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Comparison of methodologies for determination total humidity in two types of Andean corn (*Zea mays* L.)

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Abstract. Corn is one of the most consumed crops nationwide, where consumption and production increasing constantly. The development improved varieties have been an arduous work for the National Institute of Agricultural Research (INIAP-Ecuador), where two varieties stand out from the rest; soft maize (INIAP-122) and the semi hard maize (INIAP-180). These varieties present higher production yield, and tolerance to pests and diseases. Humidity of the grain is a key factor, because it closely related to the storage, processing and marketing of the product. The objective of the research is to evaluate the different moisture methods and their principles. For this research, 7 lots of grain humidity were elaborated (10%, 11%, 12%, 13%, 14%, 15%, 16%). After this, a homogeneity test was carried out, in which a statistical analysis was applied in order to evaluate the degree of homogeneity within each of the lots. Moisture was measured applying methods a) ISO 6540:2013 method (oven), b) AACC 44-15A method (oven), c) rapid method as Agratronix MT-16 (electric-capacitance), d) Infrared method (thermogravimetric) and e) INIAP method (electric-impedance). All lots of soft and semi hard corn (10-16%) were homogenous before comparison test. There was significant difference between moisture methods tested in both varieties.

1. Introduction

In Ecuador, maize (*Zea mays*) is the first crop in cultivation area and the fifth in yield. Ecuador has a wide range of habitats (climate, soil and topography), generating a great maize diversity in genotype, expressed by means of color, shape and size of the grain as well as type of endosperm, among others. This allowed the identification of 26 corn races in the Ecuadorian Andes. These germplasms are adapted to biotic and abiotic stress conditions broadening the genetic base which enabled plant breeding development, creating novel varieties [1,2]. Environmental factors such as drought and heat affect yield and quality of the grain, inducing kernel abortion [3].

Soft maize variety (INIAP-122) is characterized by its precocity, low plant heath, lodging resistance and good grain quality. The physical characteristics this variety is: 10 rows/ear; yellow color of dry grain; creamy color of the sweet corn; pink, white, purple color of the tussah, floury grain; soft grain texture. Its protein content is 8.13 %; total sugar content 2.32 % and starch 74.57 %. INIAP-122 is tolerant towards foliar diseases such as leaf blight (*Helminthosporium turcicum*), rust (*Puccinia* sp), and to ear rot caused by *Fusarium moniliforme* which affect maize production worldwide [4]. The current cultivation area of this type of maize in the Highlands of Ecuador is 89 642 ha with 120 019 t, whereas the area devoted to this variety in1997 amounted 5000 ha. This genotype adapts to altitudes between 2200-2800 masl being obtained from multiple crossings between 4 local maize varieties from Chaltura (Ecu-07203), La Florida (Ecu-07297), Natabuela (Ecu-07302) and Imantag (Ecu- 07310), from Imbabura province–Ecuador.

The Ecuadorian Andes Maize Program developed a new improved variety of semi-hard "free-pollinated" corn called INIAP-180 used as forage. The largest area producing forage maize in the Highlands of Ecuador is located between 2200 and 2800 masl, being used mainly as silage. Table 1 shows the characteristics of the studied cultivars INIAP-180 and INIAP-122 and the agroclimatic requirements by these two varieties.

Table 1. Characteristics of corn cultivars and agroclimatic requirements

Characteristic	Semi hard maize INIAP-180	Soft maize INIAP-122
Rain fall: mm	700-1300	600-1000
Temperature: ºC	12-18	12-18
Soil	clay to sandy soil	good drainage
Altitude msnm	2200-2800	2000-2900
Color of the grain	Yellow	dry= yellow; sweet corn =creamy
Shape of grain	Elongate	Cylindrical
Crop cycle (dry corn)	8 months	7-8 months
Crop cycle (sweet corn)	6-7 months	4-5 months
Row distance (cm)	80	80
Plants distance (cm)	50	50
Yield	53 t/ha (foliage)	3,9 t/ha (dry grain) 9,5 t/ha (sweet corn)

The main factors that affect the quality of the grains can be divided into two categories; genetic and environmental. The environmental ones are classified as abiotic and biotic. Among the abiotic factors, post-harvest handling including, hauling, conditioning (initial humidity of the grain and drying temperature) and storage are considered, where temperature and moisture are basic indicators affecting the successful long-term grain storage [5].

There are several methods to determine the moisture content (MC) in grains. They are based on different physical principles for their operation. The method involves determining the MC mass of an undried and dried sample to determine the amount of water in the grain. Other methods determine indirectly the MC from the empirical relationship between physical and chemical features [6]. The methods of mass, colorimetry and gravimetry, for example, give information of the moisture content in absolute form, while others such electrical characteristics of the grain (such as capacitance and conductance relationship between physical and chemical features) do it based on calibration curves generated to compare against reference methods [7].

One of the main challenges of the grain and seed chain is that each moisture determination method with their respective measurement principles should present similar results in the same arrays tested. There are some studies regarding the comparison of methodologies for the determination of humidity in grains. Their results suggest that there are significant differences between methodologies [8–10]. Different moisture values of the same tested matrices can affect the harvesting, drying, storage and commercialization procedures of the grain [11,12]. Therefore, the objective of this work is to make comparisons of moisture determination methodologies for the Andean maize varieties INIAP 122 and 180.

2. Materials and methods

The research was carried out with the variety soft maize (*Zea mays* L.) INIAP-122 and semi hard maize INIAP-180, the plant material was development from the National Institute of Agricultural Research (INIAP) of Santa Catalina Experimental Station located in the Highlands, province of

Pichincha- Ecuador. The study had four phases: a) drying the corn kernel at 9% humidity, b) conditioning the corn grains into seven humidity (10-16%), c) evaluating the homogeneity of the 7 humidity of the grain, d) comparison of five corn grain moisture methodologies and e) correlation and regression of the moisture values obtained with the different methodologies in relation to the reference method ISO 6540: 2010 [13].

2.1. Drying process

The initial drying of the grain was carried out with hot air (60° C) using the Proingel equipment, model OA, following the drying protocol of the FAO [14]. The calculation to determine the initial moisture of the grain equation (1) was used.

Equation:

$$W2 = W1 - \frac{W1\,(M1 - M2)}{(100 - M2)} \; [14]$$

W1: initial weight of the grain (kg)
W2: final grain weight (kg)
M1: initial moisture of the grain (%)*
M2: final grain moisture (%)*
* The moisture percentage of corn grain was made with the methodology of ISO 6540: 2010.

2.2. Conditioning of the corn kernel at the established humidity

Seven percentages of corn grain moisture were established by the conditioning (10%, 11%, 12%, 13%, 14%, 15%, and 16%). In order to standardize the procedure and obtain the pre-established humidity (Gough, 1975 quoted by Maqsood ul Haque 2013) [15,16] equation (2) was used. Each batch (% humidity) of corn grain was formed by 3 experimental units of 1kg. The conditioning of the lots started with humidity of 9%.

Equation:

$$Q = \frac{A(b - a)}{100 - b}$$

Where:
Q: weight of the water to be added
A: initial weight of the sample
a: initial moisture content of the sample
b: final moisture content of the sample

Once the humidity of each batch was reached, the aluminum containers were sealed and stored at 5 ºC ± 2ºC; for 7 days, the sample was homogenized manually (2-3 minutes).

2.3. Test of moisture homogeneity of the corn grain

An assay was performed to evaluate the homogeneity of moisture in 7 batches of hard corn (10 to 16%). The homogeneity was verified within and between experimental units (30 samples) of each lot with the descriptive statistics analysis (F calculated vs. F critical). This process was carried out in all the batches studied.

2.4. Comparison of moisture determination methodologies of hard corn grain

Once the homogeneity of the corn grain of the seven lots was confirmed, it proceeded to the comparison of five moisture determination methodologies described above. The analysis was carried out using a Random Block Design with 6 repetitions. When there were statistical differences between the treatments (methodologies), the functional analysis was performed with the Tukey test (5%).

2.4.1. Method of determination of humidity in the oven (method AACC 44-15 A). Weighed 30 g of soft and semi hard corn was weighed in a capsule and placed in the oven (Memmert brand model SNB 400) for 72 hours at 103ºC ± 1ºC [17].

2.4.2. Method of determination of humidity in the oven (ISO 6540: 2010). Weighed 15 g of soft and semi hard corn and put in a capsule, which was placed in the oven (brand Memmert model SNB 400) for 38 hours at 130 ºC ± 1 ºC.

2.4.3. Infrared moisture determination method. This method is based on the principle of thermogravimetry, ground corn was used. The soft and semi hard was ground separately in the mill KitchenAid brand KSM2FPA, and 2 g were placed of corn in the Boeco infrared equipment, model BMA I50, at 130 °C for 15 minutes.

2.4.4. Moisture determination method by capacitance (Agratronix MT-16). For this, 48 g of corn was weighed (without split kernels), and the sample was placed inside the Agratronix MT-16 equipment. The ambient temperature fluctuated between 20 and 25°C.

2.4.5. Method of determination of humidity by capacitance (Steinlite Moisture Tester 400G). 100 g of corn were weighed, which were placed in the equipment Steinlite Moisture Tester model 400G. The humidity reading follows the protocol of correction factors that details the equipment.

2.5. Correction of the values obtained in the comparison of moisture determination methodologies considering the methodology (ISO 6540: 2010) as reference

With the values obtained from the average of each humidity range (10-16%) of the 5 tested methodologies, regression equations and correlation coefficients were established between the ISO 6540: 2010 (Y) method and the percentage of humidity determined of the other four methodologies (X). In order to harmonize all the results in relation to a single reference methodology. It should be noted that you can choose any methodology as a reference according to the legislation or specific needs.

3. Results and discussions

3.1. Drying and conditioning of soft and semi hard corn kernels

According to equations 1 and 2, the drying and conditioning of grains was carried out in the range of 10 to 16% humidity. It should be noted that the final moisture values of the drying and conditioning were verified by the oven method of ISO 6540: 2010 in the homogeneity test. It is important to highlight that, to optimize the time, previous analyzes of grain moisture determination both for drying and for conditioning were made with the Agratronix MT-16 (capacitance) equipment with a standard deviation ± 0.3% of the humidity percentage.

3.2. Moisture homogeneity test inside bag of each batch

Table 2 and 3 (soft corn and hard corn respectively) shows the results of humidity homogeneity within the three bags (each batch of humidity in the range of 10 to 16%). There were 30 repetitions for each batch.

Table 2. Moisture Homogeneity test in corn grains inside bags of each lot (soft corn kernels INIAP-122).

Moisture corn (%)	Homogeneity F calculated	F critical
Batch 1 (10,03 ± 0,10)	2,1224E-12	2,262
Batch 2 (10,90 ± 0,18)	0,000143049	2,262
Batch 3 (11,93 ± 0,08)	4,21154E-05	2,262
Batch 4 (12,90 ± 0,09)	1,37212E-05	2,262
Batch 5 (13,65 ± 0,17)	4,57477E-06	2,262
Batch 6 (15,15 ± 0,11)	1,55587E-06	2,262
Batch 7 (16,86 ± 0,43)	2,13885E-07	2,262

[a] The lower the value of F calculated, the more homogeneous the sample is presented.
If the critical F value exceeds the calculated F, the sample is not homogeneous.

Table 3. Moisture Homogeneity test in corn grains inside bags of each lot (semi hard corn kernels INIAP-180).

Moisture corn (%)	Homogeneity F calculated	F critical
Batch 1 (10,19 ± 0,34)	0,000280516	2,262
Batch 2 (10,81 ± 0,27)	0,000188644	2,262
Batch 3 (12,06 ± 0,28)	3,7194E-05	2,262
Batch 4 (13,31 ± 0,27)	9,04301E-06	2,262
Batch 5 (14,32 ± 0,30)	2,83581E-06	2,262
Batch 6 (15,44 ± 0,32)	9,3431E-07	2,262
Batch 7 (16,31 ± 0,26)	9,79065E-07	2,262

[a] The lower the value of F calculated, the more homogeneous the sample is presented.
If the critical F value exceeds the calculated F, the sample is not homogeneous.

In both tables (2, 3) it can be seen that the values of, F calculated for both the homogeneity test within bags do not exceed the critical F values 2,262. This allows to infer that the humidity in each lot is homogeneous. This also indicates that a good process of drying and conditioning of grains was performed.

3.3. Comparison of methodologies for determining moisture

The tables 4 and 5 show the results of comparison of five methodologies in the humidity range of 10-16%.

Table 4. Evaluation of 5 methodologies to measure moisture variability (%) of soft corn kernels INIAP-122

Moisture Method	10%	11%	12%	13%	14%	15%	16%
ISO 6540:2010 (Oven)	10,22 ± 0,13 C	11,08 ± 0,28 D	11,89 ± 0,09 D	12,99 ± 0,10 D	14,21±0,16 D	15,35 ± 0,21 D	16,88± 0,34 B
AACC-44-15A (Oven)	10,02 ± 0,21 C	10,92 ± 0,02 D	12,31 ± 0,17 E	13,11 ± 0,15 D	14,09± 0,11 C	15,08± 0,13 D	15,50 ± 0,5 A
Agratronix MT-16) (Capacitance)	6,68 ± 0,33 A	8,18 ± 0,29 A	9,10 ± 0,38 A	10,03 ± 0,25 A	10,98 ± 0,25 A	12,27 ± 0,15 A	15,65± 0,39 A
Infrared	10,10 ± 0,36 C	10,53 ± 0,15 C	11,31 ± 0,17 C	12,51 ± 0,36 C	13,77± 0,37 C	14,63 ± 0,18 C	15,50± 0,33 A
Steinlite(G-400) (Capacitance)	8,45 ± 0,25 B	9,26 ± 0,22 B	10,46 ± 0,12 B	11,00 ± 0,09 B	12,06 ± 0,19 B	13,72 ± 0,22 B	15,70± 0,26 A
(CV)%	3,22	2,12	2,01	1,79	1,74	1,29	2,38

[a] Averages followed by the same letters in the column are statistically the same using Tuckey test (5%)

Table 5. Evaluation of 5 methodologies to measure moisture (%) of semi hard corn kernels INIAP-180.

Moisture Method	10%	11%	12%	13%	14%	15%	16%
ISO 6540:2010 (Oven)	10,17 ± 0,35 B	11,66 ± 0,09 D	12,38± 0,14 C	13,29± 0,27 C	14,22±0,33 C	15,51 ± 0,23 D	16,25± 0,15 C
AACC-44-15A (Oven)	8,82 ± 0,18 A	10,56 ± 0,14 B	11,38 ± 0,07 B	12,33± 0,17 B	13,31 ± 0,21 B	14,58 ± 0,20 B	15,23± 0,17 B
Agratronix MT-16) (Capacitance)	10,42 ± 0,27 B	11,55 ± 0,24 D	12,30 ± 0,012 C	12,98± 0,12 C	13,97±0,12 C	15,15 ± 0,27 C	16,03± 0,31 C
Infrared	8,69 ± 0,16 A	9,06 ± 0,04 A	10,76 ± 0,12 A	11,44± 0,23 A	12,56 ± 0,18 A	13,53 ± 0,36 A	14,66± 0,42 A
Steinlite(G-400) (Capacitance)	8,59 ± 0,24 A	11,11 ± 0,18 C	12,34 ± 0,10 C	13,40± 0,05 D	14,00±0,24 C	14,99 ± 0,09 C	15,82± 0,31 C
(CV)%	2,78	1,45	1,5	1,62	1,72	1,74	1,73

[a] Averages followed by the same letters in the column are statistically the same using Tuckey test (5%)

The results presented in table 4 suggest that, there are significant differences between the five moisture determination methodologies studied for soft maize (INIAP-122). It is important to highlight, there were similarities between the oven methods (4 of the 7 grain humidity) studied ranges, due to no significant differences were found.

When the percentage of semi-hard corn (INIAP 180) was analyzed, it was verified that the oven method (ISO 6540: 2010) was similar with the capacitance method (Agratronix MT-16), except for the corn grain that had 15 % of humidity. Regarding, comparing the two oven methods (Table 5), it was observed that there were differences in all the humidity studied. The coefficient of variation (CV %) between the methods varied from 1.29 to 3.22, this indicates that the differences found are due to the methods and not to external factors to the study.

3.4. Correlation and regression of the reference method versus four methodologies in relation to grain moisture.

The tables 6 and 7 show the regression equations obtained from values with the different methodologies in relation to the reference method ISO 6540: 2010 [13].

Table 6. Regression equations and correlation coefficients between the ISO 6540: 2010 (Y) method and the percentage of moisture determined by other methodologies (X)-Variety: Soft Corn INIAP-122

Statistics	**Methodologies**			
	AACC 44-15A (Oven)	Capacitance (Agratronix MT-16)	Infrared	Capacitance (Steinlite Moisture.400G)
Regression equations	y = 0,8427x + 1,8543	y = 1,2085x - 5,5761	y = 0,8669x + 1,1508	y =1,0477x - 2,3348
Correlation coefficients	0,9591	0,9655	0,9886	0,9853

Table 7. Regression equations and correlation coefficients between the ISO 6540: 2010 (Y) method and the percentage of moisture determined by other methodologies (X)-Variety: Semi Dark Corn INIAP-180

Statistics	**Methodologies**			
	AACC 44-15A (Oven)	Capacitance (Agratronix MT-16)	Infrared	Capacitance (Steinlite Moisture.400G)
Regression equations	Y=1,0521x - 1,735	Y=0,9235x + 0,8669	Y=1,0181x - 2,065	Y=1,1238x - 2,1182
Correlation coefficients	0,9979	0,9965	0,9713	0,9614

After obtaining the results of the comparison of methodologies, we proceeded to establish a correction factor to harmonize the data obtained in relation to the ISO 6540: 2010 methodology. Regarding the data obtained from soft corn (INIAP-122), we can say that the method with the best linear behavior in relation to the ISO 6540: 2010 standard is the infrared; followed by the capacitance method (Steinlite Moisture 400G equipment), the agratronix MT 16 and finally the oven method 44-15A. The linear behavior of the methods in relation to the semi-hard maize (INIAP-180) is different, being the AACC 44-15A oven method with greater linear tendency, followed by the capacitance method (Agratronix MT-16), infrared and finally the Steinlite Moisture 400G. It is interesting to mention that the data presented in tables 3 to 7 are unpublished in the Andean maize varieties addressed in this study.

4. Conclusions

In relation to the homogeneity test of the moisture of the grain of the two varieties, it is concluded that all the lots were homogeneous. Regarding the comparison of moisture determination methodologies of the two varieties, the results suggest that all methods have significant differences, except for the two oven methods. In general, statistical differences and similarities were found among the methods evaluated to measure grain moisture in the two varieties of corn. When correlating the moisture corn between the reference method (ISO 6540: 2010) with the others, a linear behavior was observed.

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