Looking for variability sources in the measurement of grain Quality

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INTRODUCTION

This paper presents a study of the variability source related with tests to define de physic quality of grains. The main concern in this regard is the high data dispersion obtained in defects grains tests that impact mainly on the validation parameters accuracy and uncertainty of measurement. This makes it difficult and adds complexity to the product evaluation considering the actual quality product specifications recommended internationally and / or used for trade. We select two types of measurements: fermented grains in soybeans and damaged and broken grains in milled rice. Soybeans and rice samples are selected due to: the importance in world consumption of these products, they are national production crops, they are grains of different sizes (medium and small respectively) and finally they have different end use (for human consumption and as raw material for oil or proteins extraction). The different sources of error related with the measurements are analyzed, especially the errors due to the assumptions of statistical behavior of the data used for the estimation of the validation parameters, the random and systematic errors of the method, the error due stability and sampling.

EXPERIMENTAL METHODS

Measurement Methods

The analytical method procedure is segregating the defective grains from the test sample, weighing these defective grains and expressing the test results as a percentage of the original sample. Defects are defined as follows:

-Broken grains in rice (BK): Visual identification in function of their size and, in case of doubt, measure with caliber.

-Damaged (Stained) grains in rice (ST): Visual identification of stained grains.

-Fermented grains in soy (FR): Visual identification of fermented grains and, in case of doubt, cut the grain crosswise with a scalpel to identify the defect in the cotyledon.

Tests comply with ISO/IEC 17025:2017.

Statistical data processing

Exploratory and inferential statistical studies are done to identify data distribution and outliers. Data analysis of accuracy studies are made by appropriate statistical tests.

Accuracy and uncertainty studies

Accuracy is evaluated in term of precision and trueness of each of the methods through appropriate proficiency test studies and by expert comparisons. Performance studies are conducted in more than 15 laboratories in 11 rounds. Precision is evaluated as Relative Standard Deviation (RSD) under conditions of repeatability (RSD_{rep}), intermediate precision (RSD_{ip}) and reproducibility (RSD_R) based on the guidelines of ISO 5725-2:1994. Proficiency tests comply with ISO/IEC 17043:2010. Three independent uncertainty estimation methods are used in order to evaluate the dependence of these results with the mathematical assumptions assumed by each one: Monte Carlo Method, from validation data and by individual uncertainty sources.

Robustness studies

Robustness studies are carried out related to the size of the test sample and the stability of the measurand. Tests are carried out in different size samples and different days.

RESULTS AND DISCUSSION

Behavior data (Table 1) is compatible with a normal distribution. Precision data (Table 2) show high variability of the stained and fermented grain tests being smaller for the broken grain tests. This high variability seems to be characteristic of the method, probably because defects arise by chemical or biological reactions and the classification criterion is visual observation. Variability of the broken grain tests is lower, probably because this defect is associated with the industrial process and the classification criterion is related with instrument measurements. Robustness sample size studies suggest no significant differences related. Control samples for Stained and fermented grain defects are stable for six months and control samples for broken grain defect is stable for a year when rice is preserved with its husk. Uncertainty estimation demonstrates no significant differences between different approaches.

Table 1. Statistical data processing

Statistic/Defect	BR	ST	FR
Kurtosis	0.2	-1.1	0.3
Asymmetry	-0.1	0.5	0.5
Shapiro-Wilk	p>0.05	p>0.05	p>0.05
Outliers_Grubbs	no	no	no

Table 2. Precision data

Defect	RSD (%)
BK	RSD_{rep} (7.7) ≈ RSD_{ip} (9.4) < RSD_R (20)
ST	RSD_{rep} (31) < RSD_{ip} (47) ≈ RSD_R (48)
FR	RSD_{rep} (23) ≈ RSD_{ip} (29) < RSD_R (64)

CONCLUSION

Tests involved in this paper show high variability due to nature of the measurand. Proficiency testing studies seems to be an essential tool for validation purpose, improving the comparability of different laboratories and compatible with the stability of the test samples.

REFERENCES
1. ISO 7301:2011. Rice Specification
2. Instrucao Normativa 11/2007 del MAPA de Brasil.

KEYWORDS (5 keywords)
Grains, defects, broken, damaged, fermented