Sorption characteristics of red lentils as affected by postharvest conditions

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INTRODUCTION

The total value of Canada's red lentil crop is about $260 million (Jubinville and Kostal 2013) and 99% of the total production comes from Saskatchewan. Canada produces about 18% of the world's lentils (green and red) and contributes over 40% of this to the export market. Most buyers specify stringent end-use quality characteristics but due to lack of research data, the optimal conditions for storage are not known. Therefore, when Canadian lentils are exported to importing countries (mainly in Asia and the Middle-East), the quality of lentils gets adversely affected due to change in climatic conditions.

The available agricultural land in other lentils producing countries such as Turkey, India, and Australia has constrained any significant increase in production. Canada, on the other hand, has no dearth of cultivable land and the production of red lentils can be easily boosted. The unique problem that Canada faces is that our climate is very different from other lentil producing and importing countries. Consequently, when lentils are exported to regions with entirely different climatic conditions, they undergo biochemical changes that adversely affect their end-use. Considering the financial benefits of increasing the red lentil production in Canada, there is a need to overcome some of the bottlenecks related to agronomy, post-harvest practices, grading, processing, health and nutrition, value-added product development, and marketing.

Moisture adsorption and desorption of hygroscopic materials such as lentils are complicated physical processes during which moisture and heat are transferred through the
material and are exchanged with the environment. The knowledge of the equilibrium moisture content (EMC) of a biological material is essential for the efficient operation of systems for wetting, drying, or storing the material. The EMC for a given environment may be defined as the moisture content of the material when left in the environment for an infinite period of time. The relationship between EMC and relative humidity (RH) of the inter-granular air during drying either adsorption or desorption are of primary importance in maintaining seed quality during storage and processing. Almost all biological materials exhibit a hysteresis loop. Multon et al. (1981) conducted studies on the effect of sorption hysteresis on grain fitness for milling. They concluded that the sorption history (adsorption or desorption) affected the apparent modulus of elasticity of wheat kernels and consequently their milling behavior. Cenkowski et al. (1981) reported that moduli of elasticity for canola kernels were significantly lower for kernels brought to 7% moisture equilibrium through desorption then those brought to equilibrium through adsorption. In typical storage conditions, the material is exposed to variable temperature and RH. These conditions provide cyclic drying and rewetting of the stored material, which in turn affects the EMC behavior (Bielewicz et al. 1993). Also, bulk storage exposes lentils to freeze-thaw cycles, which may have an effect on their quality and EMC. Lentils at high moisture content can promote insect and mold growth, degrading lentil quality. Even through storability, in general, is longer at lower moisture, very low moisture content of lentils may cause seed cracking and splitting and lower bulk density. A reduction in product bulk density is disadvantageous to producers, as lentils are sold on mass basis.

Majority of work in Canada on lentils was done on two varieties (Laird and Eston) in the 1980s and 1990s (Bhatty 1988, 1995; Cenkowski et al. 1989; Tang and Sokhansanj 1993a, 1993b, 1993c, 1994; Tang et al. 1994). It was reported by Bielewicz et al. (1993) that variety and growing location have a substantial effect on EMC characteristics and; therefore, historical data on lentils do not reflect the true behavior of new varieties or the same variety grown in different locations. There is no data available on equilibrium moisture content (EMC) for red lentils produced in Canada while 50% of the world trade is in this crop.

The objective of this study was to define the EMC characteristics of three red lentil varieties (CDC Robin, CDC Blaze and CDC Redberry) and identify a mathematical model that best predicts the EMC-ERH (equilibrium relative humidity) relationship. This was accomplished by experimentally determining the sorption characteristics of red lentils between 5 and 30ºC. For each of the three varieties, samples of three initial conditions were tested. These were (i) freshly harvested, (ii) successively rewetted and dried, and (iii) samples exposed to freezing and thawing cycles.

MATERIALS AND METHODS

Materials
Three varieties of red lentils in commercial production (CDC Blaze, CDC Redberry, and CDC Robin) were chosen for this study. This selection gave the maximum spread in the seed size of red lentils, which is a factor that affects EMC characteristics. The three lentil varieties (2006 crop) were acquired from Gregoire Farms (North Battleford, SK). These three varieties were certified by the Canadian Seed Growers’ Association (CSGA) indicating that the crop met the requirements of the CSGA for varietal purity. The three lentil varieties were stored in their original polyethylene packaging (45 kg polyethylene bags) at a temperature of 4ºC. The lentil content was approximately 13.8% db (dry basis) during storage.

ERH Procedure
The ERH-EMC characteristics for red lentils were determined using the ERH method involving enclosing a relatively small mass of air with relatively large mass of lentils and allowing the two to achieve equilibrium at a constant temperature. The experiments were conducted for three lentil varieties and two conditioning regimens (rewetting/drying and freezing/thawing cycles) for the storage temperature in the range of 5 to 30ºC.

Apparatus
The diagram of the ERH apparatus used in this study is shown in Fig. 1. Ten such devices were constructed so that trials could be conducted in parallel. The ERH apparatus were placed in a container filled with water. The water temperature was controlled by a water bath whereas the headspace temperature was controlled by a separate air temperature control system.

Fig. 1. Diagram of the equilibrium relative humidity (ERH) apparatus.
Each device was composed of a sealed stack of ten PVC plastic sample trays with #30 stainless steel wire mesh bottoms that allowed air to pass through the stack. The PVC trays had a 96 mm internal diameter, and allowed for a maximum sample depth of 10 mm.

Air was re-circulated through the stack by an ELITE 802 aquarium air pump (Rolf C. Hagen USA Corp, Mansfield, MA). The air pump casing was sealed to ensure that no outside air was introduced into the system.

Each stack of trays was placed inside a larger PVC column, which formed the inner wall of a water jacket. A threaded top cap housed the RH and temperature sensors and when threaded onto the large PVC column, it compressed and sealed the sample trays. Pipe fittings in the top and end cap of the PVC column allowed circulated air to enter. Compression pipe fittings on the top and end cap of the PVC column allowed connection to the inlet and outlet lines of the air-circulating pump.

All ten ERH apparatus were secured within an insulated water bath that maintained a constant operating temperature during the course of the experiments. Two chillers capable of maintaining water temperature to within 0.1°C of the desired value maintained the insulated water bath’s operating temperature. By passing the plastic tubing connecting the sample column and the air pump through the water the heat added to the air by the air pumps was eliminated. In addition, a small fan and heat exchanger placed within the airspace above the water line ensured that air temperature matched the water temperature set point.

A relative humidity (RH) sensor and thermocouple sensor were placed inside the top cap, allowing RH and temperature of the air to be measured. The RH sensors were HIH 4000 series thermoset polymer capacitive sensing elements (Honeywell Inc., El Paso TX). The HIH 4000 series sensor has an accuracy of 2% RH at 25°C. The sensor is accurate within the full range of relative humidity (0 to 100% RH) between 0 to 50°C. A T-type thermocouple was used to measure air temperature, with an accuracy of 0.8°C.

A 5V regulated DC power supply was used to power the RH sensors. Both, the RH sensors and thermocouples were attached to an Agilent 34970A data acquisition device (Agilent Technologies Inc., Santa Clara, CA) to measure sensors’ output voltages. A Windows PC running Agilent’s Benchlink software was connected to the 34970A to monitor and record the sensors’ output.

Experimental Procedure

Information on the initial moisture content of red lentils is required for sample preparation and also the lentil final moisture content needs to be measured at the end of individual experiments. This final moisture content is termed as EMC. The American Society of Agricultural and Biological Engineers (ASABE) standard S352.1 does not include a procedure for lentil moisture content determination using the convection oven method for whole grains (ASABE 2006). A method proposed by the American Association of Cereal Chemists (AACC) AACC Method 44-15A did not yield adequate results due to sample exposure to dry air during handling and grinding. Therefore, a method developed by Tang and Sokhansanj (1991) was used for lentil moisture determination. Tang and Sokhansanj’s method follows the ASABE general procedure for moisture determination of whole grains, specifying a 16 g sample dried at 130°C for 20 h.

The experimental procedure used in this study required lentil samples to be prepared to various initial moisture contents before running the EMC experiment. The required amount of fresh lentils was placed in approximately 10 mm thin layers on screen-bottomed trays in a convection oven operating at 50°C. The term “fresh” here refers to a sample obtained from a farm and stored at 4°C in 45 kg bags as described before. The lentils were left to dry for a minimum of 24 h, then removed from the oven and allowed to cool to room temperature. The lentils were then placed in a sealed plastic bag and stored at 4°C. A small sample of the 50°C dried lentils was removed from that sample of dried lentils to determine moisture content.

After the moisture content of the 50°C-dried sample was determined, the lentils were weighed out into individual 100 g samples and placed in small plastic bags. Adding predetermined amount of water needed to achieve the desired sample moisture content that would be used in the EMC-ERH experiments moisturized each 100 g sample. Distilled water was added to the sample using a pipette, and then the plastic bag was sealed and shaken for at least 30 s, ensuring that the lentils were evenly coated with water. The sealed samples were left for 48 h at room temperature to absorb and evenly distribute moisture throughout the individual seeds. The lentils were then moved to a refrigerator set to 4°C for storage.

Preparation of freeze/thaw treated lentils

The conditioned lentils needed for the determination of EMC-ERH characteristics of lentils subjected to freezing and thawing cycles were prepared from “fresh” lentils stored at 13.8% m.c (db) at 4°C. Zip-lock bags containing approximately 1200 g of the desired lentil variety were subjected to three freezing and thawing cycles at 5°C and -10°C for 24 h at each temperature. The samples were stored at 4°C once the conditioning was complete.

Preparation of lentils for successive rewetting

Preparation of the conditioned lentils used in the determination of EMC-ERH characteristics of successively wetted and dried lentils was achieved by subjecting “fresh” lentils of each variety to three drying and wetting cycles. The “fresh” lentils had initial moisture content of 13.8% (db). Lentils were dried in thin layers in a drying room set to 40°C for 16 h. The dried lentils were then divided into approximately 1200 g samples and placed in plastic zip-lock bags. Distilled water was then added to the 1200 g samples to simulate the rewetting cycle and left for 16 h to absorb the moisture. The moisture content rise in lentils was approximately 4% (db). This drying and wetting procedure was repeated 3 times for each sample.
Experimental setup and procedure
The water bath was allowed to operate until the water and apparatus reached the desired temperature. Experiments were conducted at 5, 10, 15, 20, 25, and 30°C for the three “fresh” varieties and at 10, 15, 20, 25, and 30°C for preconditioned (rewetted/dried and freeze/thawed) samples.

For each sample column, the 10 trays were loaded with 20 g of preconditioned lentils. Each tray stack contained alternating layers of high and low moisture content lentils, allowing for measurement of adsorption and desorption EMC values upon completion of the experiment. The moisture content difference between the high and low moisture content layers was approximately 3% (db). A rubber gasket was placed between each tray to stop air from escaping from the sample column. Each tray was labeled to enable sample identification at the end of the experiment.

Five sample columns were prepared, each having different average lentil moisture content, which was done to produce five different ERH/EMC values. Typically the lentil moisture contents ranged between 8 to 22% (db). The samples columns were placed within the apparatus with the top caps screwed on tightly to compress and seal the sample tray gaskets. The plastic air tubes were then connected to the air pumps. Next, the RH and thermocouple sensors were plugged into the data acquisition device.

The Benchlink data acquisition program was set to record the RH and temperature data for each column at 15 min intervals. Finally, the air pumps were engaged and the water bath controlling the headspace radiator’s temperature was adjusted to maintain the desired headspace temperature.

The RH of the sample columns was monitored periodically until the change in RH over a period of minimum 4 h remained relatively constant. The constant RH indicated that equilibrium had been achieved, and the apparatus was disassembled and the moisture content of the lentils in each tray was measured to determine EMC.

Modeling of sorption characteristics
Gervais (2007) used a mathematical model to describe the EMC-ERH characteristics of red lentils and compared four major isotherm equations commonly used in the modeling of EMC-ERH relationships in biological materials. Of the four equations, the Modified Halsey and Modified Oswin equations were found most suitable for the description of lentil moisture relationships (Barrozo et al. 2000). The Modified Halsey equation was chosen for this study since it is used in the ASABE standard (D245.5) for other pulse crops. The Modified Halsey equation is of the following form:

\[
RH = \exp \left[ \frac{-\exp(A + BT)}{(MC)^C} \right]
\]

where:

\[
T = \text{temperature [°C]},
\]

\[
RH = \text{equilibrium relative humidity [decimal]},
\]

\[
MC = \text{moisture content corresponding to equilibrium moisture [% d.b.],}
\]

\[
A, B, C = \text{constants determined based on the experiments.}
\]

Statistical analysis
A non-linear regression procedure was used to calculate the A, B, and C constants of the above equation. The experimental data was fit to the model using SigmaStat statistical software (SysStat Software Inc., San Jose, CA), which employs the Marquardt non-linear regression algorithm.

The non-linear regression procedure was performed on both the adsorption and desorption data collected for each variety and treatment combination. Additionally, the adsorption and desorption data sets for each variety were combined and modeled to produce a mixed form that is appropriate for use as a general model when the direction of moisture transfer is unknown or one of the variables is unknown.

RESULTS AND DISCUSSION
Experiments conducted using the ERH procedure produced data sets for each lentil variety and treatment. The ERH value for each trial was found by calculating the average RH value over a 1 h period after equilibrium was achieved. The EMC values are the average of the five MC values obtained for both adsorption and desorption for a given temperature and ERH.

Most hydroscopic materials have a different EMC depending on whether the material in question came into equilibrium through either adsorption or desorption. This effect is referred to as hysteresis and can impact the material’s fitness for milling or dehulling (Multon et al. 1981). A one-way ANOVA was done on the adsorption and desorption EMC data for each ERH value. All comparisons were conducted using the Student-Newman-Keuls method with a P-value of 0.05. The results of the ANOVA indicate that in the majority of cases, there is a significant difference between the adsorption and desorption EMC values. The difference between the adsorption and desorption EMC values were between 0.1% and 0.6% (db). This confirms that red lentils exhibit the hysteresis effect during adsorption and desorption.

Table 1 shows the results of the non-linear regression on the EMC-ERH data collected for fresh samples of CDC Blaze, CDC Redberry and CDC Robin. The coefficient of determination for non-linear regression (R²) ranged between 0.952 and 0.982, while the standard error of the estimated RH value was within 2.8 to 4.2%. These statistical parameters indicate that the Modified Halsey equation combined with the appropriate coefficients, are a sufficient fit to the experimental data gathered in these experiments. Generally, the model is valid between 5-30°C and 27-90% RH. The results of the non-linear regression procedure for the freeze/thaw treated lentils and the successively rewetted lentils are shown in Tables 2 and 3, respectively.
There is a significant difference between the fresh and freeze/thaw EMC models for all three red lentil varieties studied (Fig. 2-4). This suggests that lentils exposed to freezing and thawing cycles during storage have different EMC-ERH characteristics than fresh lentils. For each lentil variety, the predicted EMC value for the freeze/thaw

Since a direct statistical comparison of the sorption data collected for each treatment would be impractical, a comparison of the models appears to be a reasonable method to determine if temperature and moisture treatments affect red lentil EMC-ERH characteristics.

A graphical method of model differentiation involves plotting the 95% upper and lower confidence intervals for each model. Two models with overlapping confidence intervals would suggest that the models are not significantly different. Conversely, models without overlap over a significant range of predicted values could be interpreted as being distinct. This would be an indication that the two data sets represented by the models were significantly different.

Figures 2 to 4 show superimposed plots of the upper and lower 95% confidence intervals of the three treatment sorption models for each lentil variety. The constants A, B and C used in Eq. 1 were determined based on combined desorption and adsorption experimental data and are named in Tables 1-3 as “Mixed”. The confidence intervals are for the predicted 20°C isotherms. The simulation results are presented only for the range of the conducted experiments in this study.

Of the three red lentil varieties studied, the CDC Redberry and CDC Blaze lentils produced a significant change in EMC-ERH characteristics between the fresh and rewetted sorption models. In this case, the difference in sorption values was observed above 60% RH. The CDC Robin varieties did not show any change in modeled EMC, indicating that successive wetting and drying have little effect on the EMC-ERH characteristics of the CDC Robin red lentils.

There is a significant difference between the fresh and freeze/thaw EMC models for all three red lentil varieties studied (Fig. 2-4). This suggests that lentils exposed to freezing and thawing cycles during storage have different EMC-ERH characteristics than fresh lentils. For each lentil variety, the predicted EMC value for the freeze/thaw
### Table 1. Calculated constants for the Modified Halsey equation for combined three varieties of fresh red lentils.

<table>
<thead>
<tr>
<th>Variety</th>
<th>Isotherm Equation Constants</th>
<th>Statistical Parameters</th>
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</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>CDC Robin</td>
<td></td>
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</tr>
<tr>
<td>Desorption</td>
<td>5.520 ±0.224</td>
<td>-0.0160 ±0.0026</td>
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<tr>
<td>Adsorption</td>
<td>5.287 ±0.222</td>
<td>-0.0143 ±0.0026</td>
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<tr>
<td>Mixed</td>
<td>5.390 ±0.159</td>
<td>-0.0150 ±0.0019</td>
</tr>
<tr>
<td>CDC Redberry</td>
<td></td>
<td></td>
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<tr>
<td>Desorption</td>
<td>4.987 ±0.384</td>
<td>-0.0138 ±0.0052</td>
</tr>
<tr>
<td>Adsorption</td>
<td>4.572 ±0.355</td>
<td>-0.0100 ±0.0050</td>
</tr>
<tr>
<td>Mixed</td>
<td>4.749 ±0.260</td>
<td>-0.0116 ±0.0036</td>
</tr>
<tr>
<td>CDC Blaze</td>
<td></td>
<td></td>
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<tr>
<td>Desorption</td>
<td>5.380 ±0.289</td>
<td>-0.0080 ±0.0040</td>
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<tr>
<td>Adsorption</td>
<td>5.023 ±0.245</td>
<td>-0.0056 ±0.0035</td>
</tr>
<tr>
<td>Mixed</td>
<td>5.176 ±0.194</td>
<td>-0.0065 ±0.0027</td>
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</table>

^a standard error of estimated value

^b coefficient of determination for nonlinear regression

### Table 2. Calculated constants for the Modified Halsey equation for freeze/thaw treated red lentils.

<table>
<thead>
<tr>
<th>Variety</th>
<th>Isotherm Equation Constants</th>
<th>Statistical Parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>CDC Robin</td>
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<td></td>
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<tr>
<td>Desorption</td>
<td>5.713 ±0.298</td>
<td>-0.0077 ±0.0038</td>
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<tr>
<td>Adsorption</td>
<td>5.307 ±0.309</td>
<td>-0.0082 ±0.0042</td>
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<tr>
<td>Mixed</td>
<td>5.480 ±0.221</td>
<td>-0.0079 ±0.0029</td>
</tr>
<tr>
<td>CDC Redberry</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Desorption</td>
<td>5.801 ±0.424</td>
<td>-0.0075 ±0.0049</td>
</tr>
<tr>
<td>Adsorption</td>
<td>5.385 ±0.505</td>
<td>-0.0032 ±0.0060</td>
</tr>
<tr>
<td>Mixed</td>
<td>5.562 ±0.331</td>
<td>-0.0052 ±0.0039</td>
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<tr>
<td>CDC Blaze</td>
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<tr>
<td>Desorption</td>
<td>5.510 ±0.489</td>
<td>0.0057 ±0.0059</td>
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<tr>
<td>Adsorption</td>
<td>4.558 ±0.469</td>
<td>0.0125 ±0.0064</td>
</tr>
<tr>
<td>Mixed</td>
<td>4.922 ±0.351</td>
<td>0.0096 ±0.0045</td>
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</tbody>
</table>

^a standard error of estimated value

^b coefficient of determination for nonlinear regression

### Table 3. Calculated constants for the Modified Halsey equation for successively rewetted and dried red lentils.

<table>
<thead>
<tr>
<th>Variety</th>
<th>Isotherm Equation Constants</th>
<th>Statistical Parameters</th>
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<tbody>
<tr>
<td></td>
<td>A</td>
<td>B</td>
</tr>
<tr>
<td>CDC Robin</td>
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<tr>
<td>Desorption</td>
<td>5.002 ±0.424</td>
<td>-0.0055 ±0.0052</td>
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<td>Adsorption</td>
<td>4.653 ±0.382</td>
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<tr>
<td>Mixed</td>
<td>4.787 ±0.285</td>
<td>-0.0025 ±0.0036</td>
</tr>
<tr>
<td>CDC Redberry</td>
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<td></td>
</tr>
<tr>
<td>Desorption</td>
<td>6.444 ±0.293</td>
<td>-0.0013 ±0.0033</td>
</tr>
<tr>
<td>Adsorption</td>
<td>5.642 ±0.253</td>
<td>0.0018 ±0.0033</td>
</tr>
<tr>
<td>Mixed</td>
<td>5.968 ±0.212</td>
<td>0.0003 ±0.0026</td>
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<tr>
<td>CDC Blaze</td>
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<td></td>
</tr>
<tr>
<td>Desorption</td>
<td>6.690 ±0.373</td>
<td>-0.0012±0.0040</td>
</tr>
<tr>
<td>Adsorption</td>
<td>5.644 ±0.310</td>
<td>0.0027 ±0.0039</td>
</tr>
<tr>
<td>Mixed</td>
<td>6.011 ±0.270</td>
<td>0.0010 ±0.0032</td>
</tr>
</tbody>
</table>

^a standard error of estimated value

^b coefficient of determination for nonlinear regression
treated lentils was lower than the predicted EMC value for freshly harvested lentils at a given ERH. The difference in EMC values between the two treatments is most significant at high RH levels. In the most extreme case shown in Fig. 2-3 for CDC Redberry and CDC Blaze lentils, the EMC value for freeze/thaw treated lentils is 4% (db) lower than for fresh untreated lentils.

Lowering the EMC values for lentils exposed to repeated freezing and thawing cycles might be beneficial, since exposure to high RH conditions would be less likely to raise the lentil moisture content to the point where spoilage might occur due to mould and insect growth.

CONCLUSIONS
The EMC characteristics of three varieties of red lentils (Robin, Blaze, and Redberry) tested between 5 and 30°C after being exposed to drying/wetting and freeze/thaw cycles exhibited a hysteresis effect. The differences between the adsorption and desorption values were statistically different (P=0.05) and ranged from 0.1 to 0.6% (db).

The Modified-Halsey equation was successfully used to mathematically model the sorption characteristics of the lentils. Of the three red lentil varieties studied, a significant change in EMC-ERH between the fresh and rewetted samples for CDC Redberry and CDC Blaze was observed above 60% RH. However, successive wetting and drying of lentils had little or no effect on the EMC-ERH characteristics of CDC Robin varieties. There was a significant difference in the results between the fresh and freeze/thaw EMC models for all three red lentil varieties studied. For each lentil variety, the predicted EMC value for the freeze/thaw treated lentils was lower than the predicted EMC value for freshly harvested lentils at a given ERH. In the most extreme case the EMC value for freeze/thaw treated lentils at high moisture was 4% (db) lower than for fresh untreated lentils.

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