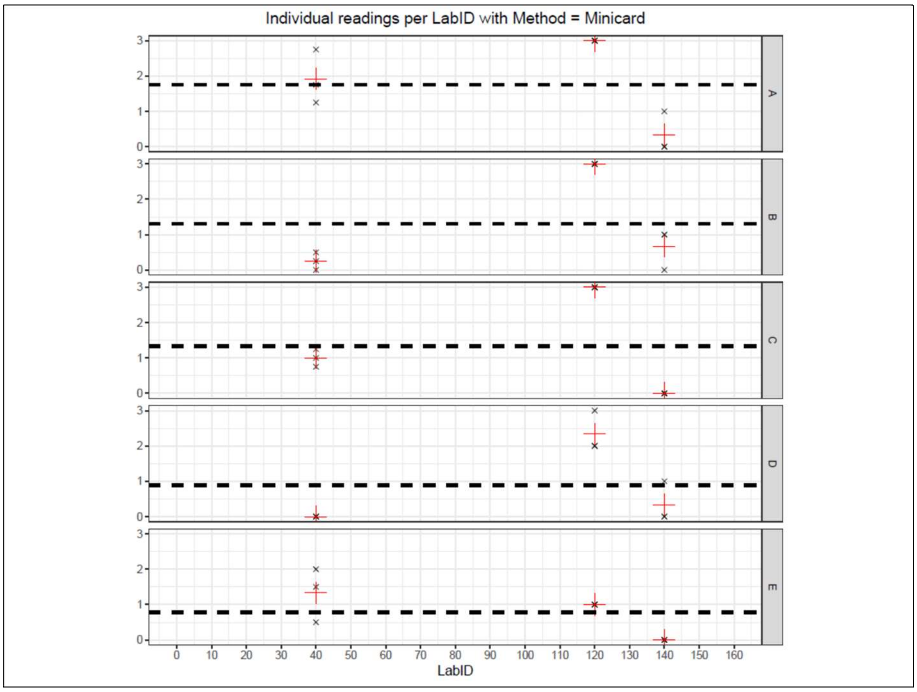

Figure 5: Extract from the RT2019-2 report: Minicard results for five cottons A to E by various laboratories.

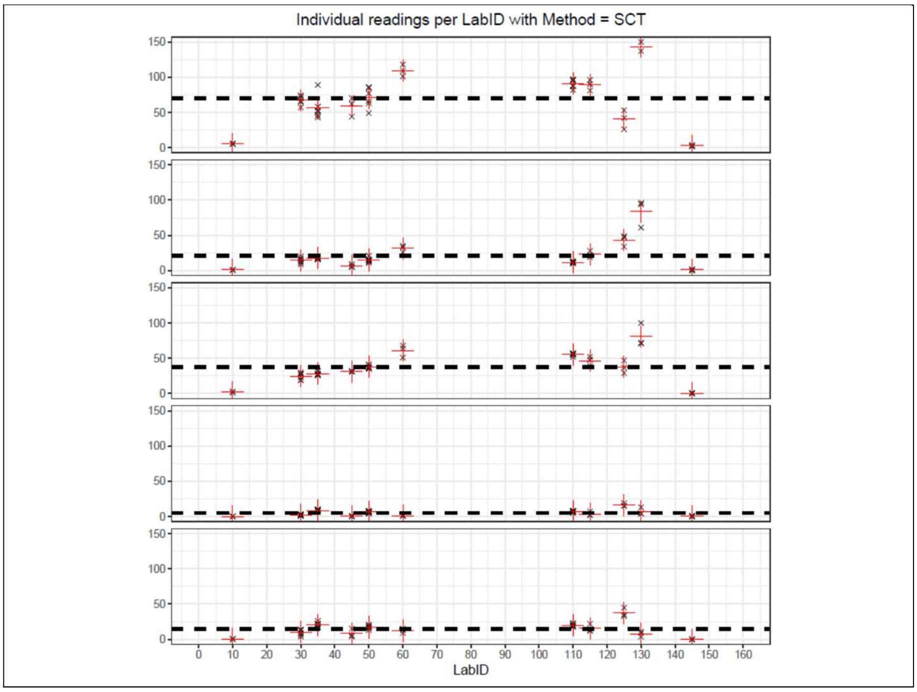

Figure 6: Extract from the RT2019-2 report: SCT results for five cottons A to E by various laboratories.

Effect of the natural variability of stickiness, and ability to detect stickiness depending on sample size

Depending on the initial population of sticky points within the bales and depending on the size of the samples used for stickiness characterization, MSR, MSS, and therefore corresponding MSC, may vary greatly. The same representation could be extended to bales and to lots of bales, also based on the same two ‘cutter samples per bale’ taken in each external layer of the bales from which sub-samples are tested.

At this point, the natural variability that exists for stickiness was approached, and it can be observed at various levels: between specimen within a sample, between samples in a bale, between samples in lots, *etc*. It is time to study the effect of cotton homogenization on sticky points distribution, to explore what happens for instance in the first operations at the spinning mill.

Effect of the material preparation on stickiness characterization results

The intensity and variability of stickiness results (MSR in this case) were measured at several cumulative steps of fibre preparation on independent cotton masses:

- 1 = Raw,
- 2 = 1 + CSITC Homogenizer,
- 3 = 2 + Minicard without flats,
- 4 = 3 + Minicard with flats.

The original material was a collection of ‘cutter samples’ taken from various bales. Between twenty and twenty-five independent samples of fibres were taken out from the cotton for each preparation modality. All samples were tested on H2SD with one measurement per sample.

Figure 7 (top chart) indicates that a trend exists indicating a reduction of the variance of the total number of sticky points depending on the number and the type of preparation steps. At the same time, Figure 7 (bottom chart) indicates that the number of sticky points increases with the number and type of operations as more and more sticky points.

The assumptions for this are that:

- more sticky points from the H2SD fibre pad, first hidden in the materials, processed by H2SD have been put in contact to the aluminium foils when the number of preparation steps were performed prior testing, and,
- the size of sticky points is shifted toward smaller sticky points when more preparation steps were involved prior testing; this could be explained by the fragmentation of bigger sticky points into smaller sticky points as soon as cotton is processed, at least by the Minicard.

As an important conclusion and consequence, only cottons processed with the CSITC Homogenizer will feed RTs in order to not or least affect sticky points size distributions. Any additional processing step of the materials would move the Round Trials samples away from realistic samples. Indeed, the final aim of the RTs is to harmonize methods results in order to predict SIP, even though a larger variance in results may complicate this harmonization.

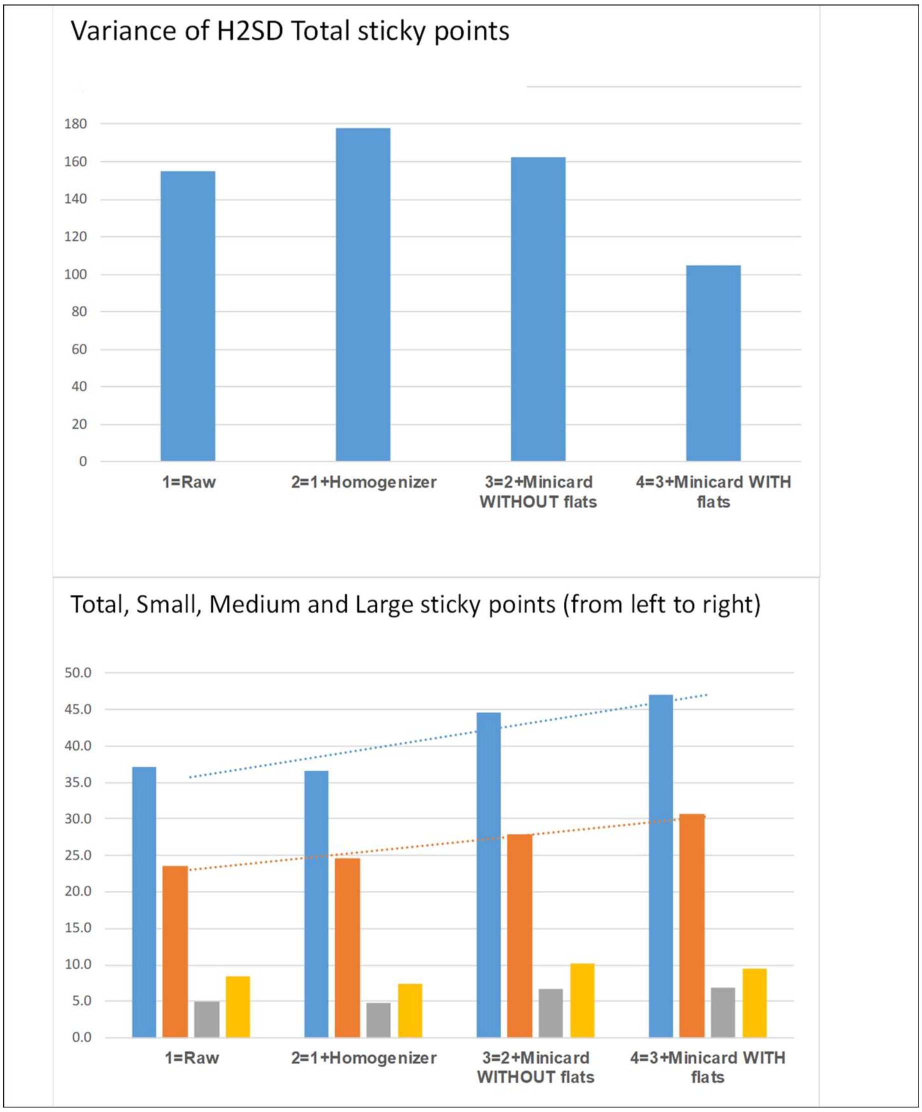

Figure 7: Effect of sample homogenization on H2SD number of revealed sticky points counts per class of size. Example for one cotton. Based on 20+ samples, one measurement per sample.

<u>Effect of the sampling of any given material into several independent samples on the repeatability of stickiness characterization results</u>

Taking care of the large variability between methods and instruments results shown in Figure 5 and Figure 6, a specific experiment was designed to test a following basic assumption made earlier in this document: this assumption was that cotton original masses are homogeneous enough (thanks to the CSITC Homogenizer preparation discussed above) to allow grabbing proper representative samples to be disseminated to participating laboratories in the RT with the sole objective of properly measuring their performances.

Therefore, for RT 2019-2, approximately fifteen additional sets of the same five cottons used to that RT were prepared. These sample sets were tested in series on one instrument of each of the following methods in the usual conditions: SCT, H2SD and Contest-S (one method=one laboratory, not all methods installed in the same laboratory). In addition, fibre characterizations (one laboratory only) were also performed in order to compare between-samples variations for stickiness to one of the other fibre characteristics.

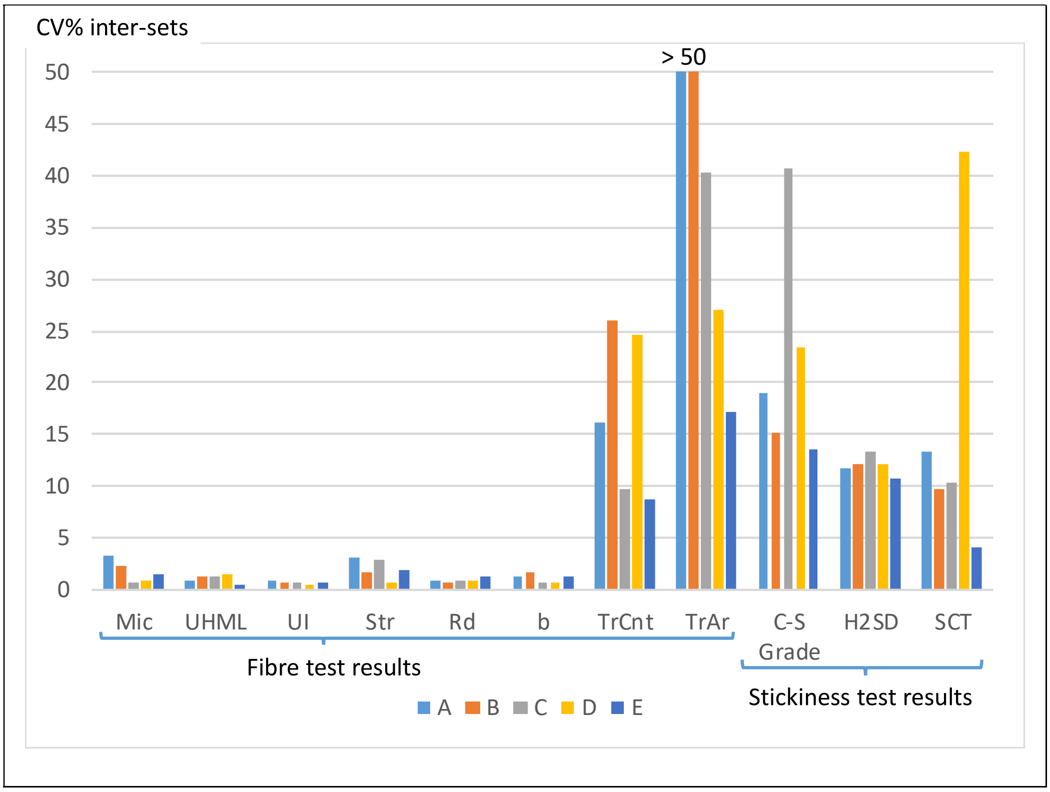

Figure 8: Observed CV% between sets of samples for RT2019-2, for Cottons A to E: in a truncated y axis to better compare CV% values for fibre characteristics to stickiness ones. b=yellowness, C-S Grade=Contest-S Stickiness Grade, H2SD=H2SD total of sticky points, Mic=Micronaire, Rd=Reflectance, SCT=SCT number of sticky points, Str=Strength, TrAr=Trash Area, TrCnt=Trash count, UHML=Upper Half Mean Length, UI=Uniformity Index.

As expected, Figure 8 indicates that the variations (CV%) between sample sets for fibre characteristics for each of the Cottons A to E are low in comparison to stickiness measured characteristics by the three used methods. Indeed, the original variability of fibre characteristics among a cotton mass is low in comparison to any contaminants present in the fibres (please remember that these cottons were homogenized in addition to be part of the RT, which improves their homogeneity). It is also important to note that stickiness measured characteristics are in the same range of variations as the ones observed for trash content and trash area which already benefit from an effort of worldwide harmonization. It can also be shown that these observed CV% can be very high just because the average levels of reading are very low. In our example, CV% for trash area for most cottons are high because the averaged readings are low (between 0.09 and 1.09%, as explained in other words in the next paragraph). For giving a quantification of the repeatability specifically for stickiness, Table 1 reports means, standard deviations and CV% for results in RT2019-2.

As a conclusion for this part, the preparation of cottons and the way samples are drawn from cotton materials for feeding RT cannot be better for stickiness measurement purposes, as the variations for fibre measured characteristics cannot be lower without affecting both fibre properties and sticky point size distributions. From this, it was decided to keep the same preparation procedure for coming RTs.

Table 1: RT2019-2 results: Between-sample-sets statistics for each of the cottons by three methods, sorted by stickiness evidence. Numbers of readings and instruments per Method may vary.

		Contest-S	H2SD	SCT
	Nb of readings/cotton	12	12	44
Cotton	Nb of LabIds	2	2	11
A	Mean	479.8	23.8	69.9
	SD	105.8	9.2	36.5
	CV%	22	39	52
C	Mean	348.3	21.5	37.6
	SD	62.4	12.0	21.0
	CV%	18	56	56
E	Mean	144.2	6.0	14.8
	SD	59.1	3.4	9.5
	CV%	41	56	64
B	Mean	87.4	5.7	21.0
	SD	41.6	5.3	20.7
	CV%	48	94	99
D	Mean	28.0	3.7	5.1
	SD	12.7	3.5	4.6
	CV%	45	95	89

Effect of measurement result level of variability in measurements

The ‘mechanics’ of statistical methods and of CV% calculations in the case of counts induces bias in the way one could interpret results. Indeed, for measurement results expressed in counts with Poisson distributions, the CV% values (between-measurement readings) decrease with an increase of the mean measurement, as the variance equals the mean. The computation of these results is given in Figure 9.

From there, the conclusion is that comparing the CV% of inter-laboratory results is not a proper way to compare methods inter-laboratory performances, as this CV% depends on the mean level reading. For instance, for a same material, a Minicard rating vary from to 0 to 3, while Contest-S results vary from 0 to 760; for sure, from Figure 9, the CV% vary from almost 100 for the Minicard, while it could vary from 100 to 5% depending on the reading on Contest-S.

Therefore, other indicator(s) than CV% (such as a dispersion index) has/have to be found to compare methods in their ability to provide precise and reproducible results.

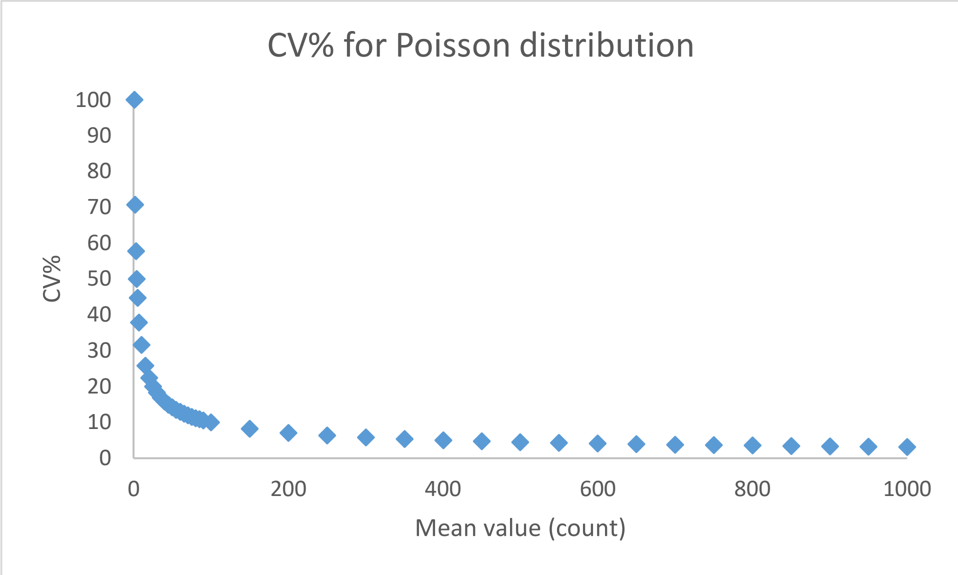

Figure 9 : Evolution of the CV% between measurements as a function of the mean of measurement results (Poisson distribution, where variance = mean).

<u>Variability in stickiness characterization results for single instruments testing one common material along several RTs from 2017to 2019</u>

Based on the ITMF-ICCTM Round Trials again, here is an exploration of results over time. The attempt is to show that results are variable within one single instrument from one measuring method in on single laboratory, even though it was based on the most homogeneous material as possible from which samples were distributed among participating laboratories. Figure 10 provides results gained with one instrument for each of four measuring methods – Minicard, SCT, H2SD and Contest-S – on one same material which was used in four of the latest RTs (low-medium range stickiness level). This material was labelled a different way at each of its participation into a RT; in these conditions, participating laboratories tested this material 'in the blind'.

As pointed out already, several units are used to report about stickiness results (MSR), and reading the charts is difficult (Figure 10, top chart). Therefore, the CommonScale(Max) scale representation (actual individual reading divided by the maximum 'MaxEver' reading possible by any given method as given in Table 3) has been first developed in order to match all methods' results. In both cases anyway, large variations can be observed in the results, meaning that laboratory performances have evolved more along time than the variation given in the tested material.

Nevertheless, the mean global evaluation and the global variation (Table 2) of stickiness seem to be almost at the same level for all these methods for that given cotton, even though a slope over time is observed in SCT results from one RT to the next (Figure 10, bottom chart); this lab may have change its practice over time.

One outcome of this study is that, given the proven efficient preparation method of the samples for feeding the RTs, the RTs can be efficiently used to check or track laboratory performances over time.

Table 2 : Variation of test results on one sticky sample in repeated Round Trials (solely based on one sample and one instrument). Top table expressed as MSR, bottom table expressed as MSS (CommonScale(Max)).

MSR

Method	Mean	SD	CV%
1-Minicard	1.3	0.6	46
2-SCT	34.1	8.6	25
3-H2SD	24.5	12.0	49
4-Contest-S	286.4	64.5	23

MSS (CommonScale(Max))

Method	Mean	SD	CV%
1-Minicard	42.2	22.5	53
2-SCT	22.7	8.0	35
3-H2SD	35.1	16.9	48
4-Contest-S	38.2	9.9	26

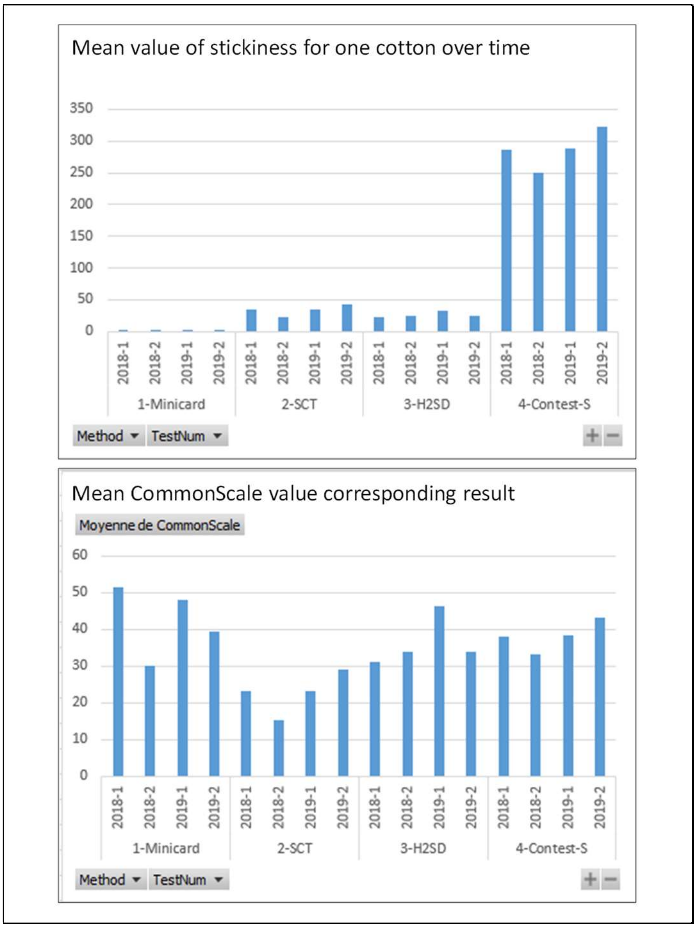

Figure 10: Results from the latest ITMF-ICCTM round-tests on stickiness measuring methods for one cotton from four methods (one instrument per method, means of 3 to 6 measurements per sample).
Top chart: raw results (MSR) provided by the instruments.
Bottom chart: CommonScale (Max) (MSS) corresponding results.

Correlations between stickiness testing methods results and choice of suitable stickiness testing methods for the future harmonization work

Results on six RTs, each with 3 to 5 samples (Gourlot et al. 2017a, 2017b, 2019b, 2019a; Gourlot, Drieling, et al. 2018b, 2018a) allowed the testing of 26 cottons (26 samples – stemming from 16 different bales/origins with some samples repeated) by up to 12 methods, each in 1 to 11 laboratories.

Table 4 shows the correlation coefficients for all 12 included testing methods. Because of the huge amount of data and graphs behind, only the R values are given and not the full correlation graphs, as there are too many information for being discussed here. In general words, the correlation between many methods to the Minicard or to thermo-mechanical methods is not very high, with the reasons explained in the first part of this publication. Hence, the study is restricted from now on to the 3 thermo-mechanical methods plus the Minicard as the reference, plus Caramelization as one wide-spread method representing for sugar-based testing methods.

Figure 11 represents this data set limited to five methods: Caramelization (here as an example of all sugar testing methods with a sufficiently high number of participating laboratories), three thermo-mechanical methods (Contest-S, H2SD and SCT) and the reference method (Minicard). It appears that there is no suitable correlation existing between Caramelization and all other methods (Figure 11, top chart), and especially with Minicard being the reference method; this is also observed with all other methods based on the testing of (simple) sugar content (Table 4).

Therefore, these results and those on the prediction of micro-spinning test results in 2014 study indicate that the harmonization effort should first concentrate on stickiness related measuring methods. So mechanical and thermo-mechanical methods only are considered, and not (simple) sugar testing methods.

Figure 11 (bottom) also shows that results from thermo-mechanical methods correlate well among themselves and with the reference Minicard method. Therefore, these four methods will be the basis of the first future harmonization efforts.

However, as Minicard is not produced anymore and shows a high variation between results compared to the other three methods, **our new harmonization focus will be on mechanical / thermo-mechanical methods only: SCT, H2SD and Contest, keeping the Minicard as the reference.** The main characteristics of these methods are given in Table 3.

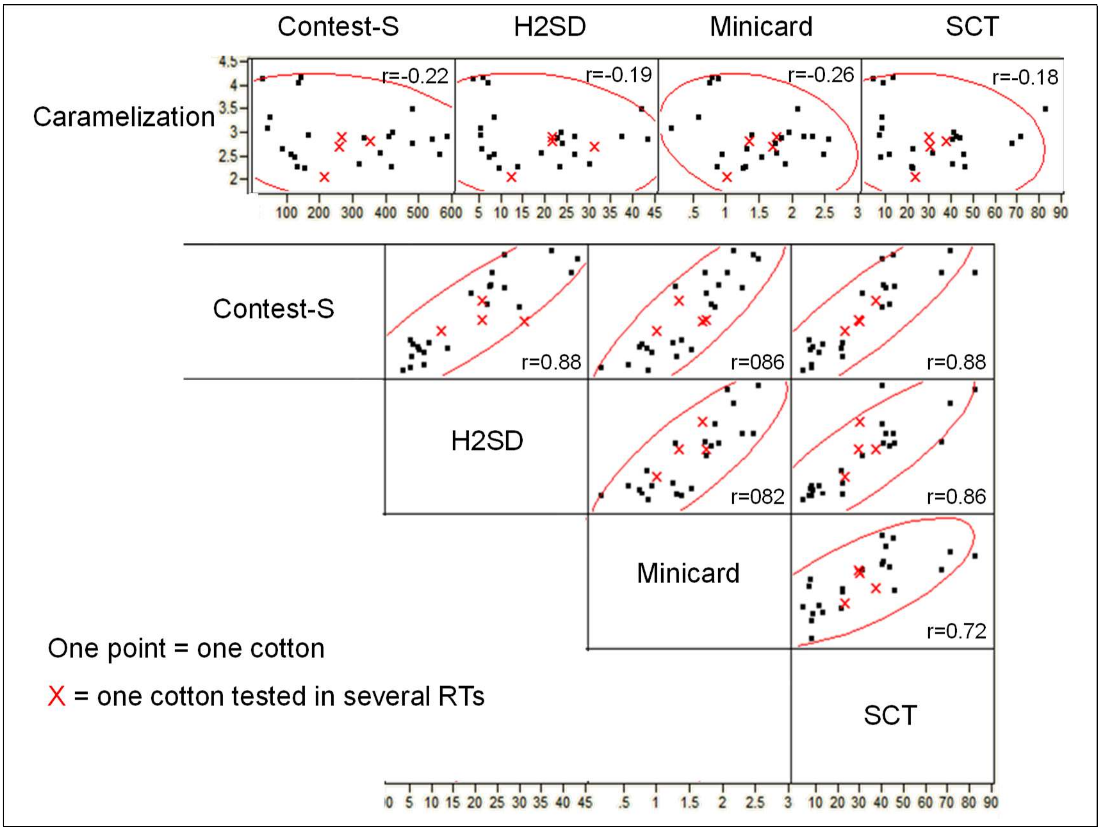

Figure 11: Correlations between Means per Method (several LabId / Method) and by Cotton (26 cottons) participating to RTs from 2017-1 to 2019-2 for five methods. Red symbol X for one the material that was used in several RTs, same data as displayed in Figure 10.

Table 3: Some characteristics of thermo-mechanical methods.

Methods	Unit	Usual range in scale (RT2019-2) between 0 and MaxEver	Temperature
Contest-S	Grade (based the numbers of sticky points considering their size)	0 - 750	35°C
H2SD	Sticky points (based on a categorization of the sizes of sticky points)	0 - 70	53°C
Minicard	ITMF grade	0 - 3	Lab climate
SCT	Sticky points (counting their number	0 - 150	84°C

Table 4: Pairwise correlations between "GrandMeans per Method".

	Benedict	Caramelization	Clinitest	**Contest-S**	**H2SD**	HSI-NIR	KOTITI	**Minicard**	Qualitative method	Quantitative method	Reactive Spray	**SCT**
Caramelization	-0.997 NS	1.000	0.069 NS	0.219 NS	0.188 NS	-0.302 NS	-0.496 **	-0.257 NS	0.000 NS	0.014 NS	0.484 *	-0.176 NS
Clinitest	-0.115 NS	0.069 NS	1.000	0.389 *	0.367 NS	-0.037 NS	0.217 NS	0.225 NS	0.009 NS	0.243 NS	0.007 NS	0.433 *
Contest-S	**-0.301 NS**	**0.219 NS**	**0.389 ***	**1.000**	**0.881 ***	**0.028 NS**	**0.609 *****	**0.859 *****	**0.248 NS**	**0.576 ****	**0.078 NS**	**0.880 *****
H2SD	**-0.613 NS**	**0.188 NS**	**0.367 NS**	**0.881 *****	**1.000**	**-0.071 NS**	**0.516 ****	**0.820 *****	**0.086 NS**	**0.587 ****	**0.030 NS**	**0.855 *****
HSI-NIR	0.300 NS	-0.302 NS	-0.037 NS	0.028 NS	-0.071 NS	1.000	0.283 NS	0.170 NS	0.427 NS	0.218 NS	0.048 NS	-0.162 NS
KOTITI	0.500 NS	-0.496 **	0.217 NS	0.609 ***	0.516 **	0.283 NS	1.000	0.594 **	0.368 NS	0.417 *	0.014 NS	0.472 *
Minicard	**-0.562 NS**	**-0.257 NS**	**0.225 NS**	**0.859 *****	**0.820 *****	**0.170 NS**	**0.594 ****	**1.000**	**0.208 NS**	**0.458 ***	**0.125 NS**	**0.716 *****
Qualitative method	1 ***	0.000 NS	0.009 NS	0.248 NS	0.086 NS	0.427 NS	0.368 NS	0.208 NS	1.000	0.432 NS	0.118 NS	0.155 NS
Quantitative method	-0.887 NS	0.014 NS	0.243 NS	0.576 **	0.587 **	0.218 NS	0.417 *	0.458 *	0.432 NS	1.000	-0.059 NS	0.623 **
Reactive Spray	-0.189 NS	0.484 *	0.007 NS	0.078 NS	0.030 NS	0.048 NS	0.014 NS	0.125 NS	0.118 NS	-0.059 NS	1.000	-0.194 NS
SCT	**-0.954 NS**	**-0.176 NS**	**0.433 ***	**0.880 *****	**0.855 *****	**-0.162 NS**	**0.472 ***	**0.716 *****	**0.155 NS**	**0.623 ****	**-0.194 NS**	**1.000**

Color code:	NS: Non significant	*: Significant at 5% risk	**: Significant at 1% risk	***: Significant at 0.1% risk

Finding a common scale

As stated above, the sole measurement result levels have an impact on the level of variability in the measurements. In addition, despite the given and shown correlations between the testing methods, the reading level (MSR) is totally different for each method, and it is nearly impossible to compare results and variation levels. Hence a suitable way of translating between the methods has to be found and fixed to compare both the results and their variability.

The easiest way for a comparison is to categorize the results from each method into the same categories (MSC), as e.g.:

- (as demanded by many): non-sticky and sticky,
- (as given from SCT): non-sticky, slightly sticky, sticky, strong stickiness, very strong stickiness.

There are two reasons, why this might not be the best solution:

- It is not possible to calculate (e.g. average, variation) between categorized results (unless by converting data in numeric values).
- As the spinning mills got their own experience on processability of sticky cotton with different levels depending on the methods they use, a continuous scale will be better for trade or spinning mill purposes.

For RT periodical reports and this publication, a first CommonScale(Max) scale expressed as a percentage has been developed since 2018 in order to match all methods results from different instruments. This means that any actual reading is divided by a reference value, which is at this stage the maximum reading possible by the given method. Therefore, all individual data points can be converted in a CommonScale(Max) value between 0 and 100. With this calculation method, the individual results for each testing method (MSR) can be transformed into MSS results, which are then suitable for all testing methods.

With the help of the CommonScale(Max), all data of one RT can be provided in one single graph, and all methods or instruments can be compared to the other ones. In the case of RT2019-2 (Figure 12, https://www.itmf.org/committees/international-committee-on-cotton-testing-methods) it can be easily seen that:

- Some laboratories provided results which are very far from the 'global mean per cotton' (bold dotted lines): sometimes with a systematic deviation toward low or high CommonScale(Max) values, sometimes for some levels only in the stickiness range provided by the choice of the participating cottons; sometimes in an erratic manner; sometimes with a conjunction of the preceding observation.
- Some laboratories provided very variable results for some cottons.
- The periodic RT reports provide all details contributing to these data interpretations.

Another solution for a CommonScale could be to use the regression relationships between the testing methods results in order to calculate the slopes and offsets from the correlations to a joint CommonScale(Cor) level. This requires an exhaustive study of all advantages and inconveniences for each calculation or comparison technique, on output being the choice of the best indicator for every day operations.

Independently, whatever kind of common scale used, it is important to fix the translation technique between these MSR levels based on a suitable number of round trials with a suitable number of participants for each chosen method. Hence, stickiness round trials will continue, including all laboratories that are interested in participating, and using results to come to the best possible definition of a CommonScale or MSS for every commercial stickiness testing method, might it be the currently given methods or other to be developed from now on.

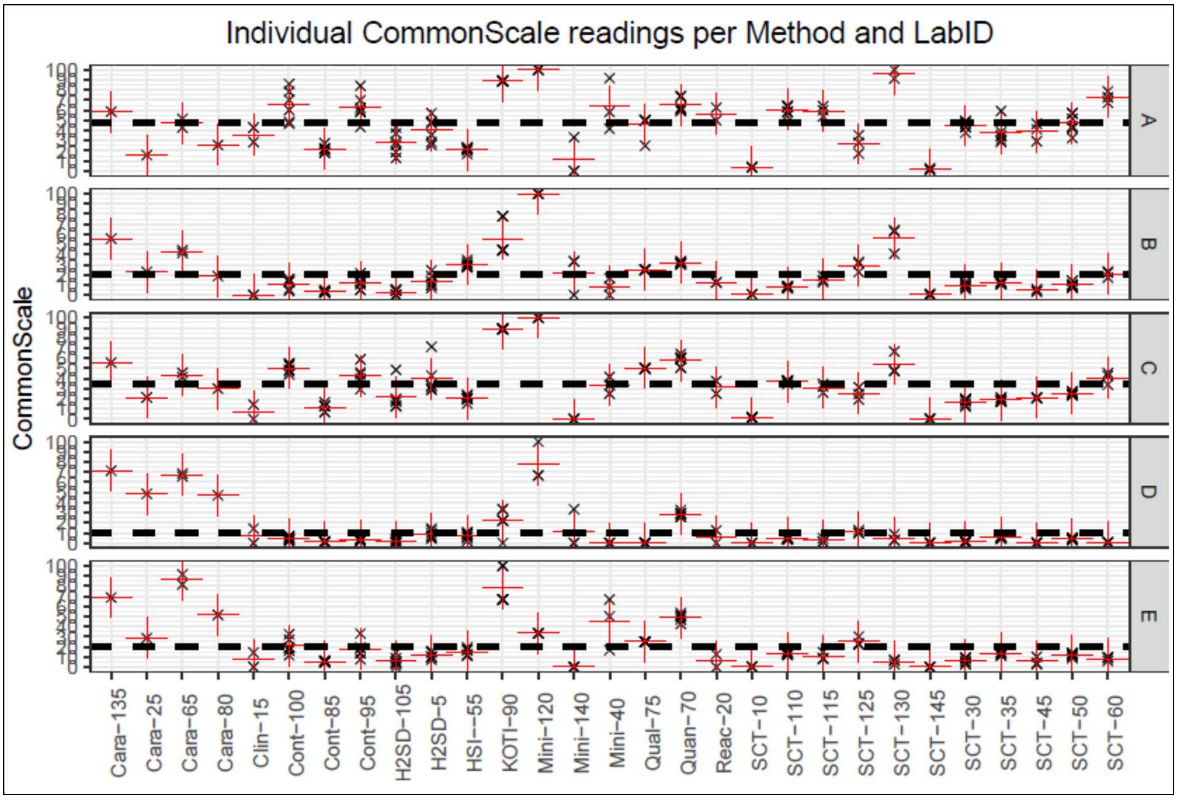

Figure 12: Extract form the RT2019-2 report: MSS CommonScale(Max) results with all methods involved for five cottons A to E by all participating laboratories. From left to right, data ordered by Method and LabID. The bold dotted lines represent the mean value of all readings per cotton A to E.

Proposed suitable harmonization steps, tools and measures for the future

If measurement results of stickiness have to be used widely in trade, then a comparable approach to what has been done with other fibre testing methods or specifically other fibre instrumental testing results over a period of 70 years is to be applied, as it has been proven to be efficient. Harmonization has generally included:

- Setting up and the acceptance of definitions,
- Technical and technological developments of the testing methods, including sampling, testing, and data management,
- Basic research for analysing the sensitivity and influences on the testing methods,
- Creation of reference materials to check and calibrate measuring devices,
- Creation of standard testing methods for international acceptance,
- Comparisons between the different testing and reference methods,
- Organization of round-tests such as the USDA, Bremen or ICAC-CSITC-RTs,
- Evaluation of the findings in international committees as the ITMF-ICCTM or ICAC-CSITC,
- Technical and non-technical discussions on the topic with researchers, people from practice and from additional sides,
- Application of the methods and according decisions in the laboratories at all levels in the supply chain, including in Cotton Association or Boards.

However, at first glance, if this approach was possible for fibre characterizations, they may, although already done for many steps, create some big challenges that have to be faced in the case of stickiness:

- Measurement of stickiness is difficult due to the great diversity in the dispersion of honeydew droplets within the raw material, and the ability of any sampling method to grab a representative contaminated material piece has to tackle this difficulty;
- Measurement of stickiness has to be assured by as reproducible as possible methods which should really measure stickiness as defined as “the propensity of honeydew contaminated fibres to stick to spinning parts during their processing”,
- Creation of realistic and suitable stickiness reference material might be difficult. However, an attempt is under study together with the ‘calibration’ procedures that have to accompany testing procedures.

Important topics to be tackled and suitable activities for the future will then be:

- Continue supporting laboratories in comparing their results to other laboratories and hence reducing inter-laboratory variations,
  - ➔ Continuation of the stickiness round trials,
  - ➔ Focusing on the chosen methods (mechanical / thermo-mechanical methods with SCT, H2SD and Contest-S, keeping the Minicard as the reference) for the harmonization efforts, but including other methods in round-trials,
  - ➔ Include more laboratories in order to have an even more grounded proof,
  - ➔ Develop an “easy to understand and to use indicator” for the laboratories to see their deviations and their need for action.
- Analyse the sources of result variabilities and different impacts on the given and new testing methods to harmonize the testing methods,
- One example is to study the impact of honeydew points with their number and their size on the test results and on SIP. Indeed, are the various methods predicting SIP suitably? Only very expensive additional spinning tests can bring the answer, assuming that the source of homogeneous sticky cottons in a full range of stickiness are available without limit.

- Develop a “stickiness testing guideline”, comparable to the ITMF/CSITC Instrument Testing Guideline where best conditions for the use of stickiness results will be developed.
- Develop CommonScale definitions, finally choose the most suitable definition and set the according parameters,
  - ➔ With this, the results of all given commercial testing methods and any new methods that will be developed will be able to express or translate their results into a same scale related to SIP.
  - ➔ Develop a system to maintain this CommonScale.
- Assure the relationship between test results (MSR, MSS) and Stickiness in Practice (SIP) results,
  - ➔ All RT cottons are currently being spun in the same conditions as for the RT in 2013-14 for checking the direct relationship between methods and instruments results to Stickiness in Practice for all methods. This would insure that that the prediction relationship between measuring methods results and stickiness (MSR) as observed in spinning (SIP) is maintained, based on monitored yarn quality and productivity parameters.
  - ➔ Develop a MSC categorization only based on MSS that is suitable for trading together with proposals for suitable trade rules.

An attempt is currently made to produce reference materials and to see the effect of their use in the stabilization of results between instruments of single methods at first. From the beginning, it will be necessary to create more of these reference materials taking care of the types of insect producing the honeydew. At this point, it is not planned to produce “artificial sticky reference materials” as it was demonstrated that insect sugars are very difficult to reproduce now.

Finally, users of stickiness measuring methods have to remind that stickiness results will never be with a low level of variability, especially when expressed in CV% for between instruments, between methods, or between cottons comparisons.

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The completion of this work would not have been possible without all providers of sticky fibres at various degrees to feed this Round Test; they will always be anonymous, but their help is precious.

Finally, our special thanks go to Serge Lassus, Samira Ainouch and Sofian Dejardin from CIRAD for their support in testing all fibre samples for allowing the preparation of this document.

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Steps towards suitable stickiness test results for trading and processing

Jean-Paul Gourlot *, Axel Drieling **

* CIRAD (France), ** FIBRE (Germany)

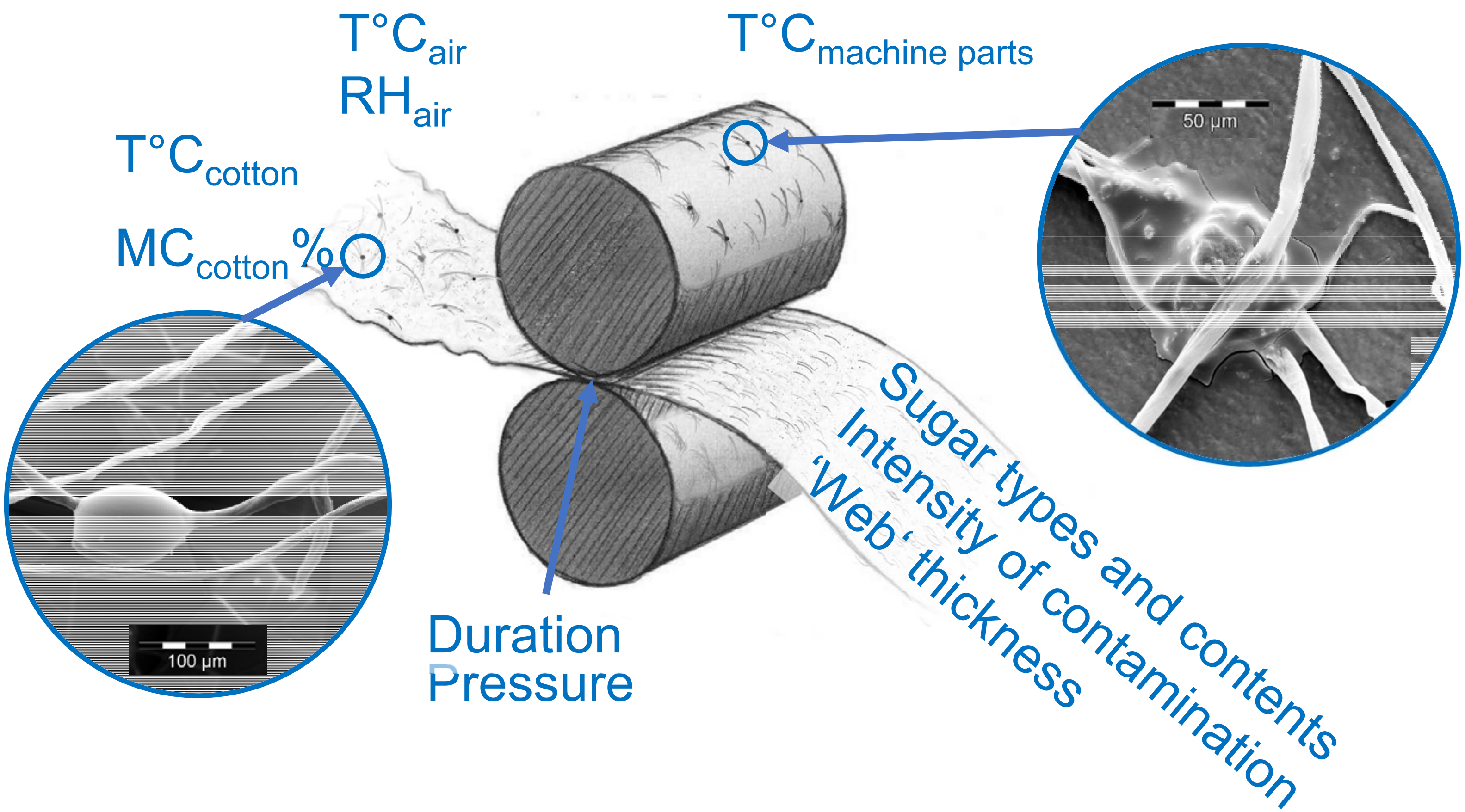
Stickiness:
what is it, what are the incidences? (1/3)

- Deposits from insect honeydew mainly onto fibers; composed by several individual sugars
- Fibers + honeydew stick on machine parts such as cylinders at spinning with yarn quality (un-evenness) and productivity (lower turnout) incidences



Pictures by Cirad

Stickiness:
what is it, what are the incidences? (2/3)



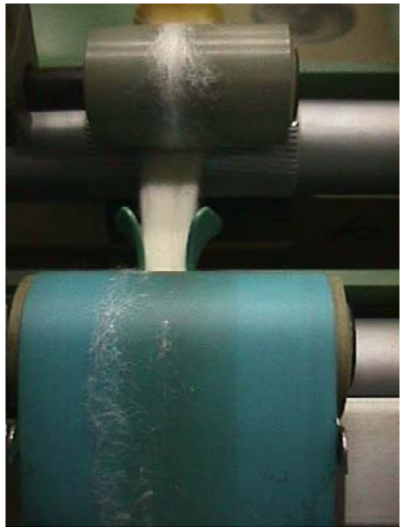


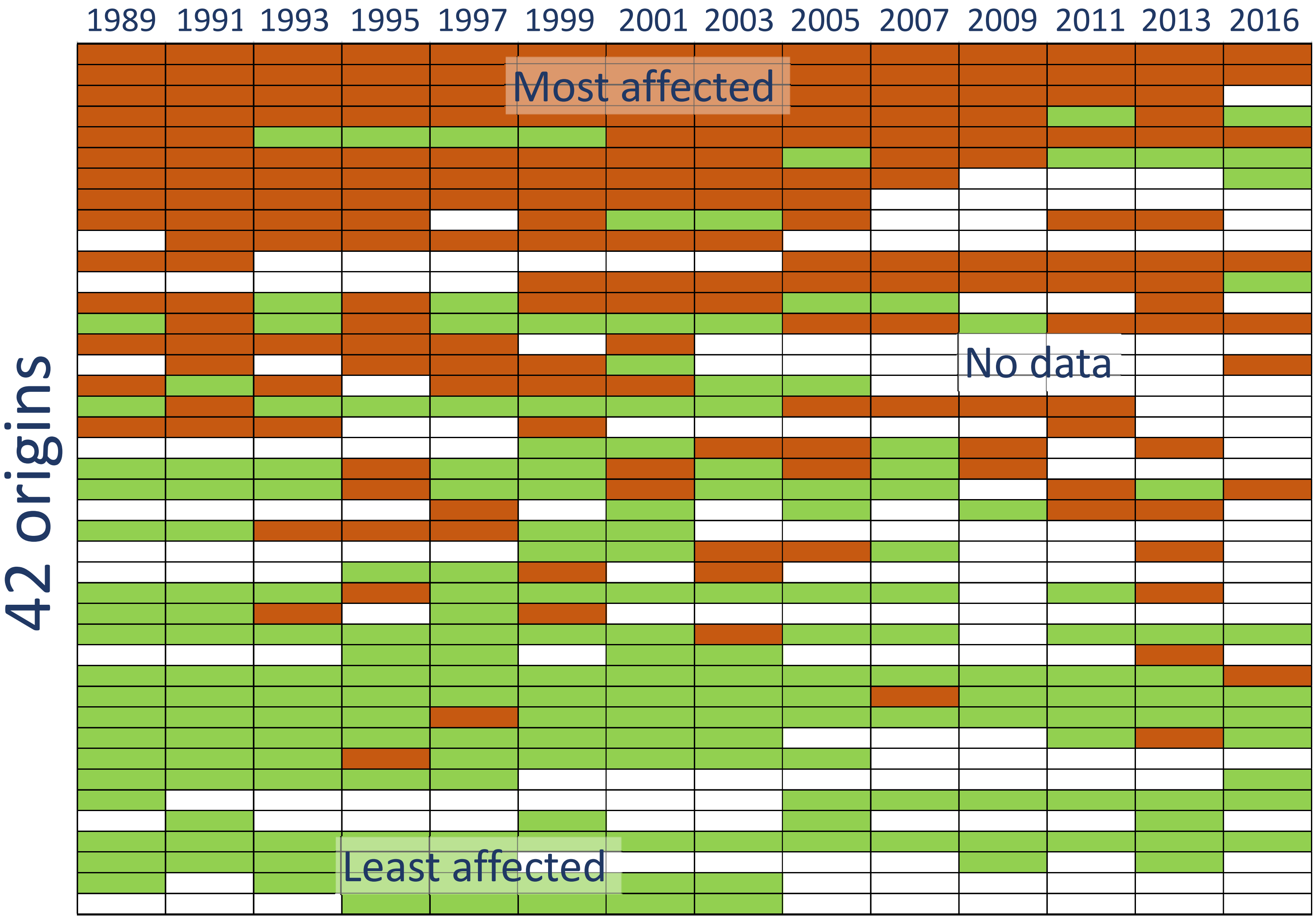
Illustration by Lena Kölsch, FIBRE

Stickiness:
what is it, what are the incidences? (3/3)

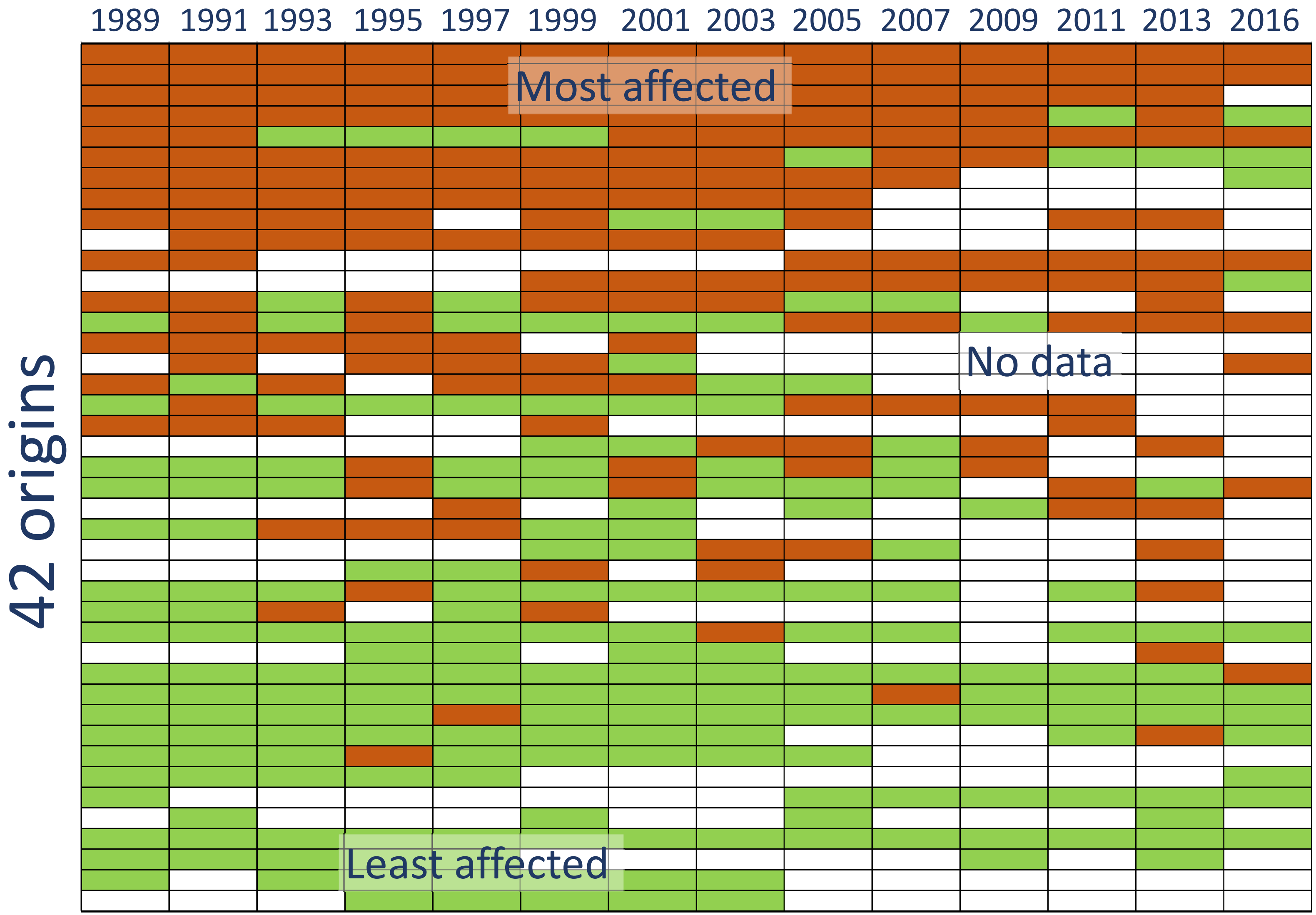
- Fibers + honeydew stick on machine parts such as cylinders at spinning with yarn quality (un-evenness) and productivity (lower turnout) incidences
- Economical incidences (claims, discounts, reputation)
- Solutions exist
  - Choose cottons
  - Blend origins
  - Change spinning mills conditions

➔**Need reliable measurement** (technical and trade uses)

ITMF Contamination Surveys over time

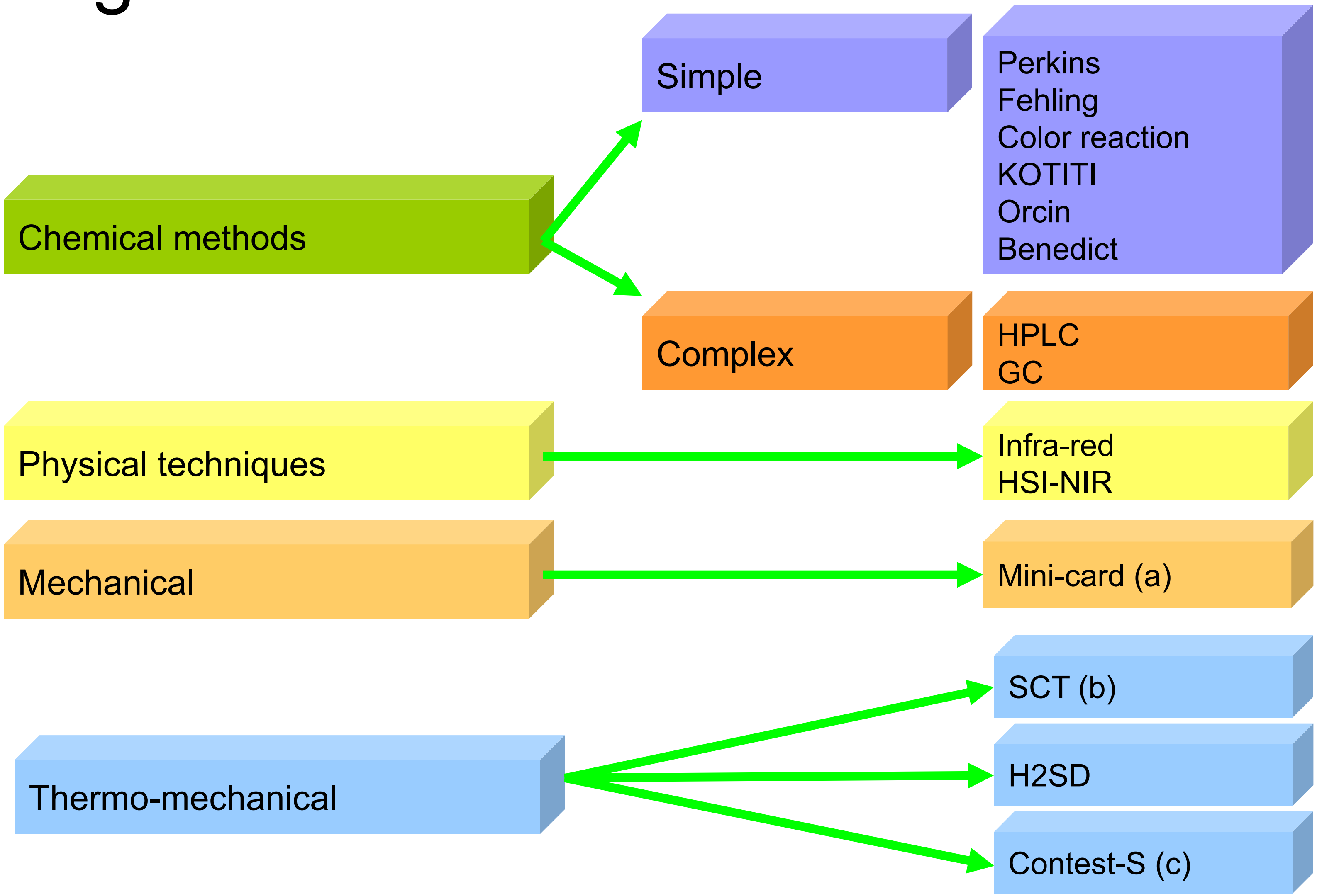


ITMF Contamination Surveys over time



Stickiness: appreciations about origins not permanent nor stable

Existing methods



(a) ITMF Reference method (b) ITMF Recommended method (c) ITMF Recognized method

Stickiness: Evaluation and measurement
Harmonization of results

Our aims

Show the variations and their causes

Harmonize between labs based on RTs including various methods

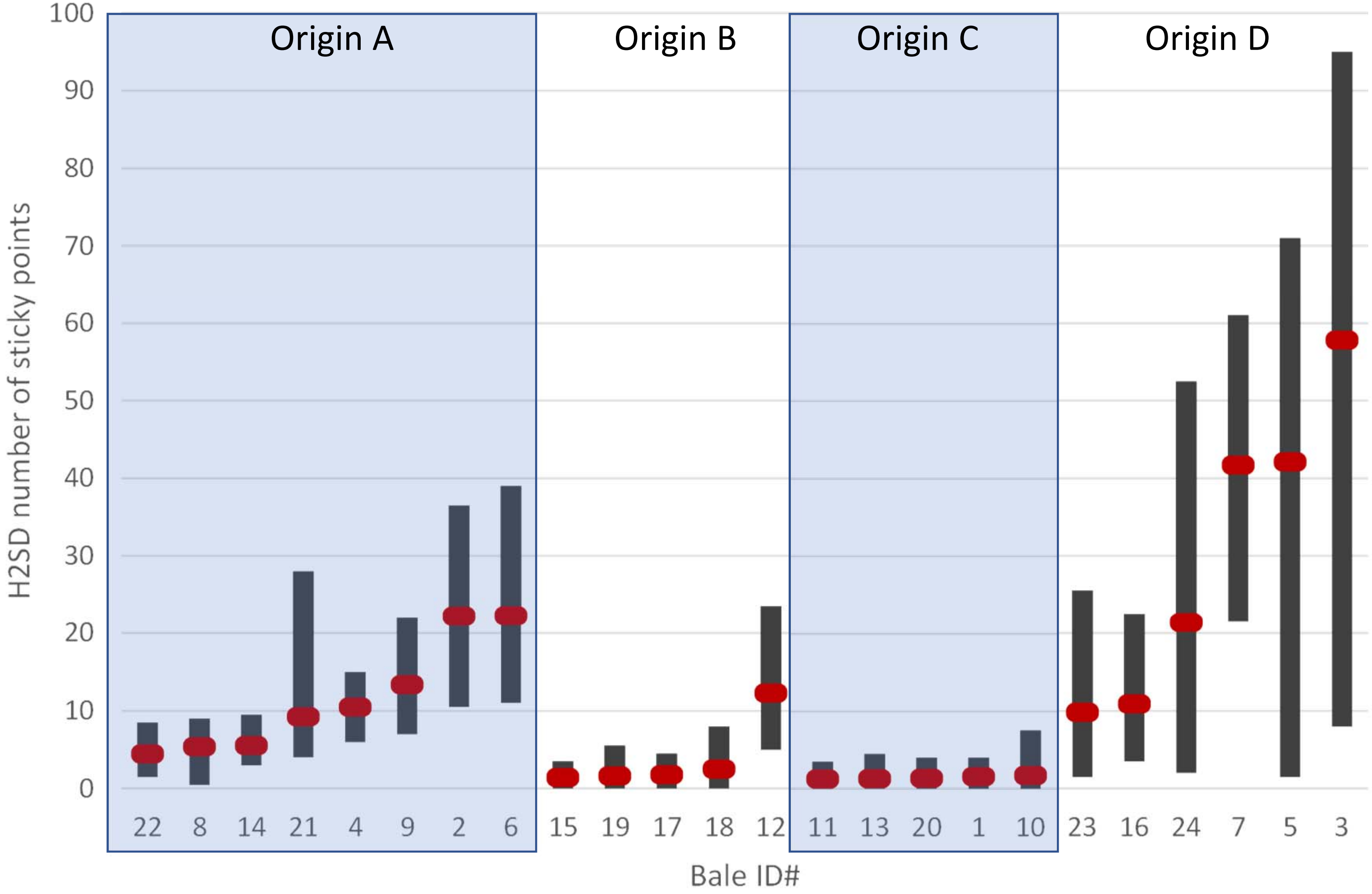
Choose methods based on

- Best correlations to SIP (stickiness in practice)
- Good correlations to each other

Allow comparisons between instruments and between methods

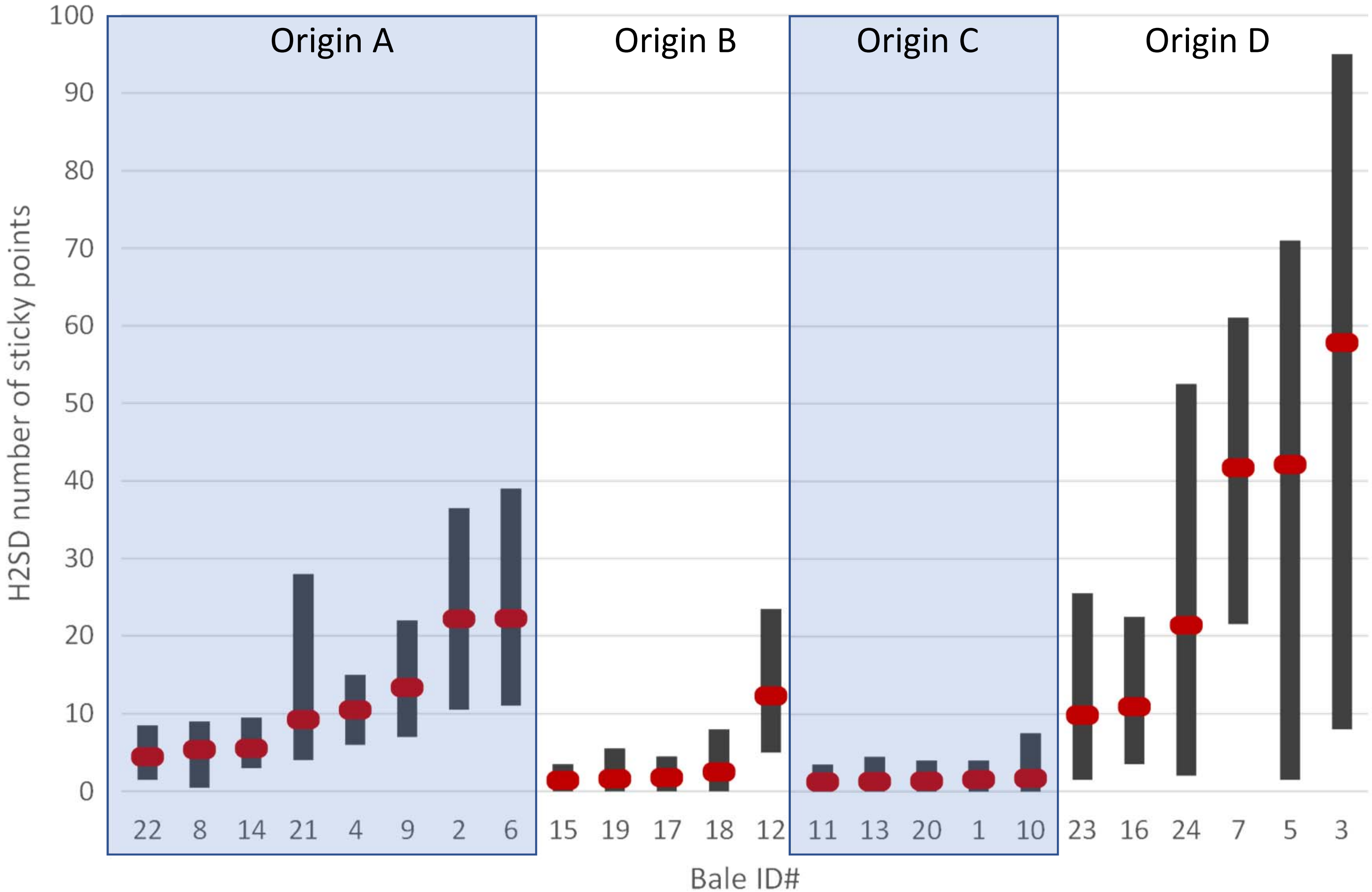
Propose future harmonization steps

Distribution of stickiness within bales



Min, Max and mean numbers of H2SD sticky points
(32 samples per bale, 24 bales from various origins. (Frydrych et al. 2004).

Distribution of stickiness within bales



Min, Max and mean numbers of H2SD sticky points
(32 samples per bale, 24 bales from various origins. (Frydrych et al. 2004).

Extreme variation even within bales
→ Difficulty to get representative samples

Stickiness: various predictive levels between results and SIP (Stickiness in practice)

Micro-ring-spinning

11 cottons

20 tex (Ne 30 or Nm 50)

23°C, 58% R.H.

Yarn productivity (8) and quality (24) parameters recorded

	Others	Card	H2SD	SCT
Productivity (max=8)	2 to 6	7	6	6
Quality (max=28)	17 to 22	22	22	22
Percent of significant relationships **(α=5%)** **Yarn = f (Fiber)**	**58 to 67**	**81**	**78**	**78**

Others are: Caramelization, Chemcare, Kotiti

Gourlot et al, ITMF-ICCTM, 2016

Stickiness: various predictive levels between results and SIP (Stickiness in practice)

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Mechanical and thermo-mechanical methods show the most significant relationships with SIP

Gourlot et al, ITMF-ICCTM, 2016

cirad AGRICULTURAL RESEARCH FOR DEVELOPMENT

UNIVERSITÄT BREMEN FASERINSTITUT FIBRE BREMEN

Observations on variations in round-tests

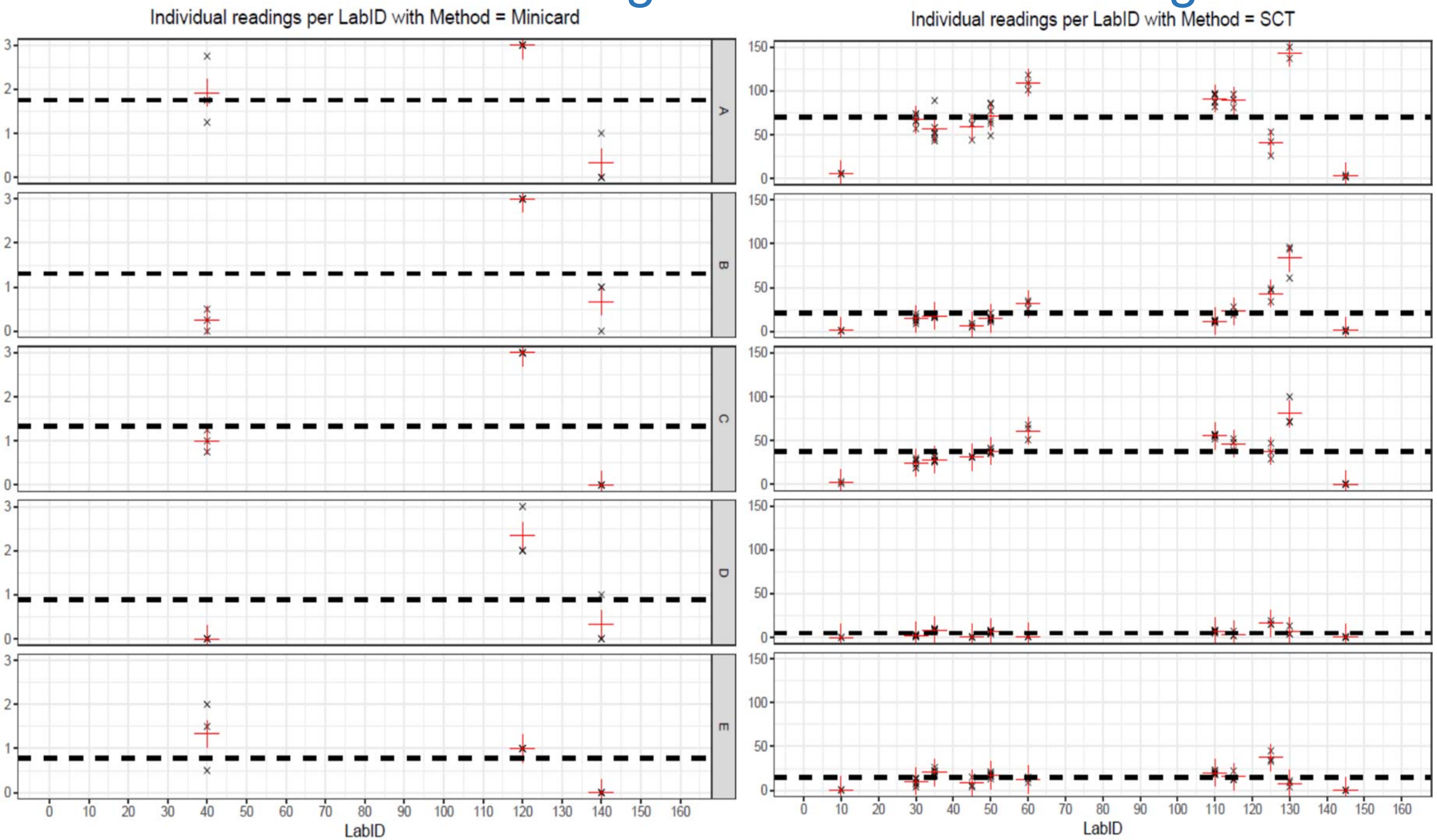
1. Effect of the reading levels for each testing method
2. (Effect of the natural variability of stickiness)
3. Effect of the material preparation
4. Effect of sampling of any material into several samples
5. Effect of the measurement result levels on the level of variability in measurements
6. Finding a common scale to report results
7. Variability in stickiness results with one material along RTs
8. Correlations between methods

Observations on variations in round-tests

1. Effect of the reading levels for each testing method
    - 2 RT / year since 2017
    - 3 to 5 cottons / RT covering a stickiness range
    - 10-12 methods used by 25-35 labs
    - 1 to 6 results per instrument and cotton

Observations on variations in round-tests

1. Effect of the reading levels for each testing method



Observations on variations in round-tests

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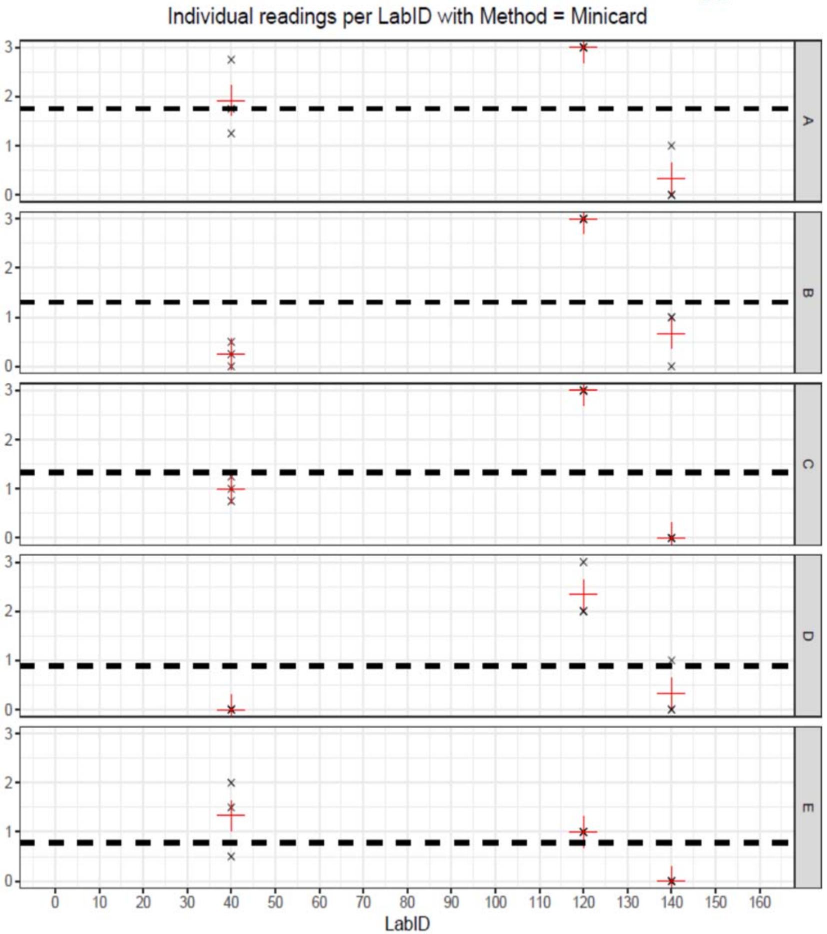
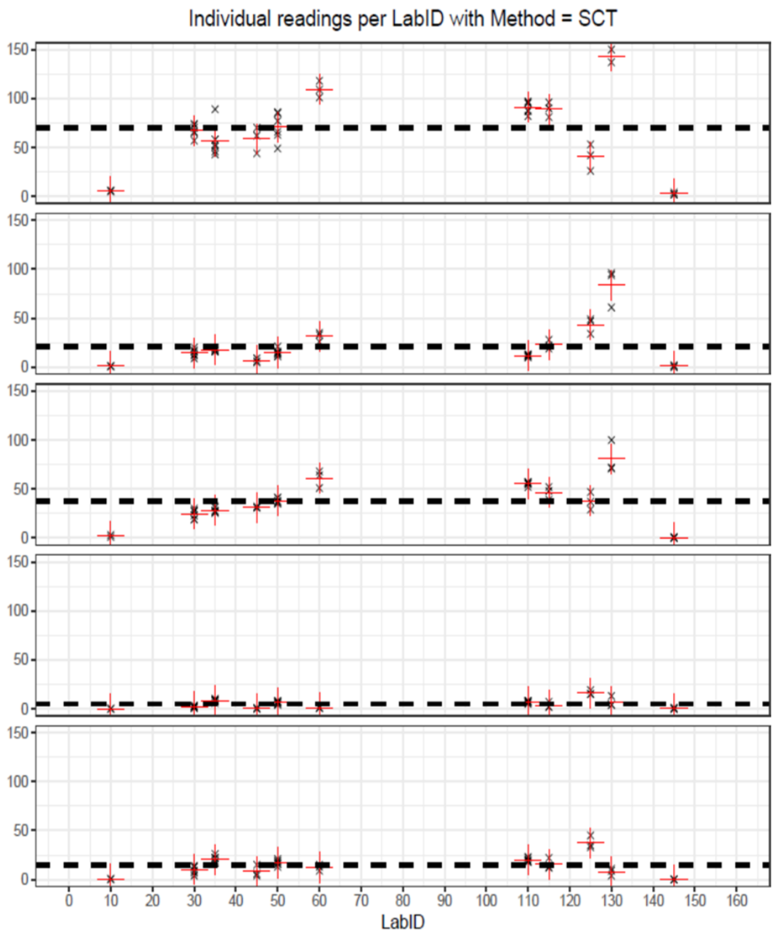




Easy to compare instrument variations within each method

- within lab.
- between labs.

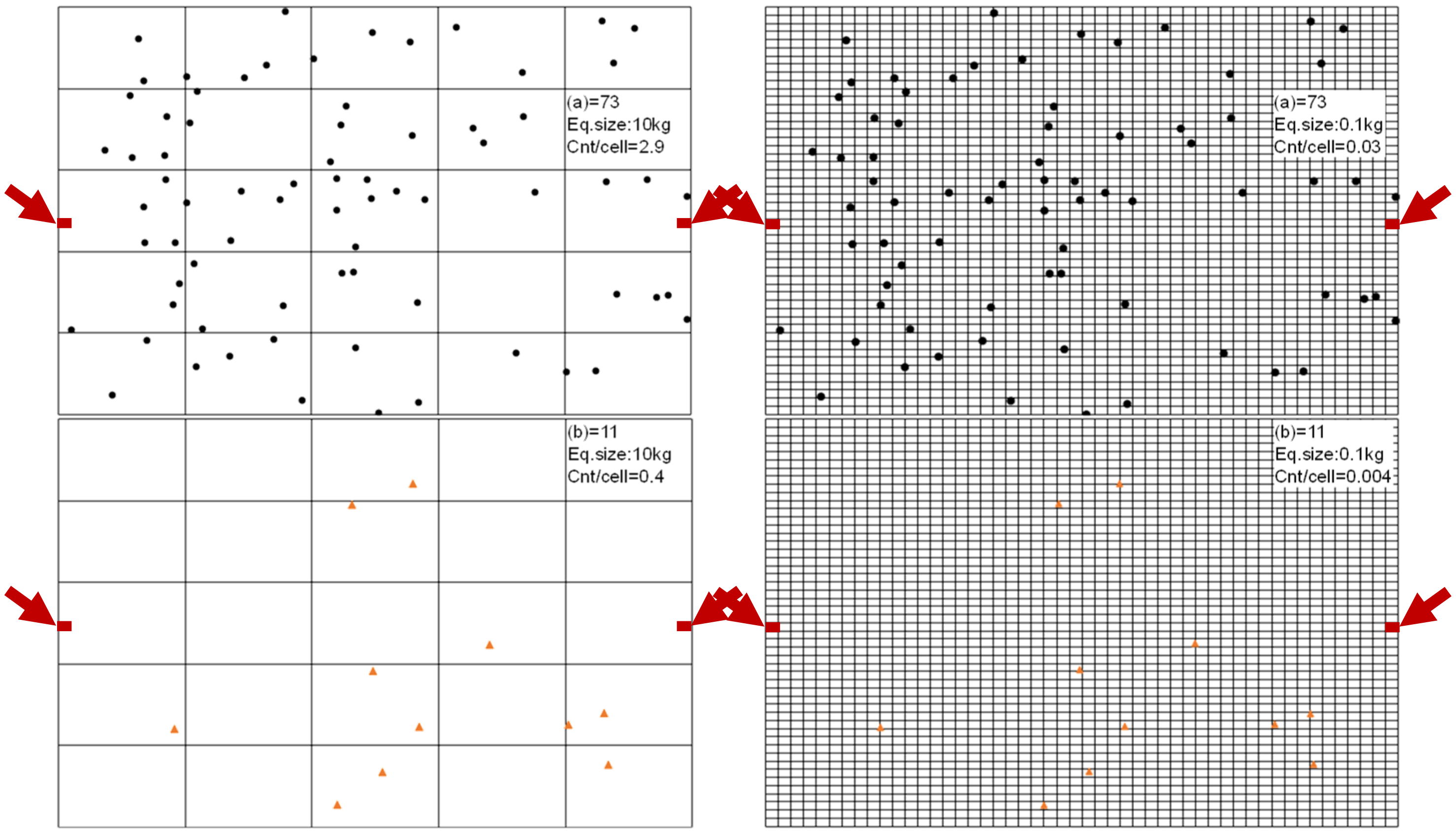
→ Labs improve

→ Best practices guide needed

Difficult to compare methods

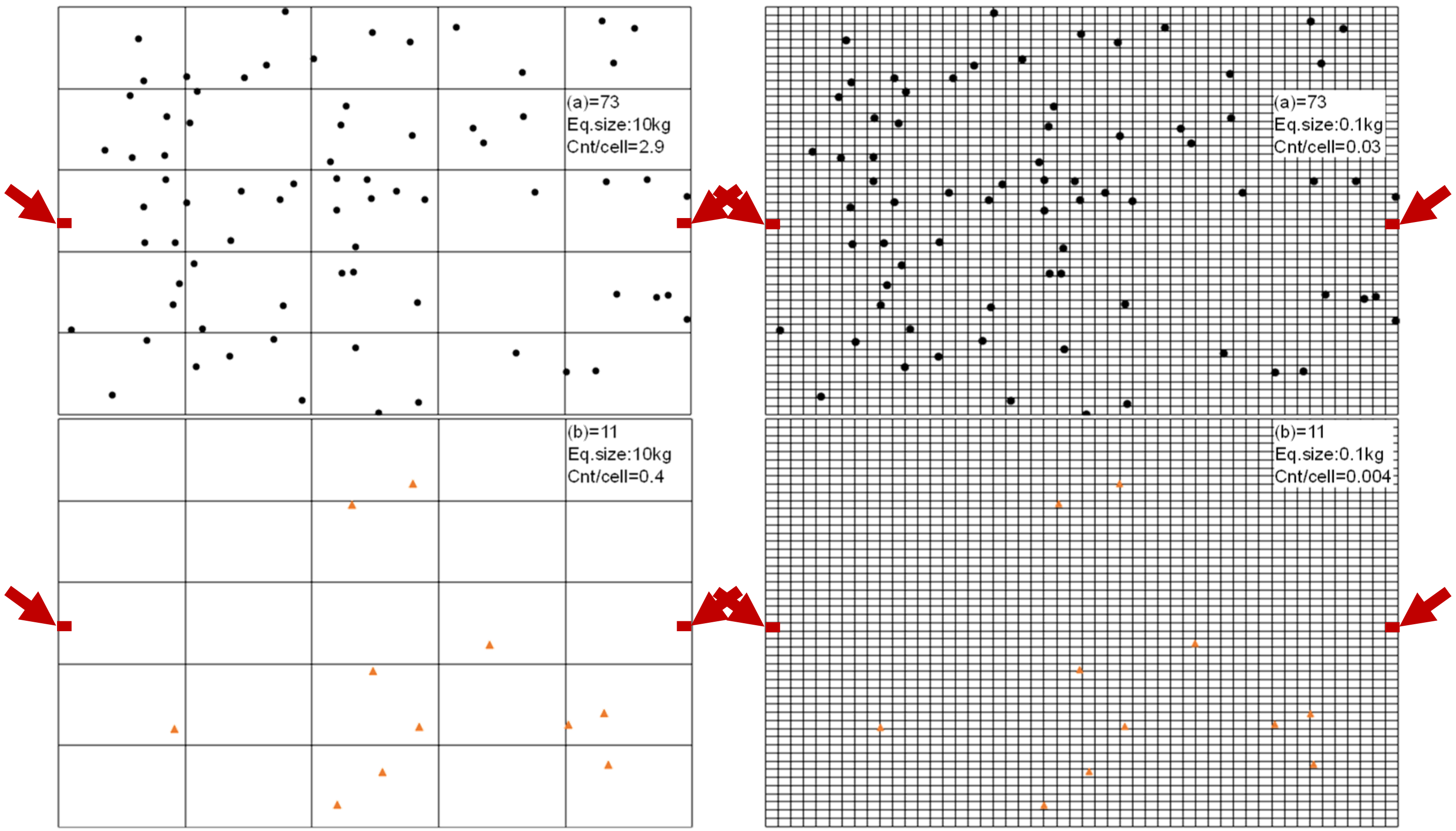
Observations on variations in round-tests

2. Effect of the natural variability of stickiness



Observations on variations in round-tests

2. Effect of the natural variability of stickiness

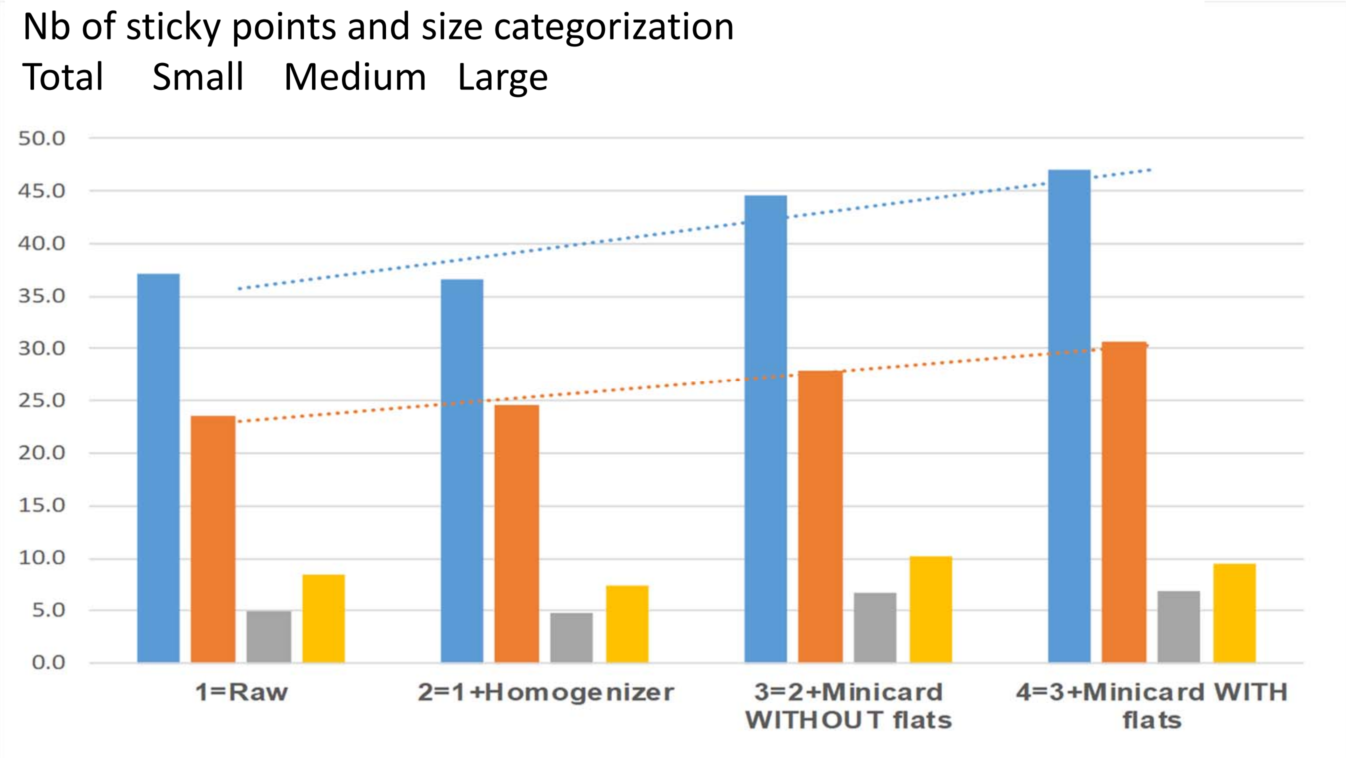


Honeydew distributed in bales
Probability to find this honeydew in sample is quite low

Observations on variations in round-tests

3. Effect of the material preparation

- 4 accumulative ways to 'prepare' the material
- H2SD counting



Observations on variations in round-tests

3. Effect of the material preparation

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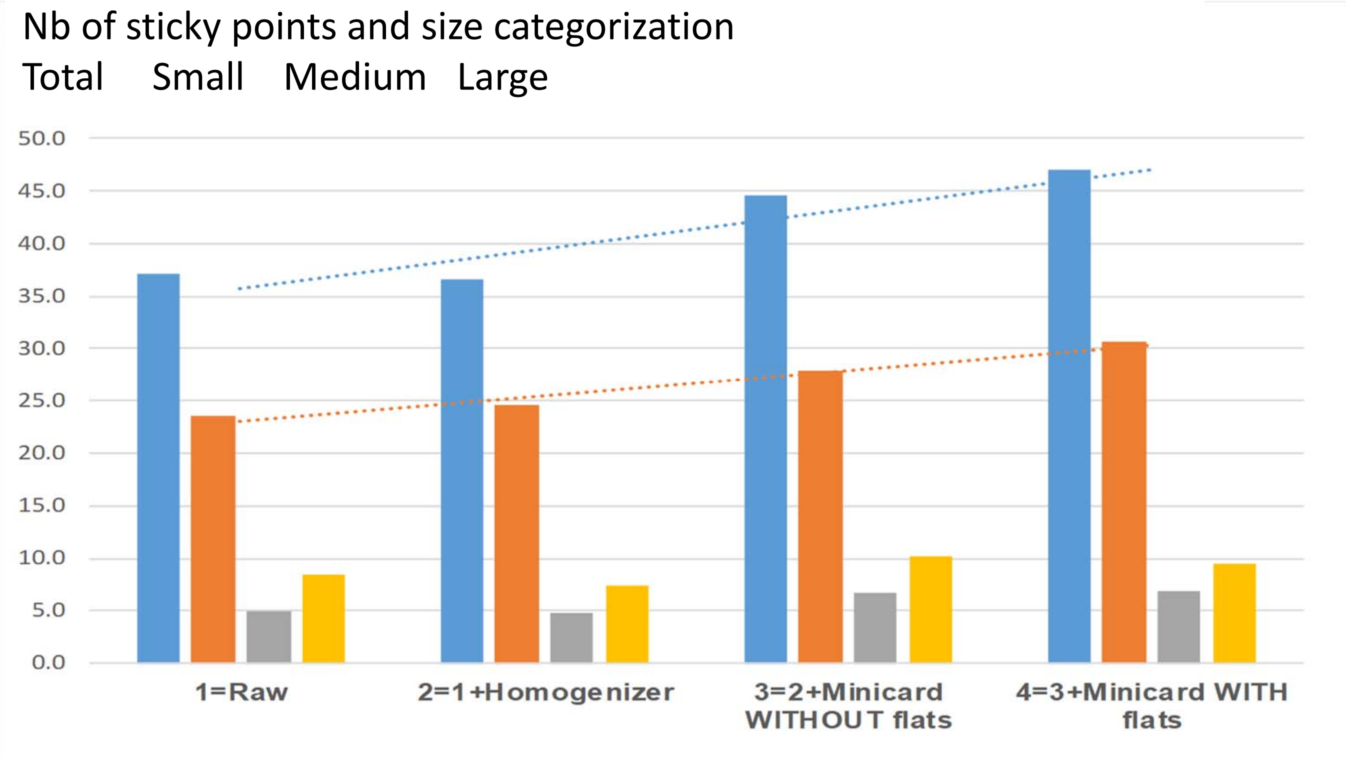


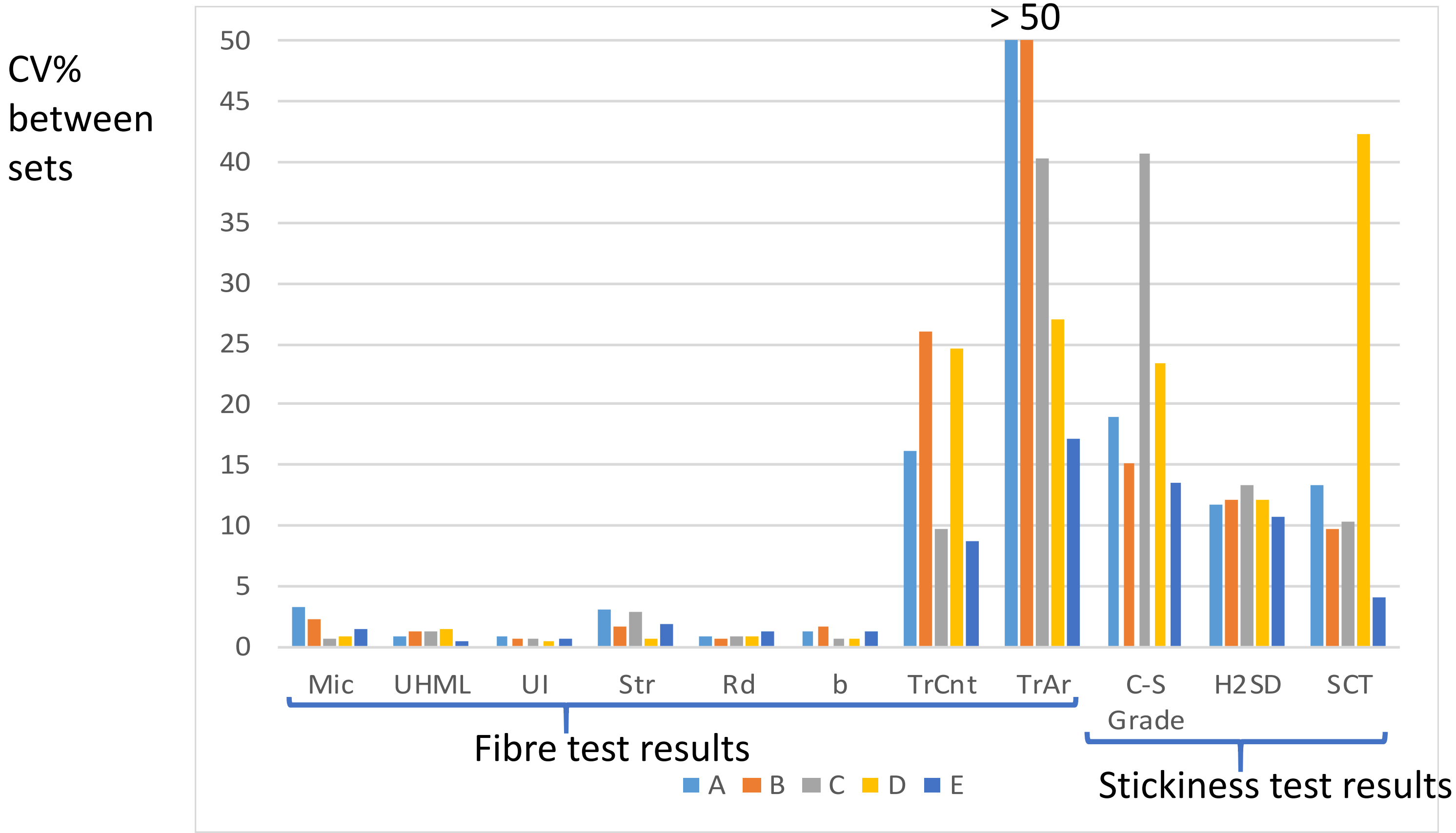
Impact of blending on number and size of sticky points
→Keep homogenizer for next RTs

Observations on variations in round-tests

4. Effect of sampling of any material into several samples
   - Aim: checking if materials are properly homogenized: observation of variations between sets of samples
   - Special sets of samples for fiber testing in addition to stickiness testing

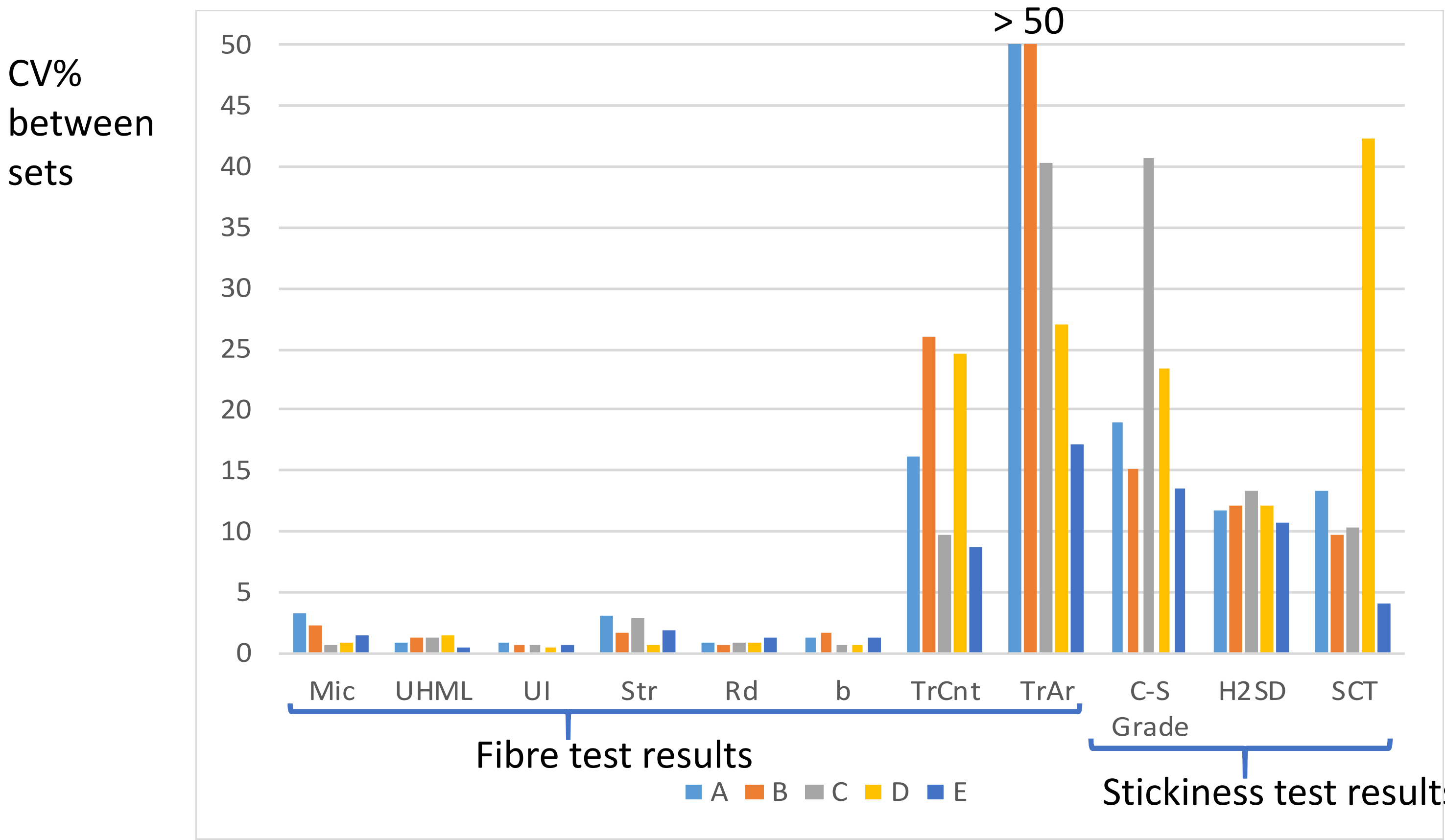
Observations on variations in round-tests

4. Effect of sampling a material into several samples



Observations on variations in round-tests

4. Effect of sampling a material into several samples



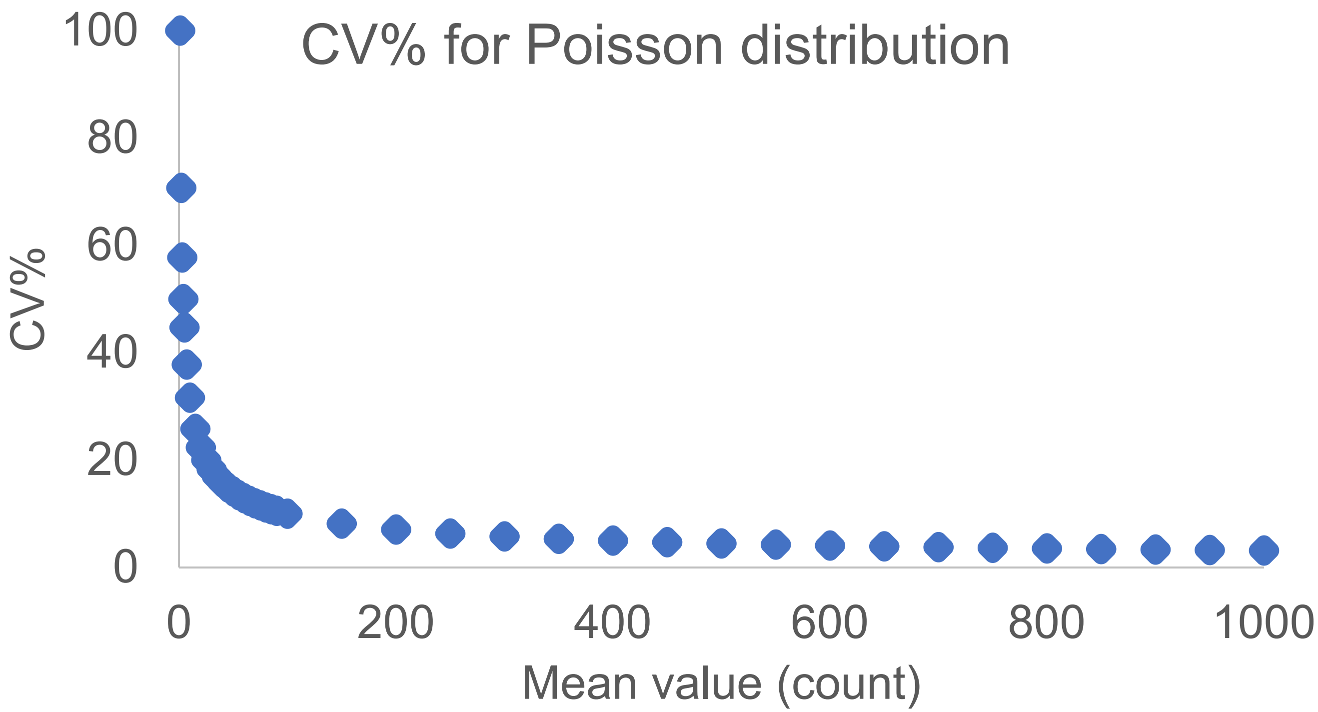
Usual fiber results CV% are low

- → homogenization is good
- → Keep the homogenizer for next RTs

Comparable CV% for Trash and stickiness

Observations on variations in round-tests

5. Effect of the measurement result levels on the level of variability in measurements



Observations on variations in round-tests

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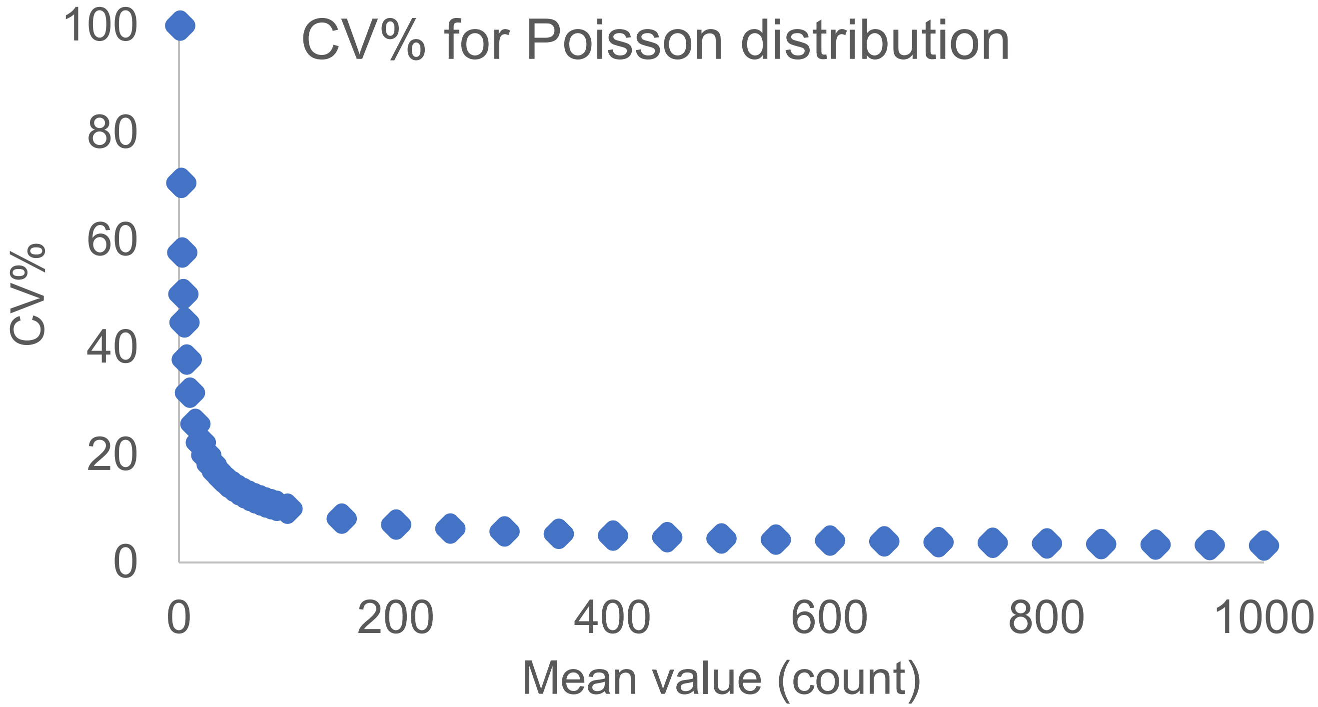


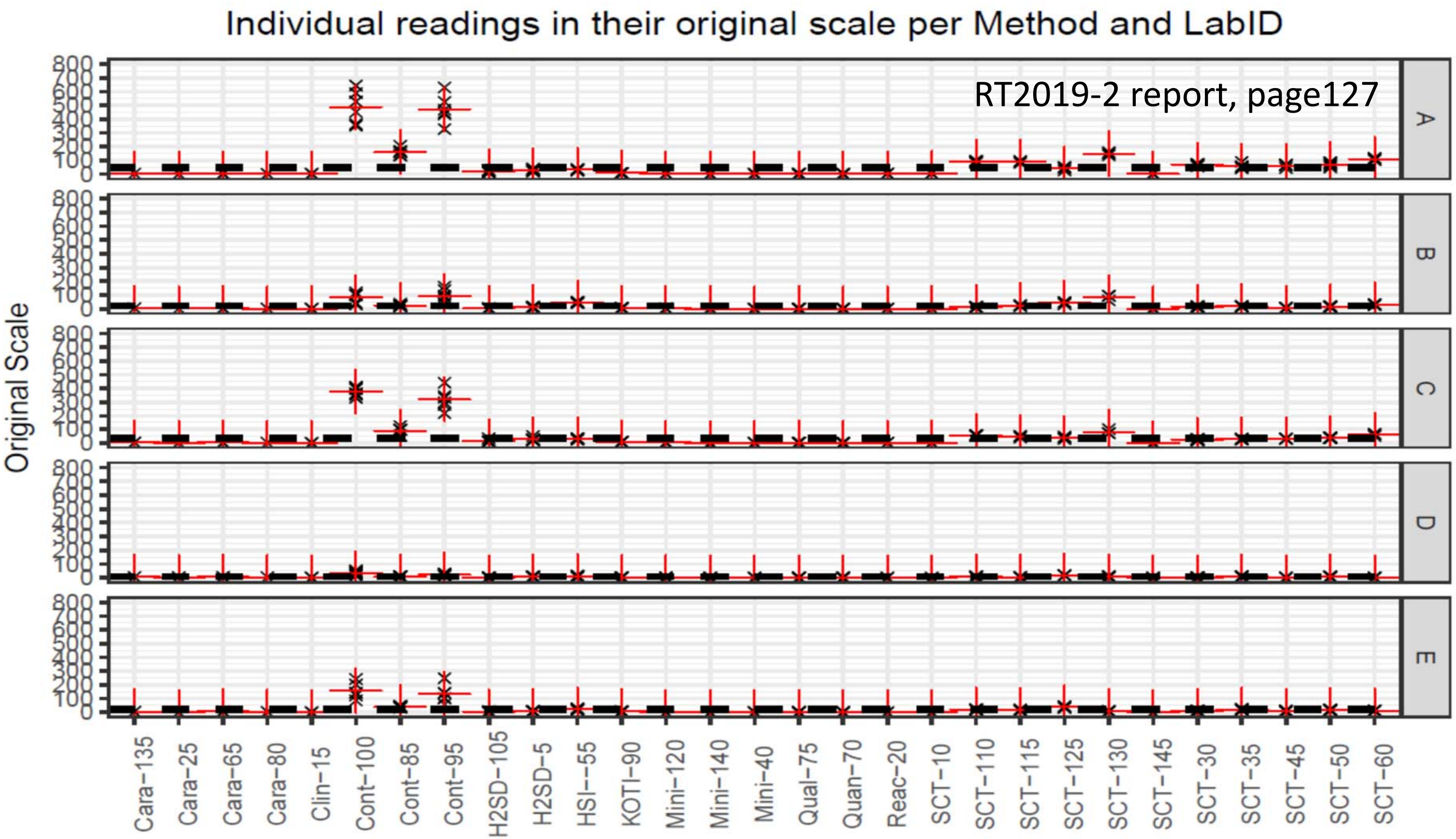
CV% changes with mean value

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

→ Need to look for better indicator

Observations on variations in round-tests

6. Finding a common scale



Observations on variations in round-tests

6. Finding a common scale
   - Aim: Ease the comparison between methods

=>CommonScale(Max) has been developed as

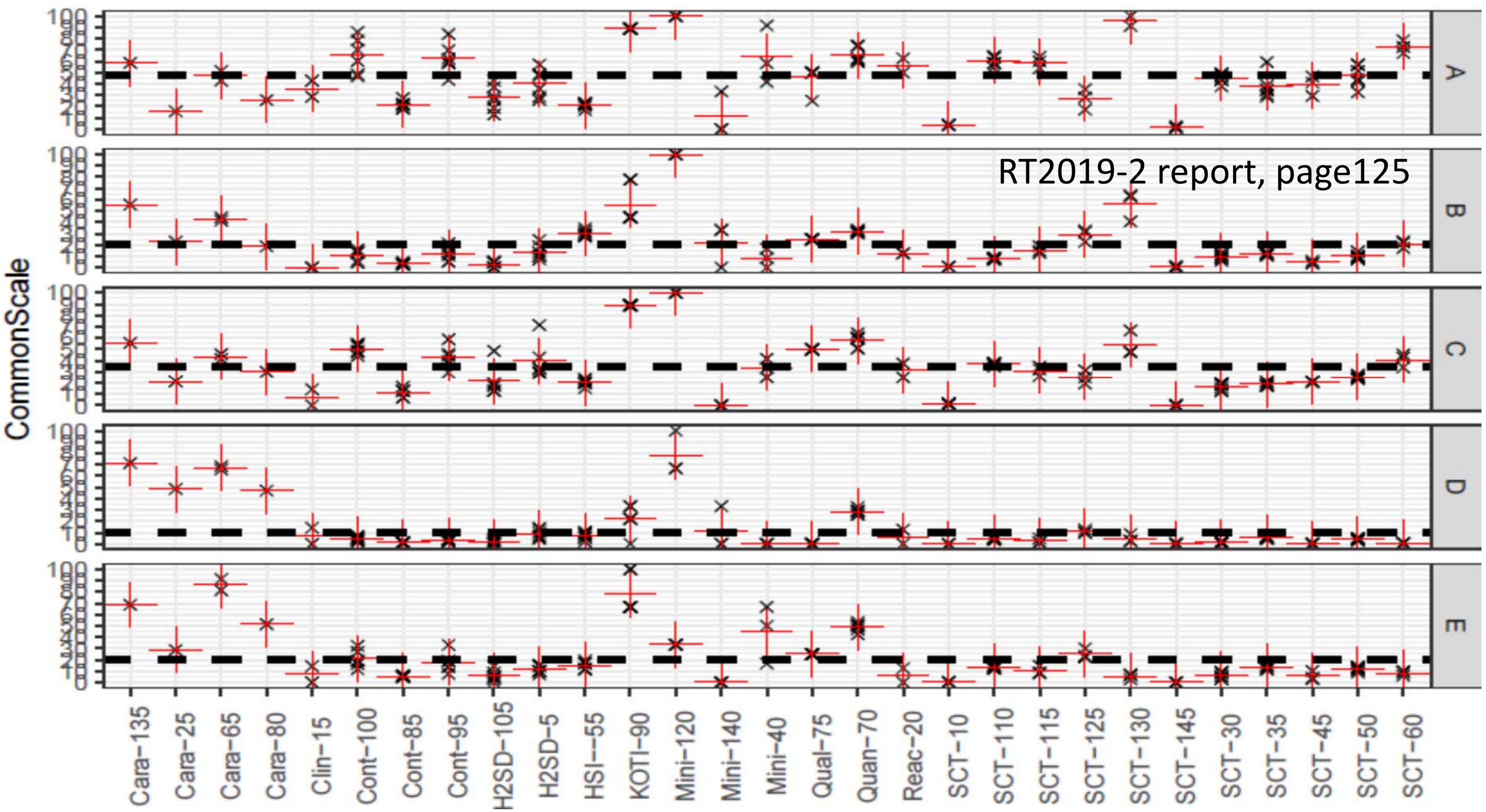
$$\frac{\text{Measured Stickiness Raw" (MSR) * 100}}{\text{MaxEver}} = \text{Measured Stickiness Scaled (MSS)}$$

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

Observations on variations in round-tests

6. Finding a common scale

Individual CommonScale readings per Method and LabID



Observations on variations in round-tests

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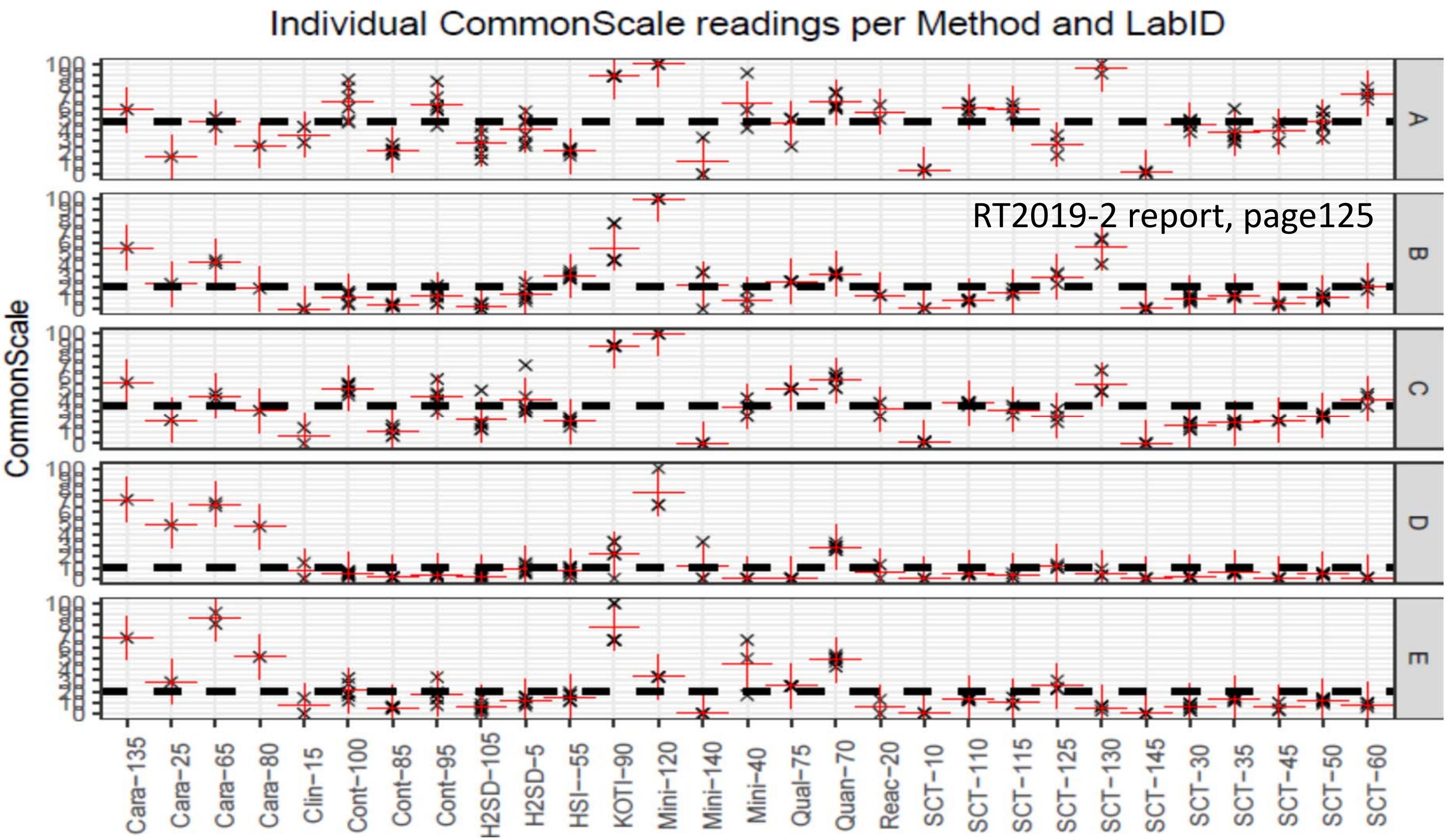


Easy to compare methods and instruments

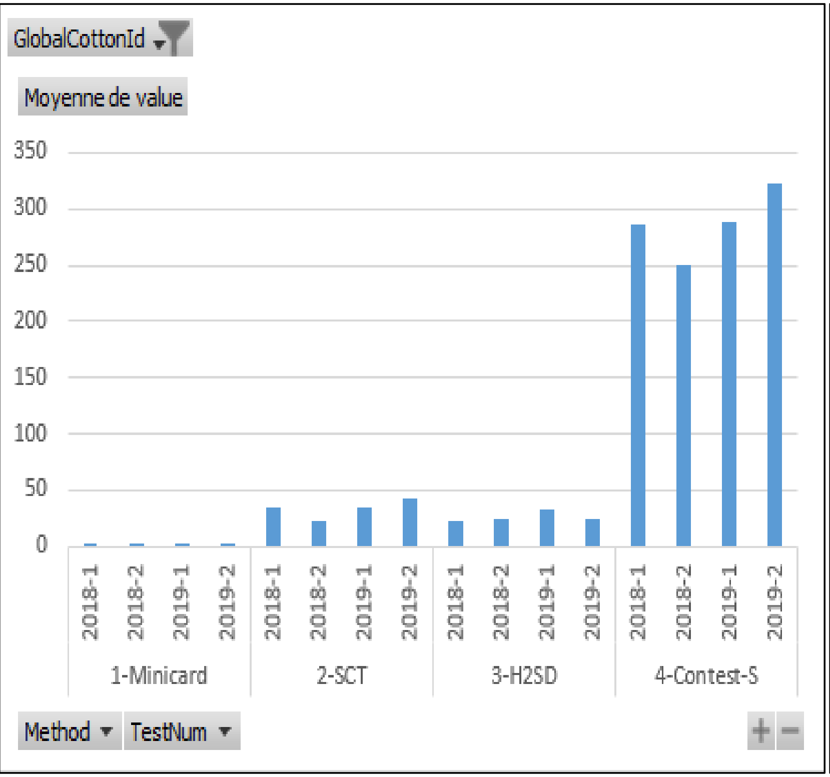
Easy to check/compare stickiness in cottons

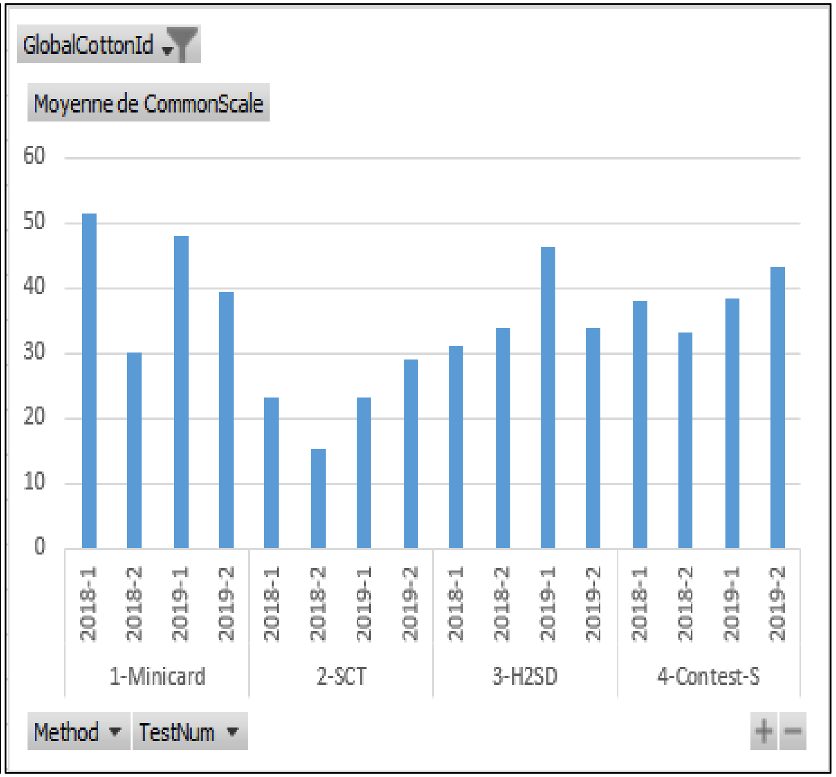
Observations on variations in round-tests

7. Variability in stickiness results with one material along RTs
   - Single instruments: mini-card, Contest-S, H2SD, SCT
   - One material
   - Four RTs: 2018-1, 2018-2, 2019-1 and 2019-2

Observations on variations in round-tests

7. Variability in stickiness results with one material along RTs





Observations on variations in round-tests

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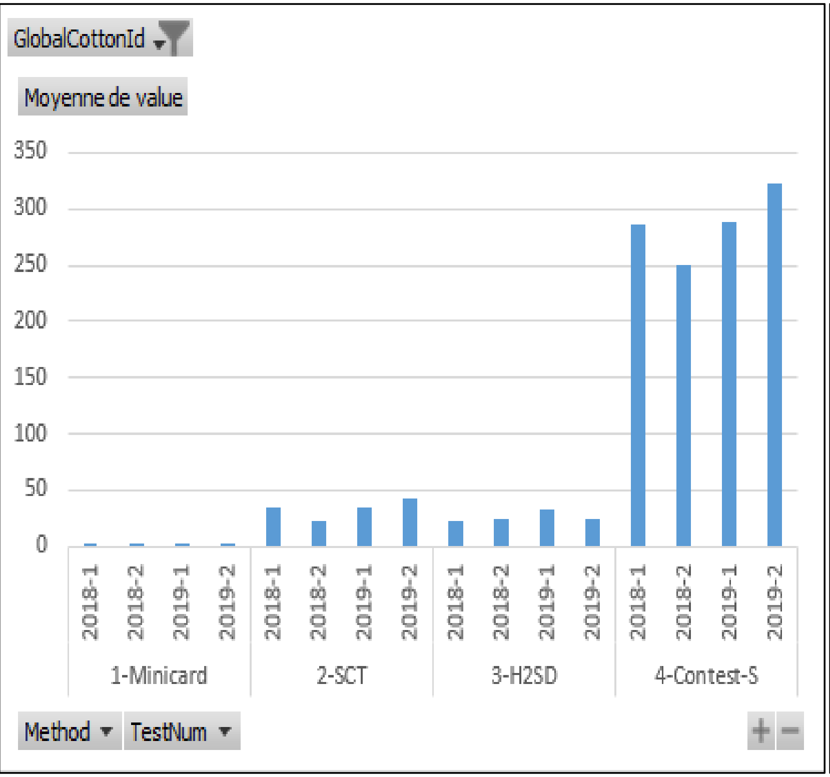

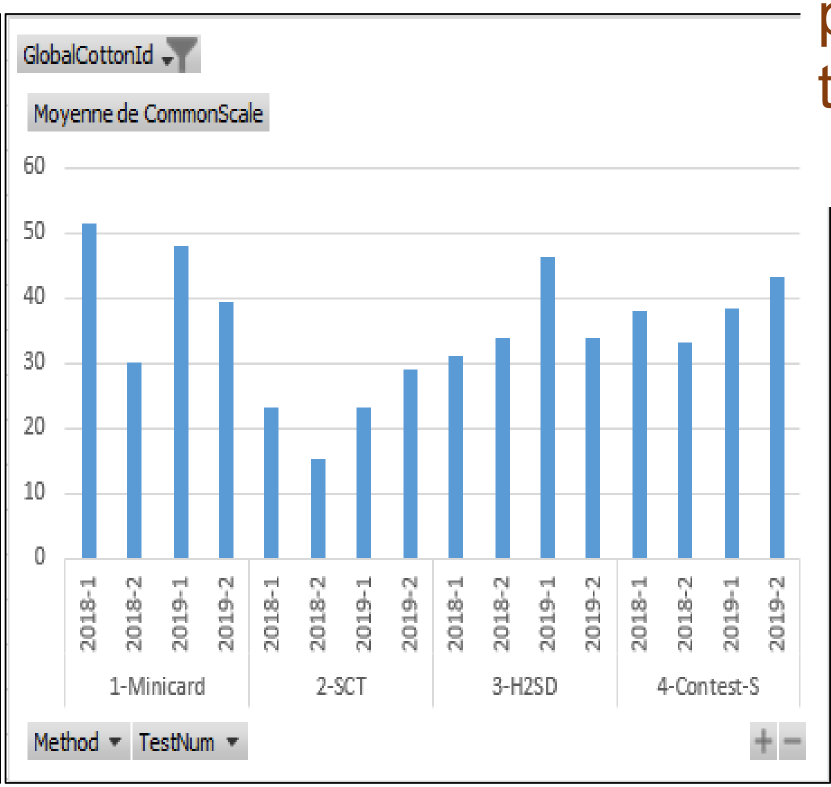

RT results allow tracing lab performances over time

Observations on variations in round-tests

8. Correlations between methods

	Benedict	Carame-lization	Clinitest	**Contest-S**	**H2SD**	HSI-NIR	KOTITI	**Minicard**	Qualitative method	Quantitative method	Reactive Spray	**SCT**
Caramelization	-0.997 NS	1	0.069 NS	0.219 NS	0.188 NS	-0.302 NS	-0.496 **	-0.257 NS	0 NS	0.014 NS	0.484 *	-0.176 NS
Clinitest	-0.115 NS	0.069 NS	1	0.389 *	0.367 NS	-0.037 NS	0.217 NS	0.225 NS	0.009 NS	0.243 NS	0.007 NS	0.433 *
Contest-S	**-0.301 NS**	**0.219 NS**	**0.389 ***	**1**	**0.881 *****	**0.028 NS**	**0.609 *****	**0.859 *****	**0.248 NS**	**0.576 ****	**0.078 NS**	**0.880 *****
H2SD	**-0.613 NS**	**0.188 NS**	**0.367 NS**	**0.881 *****	**1**	**-0.071 NS**	**0.516 ****	**0.820 *****	**0.086 NS**	**0.587 ****	**0.03 NS**	**0.855 *****
HSI-NIR	0.3 NS	-0.302 NS	-0.037 NS	0.028 NS	-0.071 NS	1	0.283 NS	0.17 NS	0.427 NS	0.218 NS	0.048 NS	-0.162 NS
KOTITI	0.5 NS	-0.496 **	0.217 NS	0.609 ***	0.516 **	0.283 NS	1	0.594 **	0.368 NS	0.417 *	0.014 NS	0.472 *
Minicard	**-0.562 NS**	**-0.257 NS**	**0.225 NS**	**0.859 *****	**0.82 *****	**0.17 NS**	**0.594 ****	**1**	**0.208 NS**	**0.458 ***	**0.125 NS**	**0.716 *****
Qualitative method	1 ***	0 NS	0.009 NS	0.248 NS	0.086 NS	0.427 NS	0.368 NS	0.208 NS	1	0.432 NS	0.118 NS	0.155 NS
Quantitative method	-0.887 NS	0.014 NS	0.243 NS	0.576 **	0.587 **	0.218 NS	0.417 *	0.458 *	0.432 NS	1	-0.059 NS	0.623 **
Reactive Spray	-0.189 NS	0.484 *	0.007 NS	0.078 NS	0.03 NS	0.048 NS	0.014 NS	0.125 NS	0.118 NS	-0.059 NS	1	-0.194 NS
SCT	**-0.954 NS**	**-0.176 NS**	**0.433 ***	**0.880 *****	**0.855 *****	**-0.162 NS**	**0.472 ***	**0.716 *****	**0.155 NS**	**0.623 ****	**-0.194 NS**	**1**
Color code:	NS	*	**	***								

Observations on variations in round-tests

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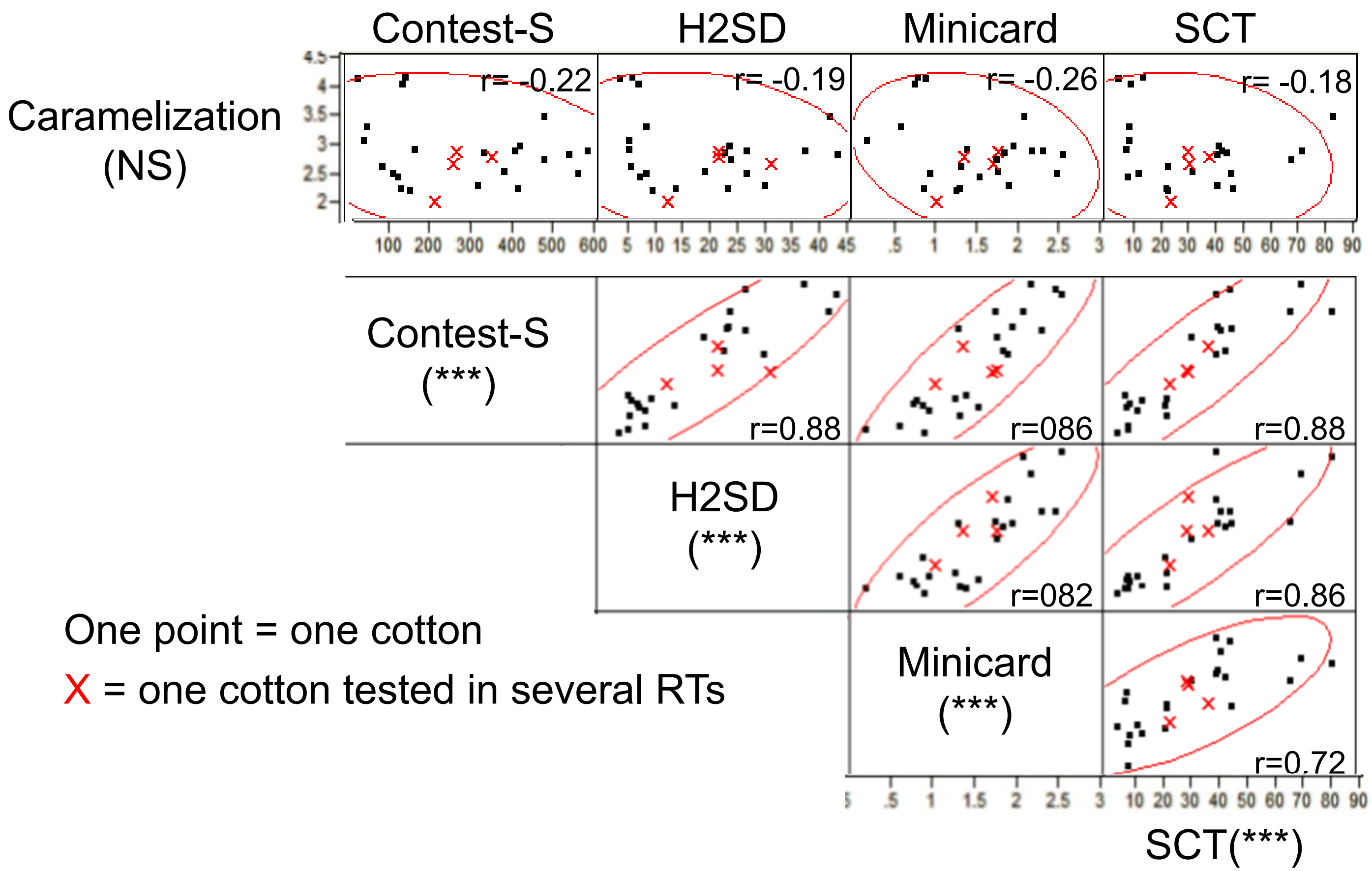
Some methods do not correlate with others

Observations on variations in round-tests

8. Correlations between methods

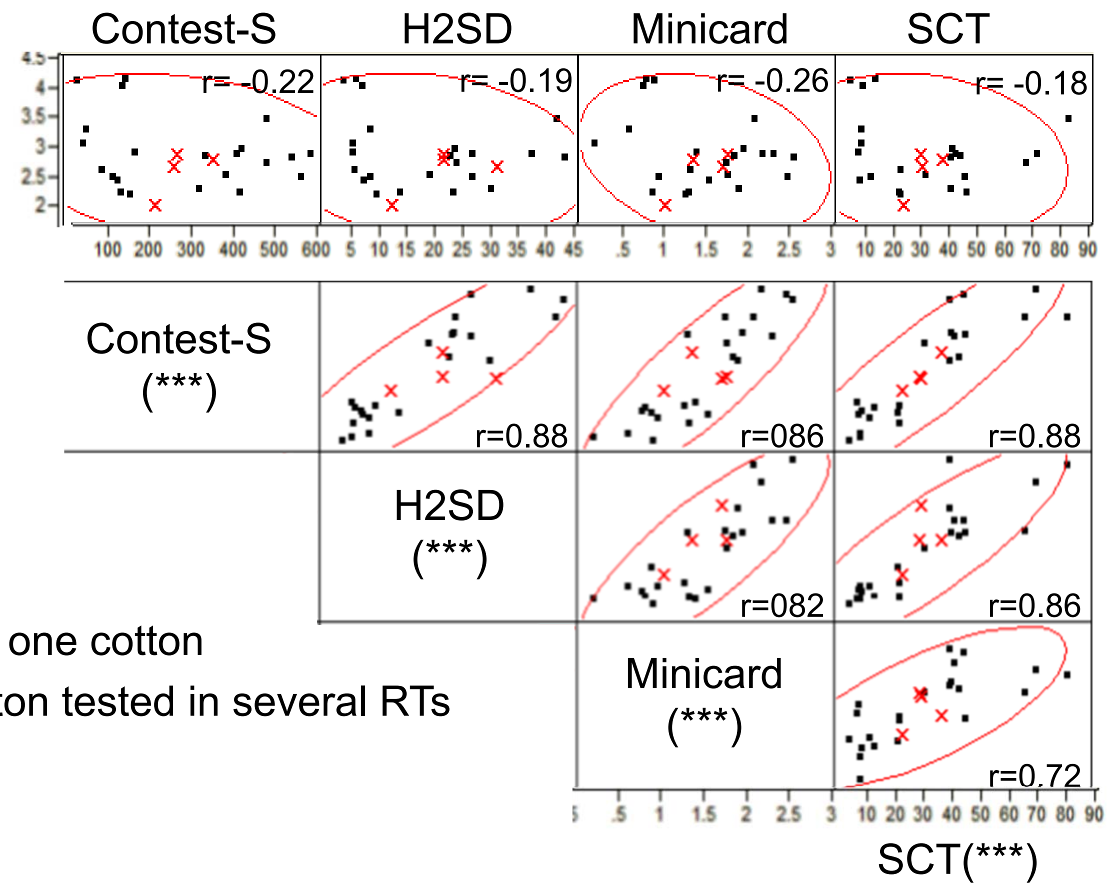


One point = one cotton

X = one cotton tested in several RTs

Observations on variations in round-tests

8. Correlations between methods



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Good correlations between thermo-mechanical methods, Minicard.
Good correlation to SIP.

→ Methods kept for further harmonization

- Contest-S
- H2SD
- SCT
- Minicard

Usual harmonization steps

- Definitions
- Technical and technological developments of the testing methods for achieving a proper sensitivity and quality of the results
- Production of reference materials
- Periodical comparisons between methods and instruments such as the USDA, Bremen or ICAC-CSITC-RTs
- Evaluation of the findings by international committees
- Application of the methods in laboratories at all levels in the supply chain, including in Cotton Association or Cotton Boards

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Challenges for stickiness:

- Stickiness is variable
- Various method principles and units
- Results to be linked to SIP, as well as between instruments and methods

What to keep in mind: next harmonization steps

- Continuation of RT as is (**welcome laboratories and materials**) with all methods
- Harmonization focus on mechanical / thermo-mechanical methods with SCT, H2SD and Contest-S, keeping Minicard as reference
  - Development of an "easy to use indicator" for the laboratories to see their deviations and their need for action
  - Continuation of the analysis of the sources of result variabilities
- Continue studying the impact on honeydew points with their number and their size on test results, spinning (SIP)
- Adoption of best practices by the laboratories with support of Manufacturers
- Development and application of CommonScale definitions on RT results
- When needed, development of a common categorization for all methods (for trade purposes), and suitably include stickiness testing in trade rules

cirad AGRICULTURAL RESEARCH FOR DEVELOPMENT

UNIVERSITÄT BREMEN FASERINSTITUT FIBRE BREMEN

Steps towards suitable stickiness test results for trading and processing

Jean-Paul Gourlot *, Axel Drieling **

* CIRAD (France), ** FIBRE (Germany)

Thanks for 'your visit in Bremen'
to participating laboratories and material providers,
and funders for this work : CIRAD, FIBRE and BBB

We welcome your questions and comments