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

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**Abstract.** The importance of humidity moisture determination for storage and harvesting lies in the quality and safety of raw materials, process control and food preservation. There is research that comparing methodologies with significant differences, but none is applied in to hard corn, so in this paper the following objectives are established: 1) Develop standards of hard corn with seven degrees of humidity to ensure homogeneity. 2) Compare six different methodologies for determining of determination of humidity in the ranges of 10-16%. A homogeneity test was performed using the equation of Gough, 1975 and a statistical (DBCA). After verifying that the batches of corn were homogeneous within and between the bags, we proceeded to compare five methodologies in which where an analysis of variance was applied, followed by a Tukey test at 5%. For the homogeneity results inside and between the covers, the lots were homogeneous. For the results of the comparison of methodologies, significant statistical differences were found in the application of one method over the other, finally then, it was made with regression equations the corrections of the moisture results obtained from the different methodologies in relation to the ISO reference method.

1. **Introduction**

Corn, also known by the scientific name of *Zea mays* L. comes from the Greek Zeo, which means to live and Mahiz which means grain. It is part of the family of grasses and its origin is attributed to Mexico. It is commercialized throughout the world, the world production is approximately 800 million tons per year and is the most cultivated cereal in the world, due to its relevancy in human and animal food in addition to the production of alcohol and starch [1–4]. In Ecuador was reported a production of approximately 1.4 million tons in a cultivated area of 358 thousand hectares, with an average yield calculated of 400 kg per hectare [5].

The great demand of the agroindustry, together with the advances of techniques of sowing, mainly in the use of quality seeds, has stimulated that Ecuador has increased in 188% its maize production, which has meant a reduction of the import of approximately 80%. It is also important to consider that the production cost of 1 ton of corn in Ecuador is approximately 900 dollars, which includes the activities of land preparation, seeding, fertilization, agricultural work, harvesting and land rent [6].

Based on the morphological characteristics in Ecuador, 29 maize races are recognized, of which 13 are not cultivated in the Andean regions. The main challenges in the coastal and Amazonian regions to increase yields in the corn crop are nutrient impoverishment, compaction and salinization [7].
The main reason why the large industries, producers and marketers of corn seek to determine the moisture content in cereals, is because moisture determines quality and harmlessness in the grain, in addition, it helps to guarantee the appropriate physical and microbiological characteristics for its commercialization and consumption [8–11]. In force, the Codex (1985) establishes 15.5% the humidity value of the corn for its commercialization; nevertheless, it states the following observation: "Lower moisture limits should be required for certain destinations in relation to the climate, duration of transport and storage [12]. Governments accepting the Standard are requested to indicate and justify the requirements in force in their country ", in this way, The World Food Program (WFP) of the United Nations suggests 13.5% to corn moisture for commercialization [13]. It is important to mention that each country and each segment of the corn chain can have its own moisture values of commercialization established, in the case of Ecuador, established in its internal legislations. In the world, there are several direct and indirect methods to determine the humidity of all types of grains, the direct methods are characterized by being non-destructive and also provide greater accuracy in the results when analyzing a sample. Indirect methods, on the other hand, are characterized by being more destructive. To determine the humidity in hard corn, direct methods can be applied, such as: stove, infrared and distillation, and indirect methods such as electrical equipment [14–16].

Although there are several methodologies and devices used to determine the moisture content in corn grains, the challenge of the chain lies in the variability of results obtained from the determination of humidity [17–19]. In this way, the objectives of the study were to compare the methodologies to determine exactly the moisture content in the hard corn that determines each method against the same homogeneous matrix and subsequently correlate the results and make the corresponding homologation corrections against a referential humidity determination methodology, which in our study is ISO 6540: 2010 [20].

2. Material and methods
The research was carried out with the hybrid maize (Zea mays L.) INIAP H-551. The plant material was development from the National Institute of Agricultural Research (INIAP) of the Pichilingue Tropical Research Experimental Station located in Quevedo, province of Los Ríos-Ecuador. The study had four steps: 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 [20].

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 [21]. The calculation to determine the initial moisture of the grain equation (1) was used.

Equation:

\[ W2 = W1 - \frac{W1(M1-M2)}{(100-M)} \]  

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 (10%, 11%, 12%, 13%, 14%, 15%, and 16%), and the percentages established by the conditioning were reached. In order to standardize the procedure and obtain the pre-established humidity Gough (1975) [22] cited by Maqsood Ul Haque,
2013) [23]. Each batch (% humidity) of corn grain was formed by 3 experimental units of 1 kg. The conditioning of the batches 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 covers were sealed and stored at 5 °C ± 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 each experimental unit and between experimental units (30 samples) of each lot with the descriptive statistics (F calculated vs. F critical). This process was carried out in all the studied batches.

2.4. Comparison of moisture determination methodologies of hard corn grain

Once the homogeneity of the corn grain of the seven batches was confirmed, it proceeded to the comparison of five moisture determination methodologies: oven methods (2), infrared (1), and capacitance (2). The analysis was carried out using a Randomize 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 hard corn was weighed in a capsule and placed into the oven (Memmert brand model SNB 400) for 72 hours at 103 °C ± 1°C [24].

2.4.2. Method of determination of humidity in the oven (ISO 6540: 2010).

Weighed 15 g of hard corn and put in a capsule, which was placed in the oven (brand Memmert model SNB 400) for 38 hours at 130 °C ± 1°C.

2.4.3. Infrared moisture determination method.

This method is based on the principle of thermogravimetry. The corn was ground in the mill KitchenAid brand KSM2FPA, and 2 g were placed into 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 broken 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, the same ones that 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 maize grain moisture between the reference method (ISO 6540: 2010) with the other methodologies.

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 corn grain
According to equations 1 and 2, the drying and conditioning of grains was carried out in the range of 10-16% humidity. It should be noted that the final moisture values of the drying and conditioning were verified by the oven method (ISO 6540: 2010). 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.

3.2. Moisture homogeneity test inside bag of each batch
Table 1 shows the results of humidity homogeneity within the three bags (each batch of humidity in the range between 10 to 16%). There were 30 repetitions for each batch.

| Moisture corn (%) | Homogeneity |
|-------------------|-------------|
|                   | F. Calculated | F Critical |
| Batch 1 (10.00 ± 0.06) | 4.99779E-13 | 2.262 |
| Batch 2 (11.23 ±0.08) | 4.60045E-13 | 2.262 |
| Batch 3 (12.26 ± 0.06) | 9.61586E-14 | 2.262 |
| Batch 4 (13.28 ±0.12) | 1.43966E-13 | 2.262 |
| Batch 5 (14.33 ± 0.14) | 6.00775E-14 | 2.262 |
| Batch 6 (15.32 ± 0.09) | 2.21731E-14 | 2.262 |
| Batch 7 (16.02 ±0.06) | 7.44578E-15 | 2.262 |

*The lower the value of F calculated, the more homogeneous the sample is presented.
*b If the critical F value exceeds the calculated F, the sample is not homogeneous.

3.2.1. Moisture homogeneity test in corn grains between bags of each lot. The homogeneity result between the three covers of each batch of the humidity range is presented in table 2. 30 repetitions were performed for each batch of the humidity range.

| Moisture corn (%) | Homogeneity |
|-------------------|-------------|
|                   | F. Calculated | F Critical |
| Batch 1 (10.00 ± 0.18) | 6.89874E-33 | 2.045 |
| Batch 2 (11.23 ± 0.11) | 2.94349E-33 | 2.045 |
| Batch 3 (12.26 ± 0.19) | 2.52967E-33 | 2.045 |
| Batch 4 (13.28 ±0.16) | 4.09096E-35 | 2.045 |
| Batch 5 (14.33 ± 0.21) | 8.18241E-36 | 2.045 |
| Batch 6 (15.32 ± 0.18) | 1.38008E-36 | 2.045 |
| Batch 7 (16.02 ± 0.12) | 2.75605E-38 | 2.045 |

*The lower the value of F calculated, the more homogeneous the sample is presented.
*b If the critical F value exceeds the calculated F, the sample is not homogeneous.

In both tables (1 and 2) it can be seen that the values of, F calculated for both the homogeneity test within bags (Table 2) and the homogeneity test performed between bags (Table 2), do not exceed the critical F values 2,262 and 2,045 respectively. This allows inferring that, the humidity in each lot is
homogeneous. This also indicates that a good process of drying and conditioning of grains was performed. Similar studies comparing methodologies for determining moisture in maize grains have been performed, [25,26], report data that included samples conditioning, but there is no evidence that the samples tested were homogeneous, in their respective trials the homogeneity of the samples was not considered.

Table 3. Evaluation of 5 methodologies to measure moisture variability (%) of hard corn kernels (INIAP H-551).

| Moisture Method       | Percentual Range (10-16%) |
|-----------------------|---------------------------|
|                       | 10% | 11%    | 12% | 13% | 14% | 15% | 16% |
| Infrared              | 7.52| ± 0.25  | 8.67| ± 0.09 | A  |
|                       | 9.11| ± 0.11  | 10.28| ± 0.21 | A  |
|                       | 11.46| ± 0.21 | A   | 12.52| ± 0.30 | A  |
|                       | 13.31| ± 0.15  | A   | 15.18| ± 0.21 | A  |
|                       | 15.24| ± 0.24  | A   | 17.50| ± 0.12 | A  |
| AACC-44-15A (Oven)    | 9.39| ± 0.14  | 10.04| ± 0.06 | B  |
|                       | 11.18| ± 0.09  | 12.30| ± 0.10 | B  |
|                       | 13.23| ± 0.14  | A   | 15.18| ± 0.13 | B  |
|                       | 15.24| ± 0.12  | A   | 17.50| ± 0.12 | B  |
| Steinlite (G-400)     | 10.05| ± 0.11  | 11.07| ± 0.07 | C  |
| (Capacitance)         | 12.12| ± 0.16  | 13.07| ± 0.07 | C  |
|                       | 14.16| ± 0.13  | A   | 15.22| ± 0.09 | B  |
|                       | 16.20| ± 0.12  | A   | 18.20| ± 0.12 | C  |
| ISO 6540:2010 (Oven)  | 10.21| ± 0.16  | 11.11| ± 0.07 | C  |
| (Oven)                | 12.32| ± 0.13  | 13.15| ± 0.07 | C  |
|                       | 14.28| ± 0.09  | 15.27| ± 0.10 | C  |
|                       | 16.28| ± 0.11  | 18.28| ± 0.11 | C  |
| Agratronix MT-16      | 12.28| ± 0.13  | 13.48| ± 0.16 | D  |
| (Capacitance)         | 14.50| ± 0.19  | 15.47| ± 0.19 | D  |
|                       | 15.65| ± 0.16  | 16.38| ± 0.17 | C  |
|                       | 17.73| ± 0.12  | D   | 19.73| ± 0.12 | D  |

CV (%)  
1.65 ± 0.09 ± 1.24 ± 1.37 ± 0.9 ± 1.16 ± 0.76 ± 0.6

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

Table 4. Regression equations and correlation coefficients between the ISO 6540: 2010 (Y) method and the percentage of moisture determined by other methodologies (X)

| Statistics          | Methodologies                                      |
|---------------------|----------------------------------------------------|
|                     | AACC 44-15A (Oven)                                 |
|                     | Capacitance (Agratronix MT-16)                      |
|                     | Infrared                                           |
|                     | Capacitance (Steinlite Moisture,400G)               |
| Regression equations| Y=0.9273x +1.6951                                   |
| Correlation coefficients| Y=1.1797x - 4.6181                               |
|                      | Y=1.0246X +2.4934                                  |
|                      | Y=0.975x +0.2963                                   |

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.

According to the Table 4, it can be seen that all the results are linear and have a significative correlation coefficient R² (> 0.9). The capacitance method (Steinlite G-400) used by the INIAP showed the high R² followed by the infrared method, oven (AACC-44-15A) [24] and capacitance (Agratronix MT-16) respectively. It is important to emphasize, that these corrections are useful in this type of hard corn and within the study humidity range (10-16%). This coefficient refers to the correction of all methodologies (X) to the reference method of ISO 6540: 2010 (Y). However, using the data from (Table 3), the coefficient can be changed according to the reference methodology to work. Table 3 shows significant differences between the methods, similar work shows the same trends [19,26,27], it should be noted that the oven method (ISO 6540: 2010) and capacitance (Steinlite G-400) did not present significant differences.
4. Conclusions
The humidity of the corn grain was homogeneous when the samples of the same batch were evaluated. F calculated was lower than the F critical in both cases, confirming the homogeneity of all batches used in the trial. When comparing the corn grain moisture measurement methods, it was found that there is no difference between the capacitance method (Steinlite G-400) with the others three methods (AACC-44-15A, infrared and Agratronix MT-16).

All the methodologies presented linear tendency when compared with the reference method (ISO 6540: 2010). It should be noted, the best correlation were the electric capacitance (Steinlite G-400) and the infrared method.

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