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EQUILIBRIUM MOISTURE CONTENT OF KABULI CHICKPEA, BLACK SESAME, AND WHITE SESAME SEEDS

P. R. Armstrong, E. B. Maghirang, Bhadriraju Subramanyam, S. G. McNeill

ABSTRACT. Sesame and chickpea are important crops in Ethiopia because both are major export crops that generate much revenue for both small farmers and the country as a whole. However, there is a lack of information about the fundamental equilibrium moisture content (EMC) relationships among these crops, which would help facilitate better monitoring and storage. Therefore, EMC adsorption and desorption prediction models based on temperature (T) and relative humidity (RH) were developed for the modified Chung-Pfost and modified Henderson models for Kabuli chickpea (KC), black sesame (BS), and white sesame (WS) seeds. The samples for conducting the adsorption and desorption tests were conditioned to various moisture content (MC) levels for the EMC test models. The samples (\sim 500 g) were placed in multiple sealed enclosures equipped with T and RH sensors, which were placed in an environmental chamber where they were exposed to three temperatures (15°C, 25°C, and 35°C). The MCdb ranges used for model development for adsorption and desorption were, respectively, 11.6% to 19.5% and 8.9% to 16.9% for KC samples, 5.0% to 8.7% and 4.3% to 6.9% for BS, and 4.2% to 8.7% and 3.5% to 7.6% for WS. Nonlinear regression was used to determine the model coefficients for the modified Henderson and modified Chung-Pfost equations. The prediction statistics for the adsorption and desorption models yielded an SEE of, respectively, 0.53% and 0.68% MCab for KC, 0.23% and 0.13% for BS, and 0.28% and 0.25% for WS. The model coefficients obtained in this study will be used in a moisture meter based on EMC measurement, which is currently being used as part of a USAID postharvest project in various African and Asian countries. These EMC models may also be important for other grain operations, which include harvesting, drying, storage, conditioning, and processing.

Keywords. Adsorption, Chickpea, Desorption, EMC, Equilibrium moisture content, Sesame.

he measurement of moisture content (MC) has long been established as critical for maintaining the quality of various grains, oilseeds, and legumes. Numerous MC measurement techniques have been and continue to be developed. The oven method is the reference method for MC and has been approved by numerous associations including the American Society of Agricultural and Biological Engineers (ASABE), the Association of Official Analytical Chemists (AOAC) International, and the American Association of Cereal Chemists (AACC) International.

The USDA Grain Inspection, Packers and Stockyards Administration (GIPSA) and the Federal Grain Inspection Service (FGIS) use an electronic meter with a Unified Grain Moisture Algorithm (UGMA) as the official moisture meter for officially inspected grains and commodities. Commercial versions of this meter are the DICKEY-john GAC 2500-UGMA and Perten AM 5200-A (GIPSA-USDA, 2016). However, time and cost constraints and/or the ease of usage for some field applications such as grain receiving, conditioning and storage facilities make these instruments impractical. Moisture meter models available for field use vary based on the commodity to be tested, sample size requirement, ease of sample collection, presentation, portability, cost, accuracy requirement, and existing technologies.

Recent work to develop an inexpensive and robust meter for developing countries utilizes temperature (T) and relative humidity (RH) measurements to predict equilibrium moisture content (EMC) (Armstrong et al., 2017). This meter has been used in several African countries and Bangladesh since 2014 and is referred to as the PHL meter primarily because its development was funded by the United States Agency for International Development-Feed the Future Lab for the Reduction of Post-Harvest Loss at Kansas State University. The above research was based on an earlier study by Armstrong and Weitig (2008), who developed a

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similar instrument for EMC grain measurement. Chen (2001) also studied EMC measurement for medium rough rice and dent type corn, and Uddin et al. (2006) examined errors in EMC predictions.

A previous study on EMC modeling for Desi chickpea was reported by Menkov (2000), who used a gravimetric static method at 5°C, 20°C, 40°C, and 60°C for water activities ranging from 0.110 to 0.877. A Fraction-Linear model was considered the best model to describe the sorption data relationships since other models showed patterned errors and higher standard errors of moisture (SEM). The modified Henderson model from the above study is currently used in the PHL meter for chickpea and has an SEM of 1.3% for desorption and 3.01% for adsorption. Moreira et al. (2002) reported a good fit of water activity to EMC at different temperatures using the Guggenheim-Anderson-de Boear (GAB) model for chickpea. They observed that the adsorption and desorption isotherms for chickpea had no hysteresis effect, which, they indicated, may be due to the use of different varieties of chickpea for the determination of EMC. However, there was no information on the specific types or varieties of chickpea used in the study.

Dairo and Ajibola (2001) fit their data to a modified Henderson desorption model for sesame resulting in standard errors of 3.3% to 4.1% for EMC. The temperature range for the data was from 40°C to 80°C, which may lead to erroneous predictions for typical day temperatures. Chayjan (2010) provided graphical experimental values of EMC as a function of temperature and the water activity of sesame seeds. The curves were moisture sorption isotherms at four high-temperature levels, with temperatures ranging from 45°C to 75°C and relative humidity from 55% to 98%. Kaya and Kahyaoglu (2006) examined the GAB and Halsey isotherm models at three different temperatures, 15°C, 25°C, and 35°C, for fitting sorption data; they found that the GAB model provided better moisture predictions than the Halsey model. The sesame used in the study was described as a brown Turkish cultivar, and the experimental methods imply that an adsorption process was used to determine the equilibrium moisture conditions.

The PHL meter is programmed to measure the MC of most major crops and includes wheat, corn, soybeans, rough rice, sorghum, and Desi chickpea using the modified Henderson equation and coefficients obtained from previous studies, ASAE Standards D245.6 (2007) and Menkov (2000) for chickpea. Users of the PHL meter in Ethiopia had requested that Kabuli chickpea and black and white sesame seeds' coefficients be added to the meter. Modeling data or coefficients specific to Kabuli chickpea were not available. Desi chickpea have thicker seed coats than the Kabuli chickpea (Sastry et al., 2014) and are usually much smaller; therefore, a specific model for Kabuli chickpea is needed. Studies related to the modeling of EMC for sesame are limited and are not in the desired range of conditions needed for the PHL meter. The existing information should be further verified.

The objective of this study was to develop the desorption and adsorption EMC models for Kabuli chickpea, black sesame, and white sesame seeds based on the relative humidity and temperature experimental data. This involved fitting the experimental data into two existing EMC models, namely, the modified Henderson and the modified Chung-Pfost equations. The results from this study will directly enhance the capability of moisture meters designed for measuring the MC of multiple and/or additional commodities based on relative humidity and temperature readings. Similarly, the adsorption and desorption EMC will provide useful information for other grain operations such as drying, conditioning, storage, and other handling operations.

MATERIALS AND METHODS

CHICKPEA AND SESAME SEED SAMPLES

Commercially available Kabuli chickpea and black and white sesame seeds (~25 kg for each seed type) were purchased from a local store in Manhattan, Kansas. The Kabuli chickpea were labeled as originating from Canada and distributed by Raja Foods (Skokie, Ill.) under the brand name Swad. The sesame seeds were both labeled as originating from Thailand and distributed under the brand name O-Cha by Global Food Trading Co., Ltd. (Pomprab, Bangkok, Thailand). The purchased seed samples came in sealed plastic bags (2 kg container for Kabuli chickpea and 200 g container for black and white sesame seeds). The average MC_{db} of seeds obtained from triplicate air oven method measurements was 13.8% for Kabuli chickpea, 6.0% for black sesame, and 5.3% for white sesame seeds.

MOISTURE CONTENT MEASUREMENT

The Approved Method ASAE S352.2 air oven method for measurement of moisture content of unground grain and seeds (*ASAE Standards*, 1988) for whole chickpea was used (103°C, 72 h, 15 g whole chickpea). There is no existing reference method for measuring MC of sesame seeds; previous studies referenced the use of the Approved Method ASAE S352.2 for flaxseed and the AOAC Standard Official Method 14:004. Evaluation of MC measurement procedures for black and white sesame at different sample sizes (7 g and 10 g) dried at 103°C until they reached constant weight (after ~4h) showed similar MC results. Thus, the MC measurement protocol selected for both black and white sesame seeds was to use 7 g sample dried at 103°C for 4 h.

SAMPLE PREPARATION TO DIFFERENT MOISTURE LEVELS

Seeds for each type were combined from each packet into one bulk sample and manually mixed. These bulks were then divided into eight sub-samples (~3 kg) using a Boerner sample divider (Seedburo Equipment, Des Plaines, Ill.) and placed in 15 L square plastic buckets with tight-fitting lids (Letica, Rochester, Mich.) to ensure proper sealing. Four sub-samples were used for adsorption samples, and the other four were used for desorption samples.

The target moisture contents used for modeling the three seed samples were selected based on the average MCs of the original samples, which were considered as safe storage MC levels. The original MC of seed samples plus three other MC levels that were lower and higher than the original MC were then selected for EMC modeling. It will be noted that the desired MC levels were higher for Kabuli chickpea (12% to 18%) compared to black and white sesame seeds (5% to 8%), which may be attributed to the differences in seed compositions. Kabuli chickpea have substantially higher carbohydrates and lower fat content at 66.5% and 5.9%, respectively while sesame seeds have low carbohydrates and high fat content (17.1% and 48.4% for black sesame and 15.5% and 52.6% for white sesame, respectively (Wang and Daun, 2004; Kanu, 2011).

Re-wetting for adsorption samples was achieved by calculating the quantities of water to be added to achieve target moisture of approximately 12%, 14%, 16%, and 18% MC_{db} for Kabuli chickpea and 5%, 6%, 7%, and 8% MC_{db} for black and white sesame seeds. Seed samples in the plastic bucket were wetted by adding distilled water by mist spraying and then continuously mixing the samples until the desired weight for each target MC was obtained. After sample preparation, the buckets were immediately sealed and placed in 6°C cold storage for at least 5 days to allow sample equilibration. The buckets were mixed daily by tumbling end-over-end at least 10 times. After the equilibration period, samples (three replicates) were randomly obtained from each bucket to verify the sample MC_{db} .

For desorption samples, the as-is MC of the originally packaged chickpea and sesame were used as one of the four desorption sub-samples. The lowest moisture levels were attained by removing the amount of water from the original samples to obtain a target moisture of ~10% for Kabuli chickpea, $\sim 4\%$ for black sesame and $\sim 4\%$ for white sesame. For the remaining two higher level moisture percentages, which are both above package moisture, the procedure involved re-wetting to ~19% MC_{db} for Kabuli chickpea and 8% MC_{db} for both black and white sesame seeds using the adsorption study procedure. The samples were then airdried in a 43°C oven to the target desorption moisture levels followed by a 5-day equilibration period. The target MC_{db} levels were ~14% and ~17% for Kabuli chickpea and $\sim 6\%$ and $\sim 7\%$ for both black sesame and white sesame seeds. The sample MC was determined after all the samples were conditioned.

TEMPERATURE AND RELATIVE HUMIDITY MEASUREMENT

For both adsorption and desorption tests, the bulk subsamples with different MC levels were divided into equal portions using a Boerner sample divider to obtain three samples for triplicate measurements.

To measure relative humidity and temperature for each moisture level, the samples were placed in small plastic boxes equipped with two RH-T sensors. The measurements for each of the three moisture levels were conducted in triplicates using three boxes; thus, six relative humidity and temperature readings were obtained for each moisture level. This method was also used by Armstrong et al. (2012). The measurement boxes were supplied by OPI-Integris (Lenexa, Kan.) and were an adaption of their in-bin moisture cable monitoring system. Each box held approximately 300 g of grain and had a flexible lid seal to prevent air exchange. There were 30 boxes, and only 27 were used to simultaneously monitor the three seed types conditioned by adsorption

treatments (3 seed types, 3 moisture contents, 3 reps). Samples conditioned by using desorption were run in the same manner.

The boxes were placed in an environmental chamber (Percival Scientific Intellus Control System, Percival Scientific, Fontana, Wis.) set at 15° C. They were kept at this temperature for approximately 24 h to allow the air inside the box to reach a temperature equilibrium followed by moisture equilibration with the interstitial air. Temperature and relative humidity values were obtained immediately after increasing the chamber temperature to 25° C. Temperature and relative humidity values were again recorded the following day, and the process was repeated at a temperature of 35° C. After data collection, the chamber was turned off, and the samples were allowed to sit in the chamber for a minimum of 48 h to reach room temperature before each box was opened to obtain samples for moisture determinations.

EMC MODEL FITTING

The PROC NLIN (SAS Institute Inc., Cary, N.C.) procedure was adopted to develop regression models for the modified Chung-Pfost (eq. 1) and modified Henderson (eq. 2) models for adsorption and desorption data sets.

$$MCdb = -\frac{1}{B}ln\left[\frac{-(T+C)\ln(RH)}{A}\right]$$
(1)

$$MCdb = \frac{1}{100} \left[\frac{\ln(1 - RH)}{-K(T + C)} \right]^{1/N}$$
(2)

MCdb is the moisture content on a dry basis and A, B, and C are regression parameters (modified Chung-Pfost). RH is decimal relative humidity, and T is the temperature in Celsius. The coefficients K, C, and N are regression parameters for the modified Henderson model.

Separate adsorption and desorption models were developed for each seed type using all moisture levels prepared for each seed type. Prediction accuracy was reported as the standard error of estimates (SEE) (eq. 3).

$$SEE = \sqrt{\frac{\sum (MCdb - MCdb')^2}{N}}$$
(3)

MCdb is the reference oven moisture content, MCdb' is the predicted model moisture content, and N is the number of data points used for model development.

RESULTS AND DISCUSSION

Table 1 shows the moisture range of KC, BS, and WS samples used for adsorption and desorption EMC modeling. The model statistics for the modified Chung-Pfost and Henderson equations are also shown. Either the convergence criterion was not met, or the data for the black sesame data failed to converge with the modified Chung-Pfost equation. The SEE values for adsorption and

Table 1. Adsorption and desorption coefficients f	for the modified Chung-Pfost and modified
Henderson equations for Kabuli chickpea, b	black sesame, and white sesame seeds.

		Moisture Content Range	Modified Chung-Pfost Equation Constants			
EMC Model	Seeds	(MC_{db})	А	В	С	Model SEE
	Kabuli Chickpea	11.6 - 19.5	309.7	10.09	178.4	0.49
Adsorption	Black Sesame	5.0 - 8.7	[a]	[a]	[a]	[a]
	White Sesame	4.2 - 8.7	402.8	31.73	186.6	0.27
	Kabuli Chickpea	8.9 - 16.9	337.0	12.65	118.6	0.59
Desorption	Black Sesame	4.3 - 6.9	[a]	[a]	[a]	[a]
	White Sesame	3.5 - 7.6	424.9	39.11	119.7	0.25
		Moisture Content Range	Modified Henderson Equation Constants			_
EMC Model	Seeds	(MC_{db})	K	Ν	С	Model SEE
	Kabuli Chickpea	11.6 - 19.5	2.53 E-04	1.0173	291.6	0.53
Adsorption	Black Sesame	5.0 - 8.7	1.35 E-04	1.8396	249.3	0.23
	White Sesame	4.2 - 8.7	4.80 E-04	1.1691	335.1	0.28
Desorption	Kabuli Chickpea	8.9 - 16.9	3.08 E-04	1.2785	100.7	0.68
	Black Sesame	4.3 - 6.9	2.07 E-04	2.0322	106.4	0.13
_	White Sesame	3.5 - 7.6	5.25 E-04	1.4466	174.2	0.26

^[a] Nonlinear regression failed to converge using all iterative methods including gradient, Newton, Gauss-Newton, Marquardt and false position (DUD). The Gauss-Newton convergence method was used for all other models.

desorption ranged from 0.49% to 0.68% MC_{db}, respectively, for KC, 0.13% to 0.23% MC_{db}, respectively, for black sesame, and 0.25% and 0.28% MC_{db}, respectively, for white sesame. All models can provide good estimates of the MC relative to the accuracy of other EMC grain models; for example, the SEEs reported by Armstrong et al. (2012) for corn ranged from 0.31% to 1.61% MC_{db}.

Considering that MC is generally reported in trade as wet basis instead of dry basis, all predictions based on the EMC models were converted into wet basis in the analyses of all adsorption and desorption model relationships. Figures 1 to 3 show adsorption and desorption model relationships between RH and EMC_{wb} for the three seeds types at three temperature settings (15°C, 25°C, and 35°C).

The results showed expected relationship patterns between RH, EMC and T for both adsorption and desorption models and for all seed types; RH was consistently higher at higher T for samples at constant MC. This pattern was less pronounced for sesame than for chickpea seeds. Small hysteresis effects were observed by Moreira et al. (2002) for chickpea seeds and by Aviara et al. (2002) for sesame seeds. Moreira et al. (2002) noted that the prior history of the seeds, such as possible adsorption/desorption cycles, might explain the low variations in the hysteresis effect in adsorption and desorption samples despite controlling the variables during the experiment. KC, BS, and WS seeds used in this study were obtained from a commercial retail store, and there was no information on prior seed handling. Thus, the samples used in this study may have already undergone prior cycles of desorption and adsorption after harvest. The calculated SEE values (eq. 3) for adsorption and desorption were respectively 0.533 and 0.678 for KC, 0.226 and 0.127 for black sesame, and 0.281 and 0.262 for white sesame. Based on these values, the models can provide accuracy in moisture prediction.

The published chickpea EMC coefficients for the modified Henderson equation from Menkov (2000) (K = 0.000328, N = 1.415975, and C = 48.96227), yielded a standard error of measurement of 1.3% MC_{db} for the desorption process. The difference in using these coefficients and the adsorption coefficients from our study is that the moisture is predicted to be 1.6% higher, on average, with the Menkov coefficients at 25°C (fig 4). This indicates a considerable difference between the two types of chickpea. The differences between MC determined by Kaya and Kahyaoglu (2006) for the brown Turkish sesame at 25°C and black and white sesame adsorption data for this study are shown in figure 5. The brown Turkish sesame did not differ as much from the black sesame as it did from the white sesame.



Figure 1. RH and EMC_{wb} relationships for Kabuli chickpea using the modified Henderson equation.



Figure 2. RH and EMC_{wb} relationships for black sesame using the modified Henderson equation.



Figure 3. RH and EMC_{wb} relationships for white sesame using the modified Henderson equation.



Figure 4. EMCwb differences between Desi and Kabuli chickpea for adsorption at 25°C.



Figure 5. EMC_{wb} prediction for black and white sesame based on our data and for brown Turkish sesame seeds from Kaya and Kahyaoglu (2006). Adsorption predictions are shown at 25°C.

CONCLUSIONS

Kabuli chickpea, black sesame, and white sesame seeds all showed expected patterns of increasing EMC with increasing RH and decreasing EMC with an increasing T at a constant RH. Black sesame seeds showed higher EMC compared with white sesame for the same RH and T. Low SEE values of 0.13%-0.68% MCdb for the three seed types indicated good prediction accuracy for the adsorption and desorption EMC models. Regression analysis using the modified Chung-Pfost model did not converge for black sesame. The modified Henderson model exhibited the best ability to predict the EMC for adsorption and desorption process for Kabuli chickpea, black sesame, and white sesame seeds.

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