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Temporal Evaluation of Corn Respiration Rates Using Pressure Sensors

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Temporal Evaluation of Corn Respiration Rates Using Pressure Sensors

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ABSTRACT

High respiration rate of a grain indicates faster degradation of its dry matter. Proper grain management requires chronological and precise measurements of carbon dioxide evolved from grain respiration during the postharvest storage duration. Therefore the main goal of this research was to develop a new technique that evaluates temporal corn respiration rate using pressure sensors. It was based on measuring pressure drop associated with the grain respiration in a closed container and using it to calculate the grain respiration rates.

Dry corn (Zea Mays L.) was procured from a local farmer and stored at 4ºC. Corn rewetting technique was applied A set of eighteen pressure sensor modules were used throughout the course of this study. These modules determine gas productivity for various biological cultures through measuring and recording the cumulative pressure as well as temperatures. A 150-gram subsample from each moisture level was placed in a glass bottle along with two NaOH vials as well as one silica gel vial. The effects of corn storage temperature (23, 35, and 45ºC) and initial moisture content (12.9, 14.8, 17.0, 18.8, and 20.7% w.b) on the cumulative respiration and respiration rate were studied for duration of nine days.

The pressure sensor method was found to be reliable in measuring corn respiration rate as affected by the tested parameters. The highest cumulative respiration of 2.625 g-CO_2/kg-corn was observed at the moisture content of 18.8% and the medium temperature level of 35ºC after nine days. It was also observed that the respiration rate increased with increases in temperature and peaked out at a temperature of 35°C. Respiration rates reached their maximum values of 0.199, 0.755, 0.987, and 1.147 g-CO_2/kg-corn.d under the medium temperature level of 35ºC and the moisture contents of 14.8% (5^{th} day), 17.0% (5^{th} day), 18.8% (3^{rd} day), and 20.9% (2^{nd} day), respectively. Additionally, an empirical equation to predict the cumulative respiration of corn as

affected by storage temperature, moisture content, and storage duration with an adjusted coefficient of determination value of 0.80. This study recommended that respiration of freshly harvested corn exposed to natural air drying samples should be evaluated.

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CHAPTER 1

INTRODUCTION

Harvested grains are living organisms that continue to perform metabolic functions during storage. Their quality starts to deteriorate during storage due to biological, mechanical, and pathological reasons (Bartz and Brecht, 2002). Grain respiration is among the biological reasons. Deterioration of grains is measured by their dry matter loss. According to the ASABE (2005), the maximum allowable dry matter loss is 0.5% under a wide range of storage temperature and moisture content. The values of storage temperature and moisture content are being used to determine the safe storage period of corn. It has been reported that the higher the grain respiration rate, the shorter the safe storage period.

Steele et al. (1969) correlated the dry matter loss with corn respiration. Several grain respiration studies were based on intermittent or destructive measurement of carbon dioxide evolved. There is a lack of continuous measurements of the grain respiration rate with a non-destructive methods. Additionally, there is a limited data correlating the storage temperature, initial moisture content and storage duration based on continuous measurements of corn respiration rate.

Therefore, the main objective of this research was to develop a method for determining grain respiration rate at lab scale based on continuous and non-destructive technique. The technique was developed and was tested for corn. These measurements took place in a closed container holding a known amount of corn and equipped with pressure sensors. During corn respiration, oxygen was consumed and carbon dioxide was produced. The evolved CO_2 was absorbed by sodium hydroxide pellets creating pressure drop in the container. Pressure drop data were collected and interpreted to CO_2 evolved during grain respiration.

The effects of corn storage temperature, initial moisture content and storage duration on the respiration rate were evaluated. Additionally, a numerical model was developed to determine corn respiration as function of storage temperature, corn moisture content, and duration of storage. This model can be used to predict corn respiration rate under the tested environmental conditions.

CHAPTER 2

LITERATURE REVIEW

2.1. Grain respiration

Synthesis of proper grain storage strategies is the key to retaining them active for a longer period without suffering any losses such as weight loss, quality loss, health risk, and economic loss (Chow, 1980). The proper storage procedures and management practices are desirable to follow as reported by Chidananda et al. (2014). One of the indications related to crop activity during their storage, that could be measured independently, is grain respiration. It is a parameter of interest to study because it affects the storage life of the commodity. Respiration is a metabolic process where oxidative breakdown of complex molecules such as sugars or carbohydrates takes place. In other words, during grain respiration, oxygen in the air combined with organic molecules in the grain tissues (typically sugar) to produce various intermediate compounds and ultimately carbon dioxide and water accompanied by generation of heat (Forcier et al., 1987). Liu et al. (2011) stated that respiration has a significant influence on crop yield because dry matter accumulation is strictly related to adjustment of CO_2 and respiratory activity. The respiration of a grain mass under aerobic conditions has been modeled as:

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6H_2O + 2835\, kJ/gram\, mole \quad (1)$$

The previous reaction could be elucidated as one mole of glucose (180 g) reacts with six moles of oxygen (192 g, 6 moles × 32 g/mol) to produce 6 moles of CO_2 (264 g, 6 × 44 g/mol) and 6 moles of H_2O (108 g, 6 ×18 g/mol). It should be mentioned that oxygen will diffuse from the air surrounding the grain to the grain cells, while CO_2 will diffuse out of the grain cells to the air (Steele and Bern, 2002).

2.2. Factors affecting grain respiration

The rate of grain respiration is influenced by internal as well as external factors. The type and maturity of the grain are the internal factors affecting its respiration rate. Respiration rate typically declines as the crop matures; commodity harvested at active growth stage respires faster than the one harvested after attaining complete maturity (Saltveit, 2004).

On the other hand, moisture content and temperature as well as oxygen and carbon dioxide concentrations are the major external factors affecting grain respiration rate. Fonseca et al. (2002) and Gonzales et al. (2009) reported that moisture content and temperature of stored grain are the main factors affecting their deterioration. Dillahunty et al., (2000) reported that the utmost important factor affecting grain respiration is storage temperature.

Brecht (2004) reported that sweetcorn respiration rate increased from 30-51 mg-CO_2/kg-corn.h at 0°C to 282-435 mg-CO_2/kg-corn.h at 25°C. Bunce (2004) showed that the low temperature reduced rates of respiration while high temperature increased rates of respiration. Accordingly, increases in grain temperature increases carbon dioxide production rate. This is proper up to a certain temperature level, after that the CO_2 production rates will decline (Dillahunty et al., 2000).

Karunakaran et al. (2001) mentioned that respiration rates of 17-19% moisture content wheat at 25ºC were higher than the 15-16% moisture content wheat stored at the same temperature. Reed et al. (2007) measured the rates of O_2 consumption and CO_2 production of re-wetted corn at three different moisture contents over a 60-day period. They reported that O_2 consumption rates of 0.5, 5 and 18 mg-O_2/kg·h for 15.0, 16.6 and 18.0% moisture corn, respectively. These linked to 1.4, 13.7 and 49.5 mg-CO_2/kg-wheat·h, respectively. Even with

small differences in moisture content, a wide and nonlinear range of O_2 consumption and CO_2 production were observed.

Chidananda et al. (2014) measured respiration rate as affected by moisture contents and temperatures for pinto bean, chickpea, and green lentils stored for one month. They found that respiration rates increased with increase in moisture content and temperature. Additionally, they found that respiration rate was positively correlated with final moisture content and free fatty acid value and negatively correlated with seed germination. Srour (1988) found that grain respiration increased in an exponential manner with increase in temperature and moisture content. High moisture accompanied by high temperature provides the optimum conditions for fungi to grow (Chow, 1980).

Oxygen and carbon dioxide concentrations, in storage tanks, also influence grain respiration rate. Decreased availability of O_2 slows down the respiration activity due to reduced metabolic activity. Similarly high CO_2 concentration in the atmospheres surrounding the grain, reduce the respiration metabolism as reported by Herner (1987). High carbon dioxide concentration had been proved as a biological activity responsible for spoilage of stored wheat (White et al., 1982).

Karunakaran et al. (2001) observed that germination dropped from 98 to 92-89% when dry matter loss was about 0.05% and visible mold occurred when the dry matter loss was about 0.1% in wheat sample having moisture content of 17-19%. They also reported that deterioration rates were determined by measuring germination capacity of grain and respiration rates of grain and microorganisms. Maier et al. (2010) found that insects, fungi, and grain metabolism cause the elevated CO_2 levels in storage tanks. Accordingly, monitoring CO_2 levels help detecting spoilage early.

Respiration rate of biological material is closely tied to its metabolic activity, which is influenced by both biotic and abiotic factors. As a result, microbial and grain respiration contribute to the total respiration in the system (Christensen, 1955). High respiration rate under high moisture provides a suitable environment for *Aspergillus flavors* to flourish and produce aflatoxins. Aflatoxins have been regarded by IARC (International Agency for Research on Cancer) as toxicity class I chemicals and carcinogenic in nature (Piotrowska et al., 2013). Chitrakar et al. (2006) reported that carbon dioxide generation during aerobic respiration is a useful measure of the microbes' activities that decompose organic materials. Hence, it is essential to measure the CO_2 concentration dynamically to monitor the respiration rate of the grains. Accordingly, these factors might be monitored and controlled to maintain grain quality and to quantify any mold growth and insect infestation.

2.3. Grain respiration measurements techniques

Various techniques had been developed during the last few decades to quantify grain respiration. The commonly used methods to measure CO_2 production are divided into static and dynamic system measurements. In the static systems, grain is placed in some airtight containers and the measurements are done either by drawing gas samples continuously or by monitoring gas concentration with some sensing device. Sufficient amount of CO_2 has to accumulate to be allow accurate detection by any one of a number of commercially available instruments that are being used to measure CO_2 concentration. Typically, gas chromatograph and infrared CO_2 analyzers are being used to quantify CO_2 concentration. Milner and Geddes (1945) reported that the concentration of CO_2 should increase linearly with time if the container is properly sealed. CO_2 production rate could be calculated by multiplying the change in concentration times the

container volume and dividing by grain weight and duration of time. It is obvious that the static systems are more suitable for small sample sizes and short test durations.

Alternatively, in a dynamic system, the difference in CO_2 concentration between the exit and inlet air represent the respiration during this period (Saltveit, 2004). Typically, the dynamic system consists of three components including air conditioning and flow management system, grain column, and moisture removal and CO_2 measurement equipment. Air passed through the container at a known flow rate. Mathematically, the system will come into equilibrium in about the same time it takes for 5 times the volume to flow through the container. Following, CO_2 concentration at the inlet and outlet is measured by taking gas samples. To determine the respiration rate, the difference in concentration should be multiplied by the gas flow rate and divided by the weight of the grain. Longer CO_2 monitoring could be achieved using the dynamic systems. The grain sample is placed in a container with continuous ventilation at a known flow air rate. The CO_2 production rate is determined by sampling the gas at inlet or outlet and testing for CO_2 concentration using gas analyzers (Campo et al., 2014).

Grain respiration measurements had been typically quantified directly or indirectly. Physical and chemical measurements are the base for the direct methods, in which physical methods have been considered as more efficient. Direct methods usually measure the concentration of carbon dioxide (CO_2) produced directly from the grain. On the other hand, indirect methods measure some properties associated with the crop, such as pressure or volume changes within a closed bottle, which is correlated with the concentration of CO_2 and O_2 liberated and consumed, respectively.

CO_2 concentrations are determined gravimetrically or by using a non-dispersive infrared (NDIR) sensor or gas analyzer. In the gravimetric method, the produced CO_2 passes through an

adsorbent material and the increased mass of the adsorbent material is monitored over time. Often, the mass change is directly related to the amount of respired CO_2, and interpreted as the grain dry matter loss (Fawole, 1969). Dynamic systems with gravimetric CO_2 measurements have been used comprehensively for monitoring corn respiration rate (White et al., 2010 and Campo et al., 2014). Corn respiration tests were performed for four days and the CO_2 production rate was determined. The advantages of the gravimetric method include the use of inexpensive raw materials, high instrumentation accuracy and resolution. The disadvantages of this method include measurements at discrete time points (Nuckols et al., 1983).

Al-Yahya et al. (1993) used column study to determine the corn respiration rate. They used KOH to capture the produced CO_2. They assumed that all the produced CO_2 was captured by KOH. Following, they multiplied the dry matter loss times the carbon dioxide molecular weight and divided by the glucose molecular weight to determine the mass of the produced CO_2.

Chitrakar et al. (2006) and Haney et al. (2008) reported that pH-sensitive, color-changing Solvita® gel kits could be used to measure CO_2 levels, generated during respiration of organic materials, via absorption. The Solvita® Corn Testing Procedure, used by the former authors, was shown to be capable of monitoring the storage state of corn over a range of moistures and durations of incubation after re-wetting. They sealed the measuring kits along with corn at 20% m.c. and noticed a change in color of the measuring kit after 4 hr period. A linear relationship was reported between corn m.c. and the amount of CO_2 produced, and an exponential relationship was observed between the amount of CO_2 produced and storage times. These kits are not expensive which could be considered an advantage of this technique. On the other hand, the interference from volatile fatty acids can result in incorrect reactions for CO_2 concentrations.

Additionally, these kits are highly sensitive to temperature so their use is restricted to the range between 20 and 25°C.

Computer controlled automatic respirometer, using manometry as a fundamental principle, was designed to access the respiration of biological materials (Janni et al., 1981). In another experiment, an electronic sensor was devised, and its credibility was established by comparing its results with gas chromatography results. The results did not show any significant difference (0.05 level) between the two techniques (Forcier et al., 1987). Chidananda et al. (2014) also, used the gas chromatographic technique to measure the respiration rate of pulses under different environmental conditions. Hamer et al. (1991) fabricated an automatic electrolytic respirometer, which measured the current flowing through the electrolytic solution, and obtained the volume of O_2 consumed in the process. In addition, a resistance-based sensor has been developed to detect the spoilage in stored grains, employing polyaniline boronic acid polymer as sensing element (Neethirajan et al., 2009). The developed sensor measured gaseous CO_2 levels in the range of 380-2400 ppm of CO_2 concentration levels. The sensor was evaluated for the influence of temperature and relative humidity and found to be reliable and repeatable in reporting CO_2 concentration at various temperature and relative humidity levels.

In an earlier study, Sadaka et al. (2006) used automated pressure sensors to measure respiration of organic materials. This method used the pressure drop due to O_2 consumption in the container for calculating respiration rate. The CO_2 produced was trapped with sodium hydroxide (NaOH) pellets to eliminate its effect on the pressure. The readings were compared with time-tested titration method and found to have a high degree of agreement (R^2=0.92).

2.4. *Significance and objectives of the study*

As mentioned earlier, the safe storage life of grain varies inversely with the rate of respiration. Most of the research has been attentive on measuring the respiration intermittently. Grain respiration activity in the storage bins may change suddenly. Accordingly, intermittent respiration measurements may not allow tracking the changes in the respiratory behavior of the crop. These intermittent respiration measurements could lead to inappropriate decision making, and the recorded data might not be entirely representative. The major issue facing grain-handling management is that the hotspots may occur far away from temperature sensors in bins, silos, and tanks making early detection of spoilage difficult. Consequently, there is a rising demand for continuous sensing of carbon dioxide (CO_2) during grain storage. This is because CO_2 sensors can be used to detect spoilage and to assess CO_2 levels in modified atmosphere storage structures as reported by Neethirajan et al. (2009). The market perspective for reliable and inexpensive CO_2 sensors is enormous because of a broad range of applications in the agri-food industry.

Therefore, there is a need to develop a technique for continuous monitoring of respiration. Hence, the goal of this study was to establish an innovative method to evaluate corn respiration rate on temporal basis. The specific objectives were to: (a) study the effects of temperature, moisture content, and storage duration on grain respiration rate; and (b) quantify the corn cumulative respiration as a function of storage temperature, moisture content, and storage duration.

CHAPTER 3

MATERIALS AND METHODS

3.1. Corn collection and preparation

Dry corn (Zea Mays L.) was procured from a local farmer and stored at 4ºC. This corn was harvested about nine months earlier. The initial moisture content of corn was determined using the standard method (ASABE, 2008). 10 kg corn sample was visually selected to avoid any damaged corn kernels. The sample was divided into five subsamples and stored in polyethylene bags. The targeted moisture content levels were 13, 15, 17, 19, and 21% w.b. The required amount of distilled water was added to the corn in the polyethylene bag with the help of spray bottles. Corn rewetting technique was found to be an acceptable method of storing the corn for further use in storage studies. Fernandez et al. (1985) reported that no significant difference in CO_2 concentration was observed between the dried-rewetted corn and the samples stored under refrigeration at same moisture level. Following, the samples in the zip lock bags were mixed vigorously to ensure uniform distribution of the added moisture. These five subsamples were again stored in the refrigerator at 4ºC. Thereafter, three subsamples were collected from each bag to determine the final moisture content. The moisture contents of the samples were found to be 12.9%, 14.8%, 17.0%, 18.8%, and 20.7% w.b.

3.2. Pressure sensor modules

In addition to a zero calibrated module, a set of eighteen pressure sensor modules (Ankom Technology, Macedon, NY, USA), were used throughout the course of this study. These modules are used to determine gas productivity for various biological cultures through measuring and recording the cumulative pressure. They also measure and record the temperatures for the

duration of the experiments. These modules are able to measure the cumulative pressure range between -68.9 kPa to 3,447.4 kPa with the accuracy of ±1% of the measured value. Each module consists of a 1-liter glass bottle, and a top compartment, which contains the pressure and temperature sensors. A zero module composed of a sensor's compartment only (no bottle) is used as a zero (no-pressure) measurement. Figure 3-1 shows photos of the experimental setup. The readings are communicated via radio frequency (RF) signal to an antenna that connects to PC using a USB connection to enable live plotting and viewing in addition to data recording in a tabulated format. A set of 36 25-ml vials containing 8 gram of NaOH pellets each and 18 25-ml vials containing 5 gram of silica gel crystals were prepared and covered until needed. NaOH and silica gel were used, in this study, to absorb carbon dioxide and water vapor, respectively produced during the respiration process.

A 150-gram subsample from each moisture level was placed in a glass bottle along with two NaOH vials as well as one silica gel vial. This amount of grain was selected to ensure that headspace volume is large enough as compared with the grain volume to provide aerobic environment throughout the course of the experiment. Before, tightening the modules over to the bottle, the vent valve was closed and the rubber gasket was properly greased with vacuum grease to ensure no leakage during the experiment. The top compartment was then secured on to the glass bottle tightly. Three replicates were tested for each moisture level. In addition to the 15 pressure modules containing corn, a set of three empty modules containing only NaOH and silica gel vials were also prepared. This set of control was used to take into account the amount of carbon dioxide existing in the atmosphere. The previously prepared modules were placed in a programmable incubator (Model No: IE75-5A, So-Low Environmental Equipment, Cincinnati, OH) adjusted to the desired temperature level. Three temperature levels (23, 35, and 45ºC) were

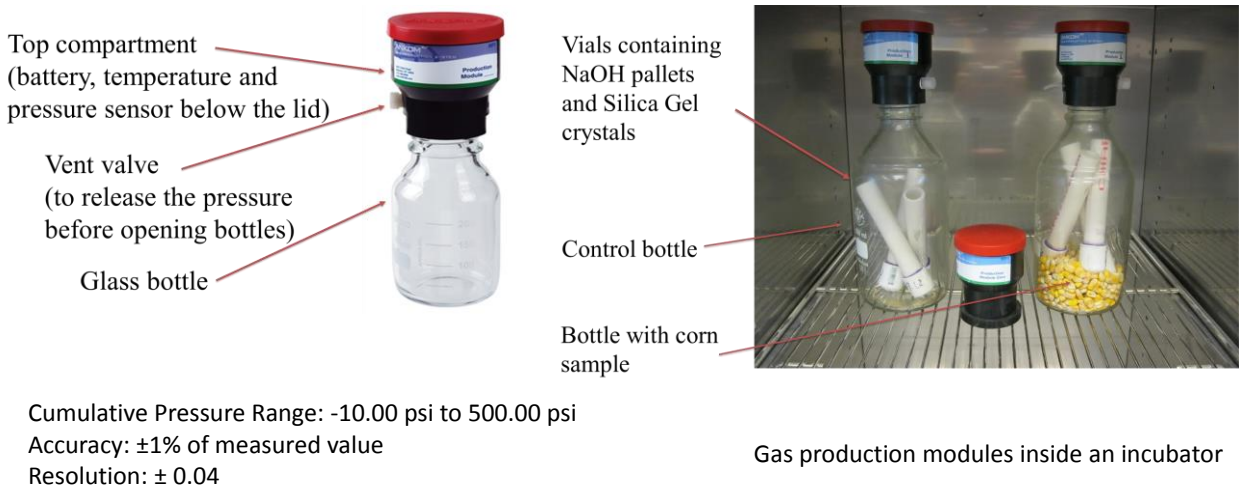

Figure 3-1. Experimental setup.

selected to test the effect of temperature on respiration rate. Accordingly, the same preparation technique was repeated at each temperature level.

3.3. Data recording using gas pressure monitoring system

The previously described modules communicated the data to the computer that has the Gas Pressure Monitoring (GPM) software running (Ankom Technology, Macedon, NY). This software can record absolute pressure, cumulative pressure, and temperature on a continuous basis at the preselected time intervals.

3.4. Determination of respiration rate from pressure drop data

The amount of carbon dioxide evolved during grain respiration is bound by the alkaline pellets (sodium hydroxide) absorbing agent according to the following chemical reaction:

$$CO_2 + 2NaOH \rightarrow Na_2CO_3 + H_2O \quad (2)$$

The difference between the room temperature and the incubator temperature affect the pressure in the module. The correlation between the pressure rise and the temperature difference can be calculated based on the ideal gas law as follows:

$$\Delta P = \frac{n \times R \times \Delta T}{V} \quad (3)$$

where,

ΔP is the pressure rise, [kPa]

n is the number of moles of substance, [kmol]

R is the general gas constant, [8.134, kJ/kmol. ºK]

ΔT is the changes in the gas temperature, [°K]

V is the gas volume, [m^3]

After temperature had equilibrated in the incubator, subsequent carbon dioxide absorption by sodium hydroxide pellets created a pressure drop as measured by the sensor. The correlation between changes in a number of moles of substance and pressure drop is given by the following equation (note that the process is isothermal):

$$\Delta n = \frac{\Delta P_{isoth} \times V}{R \times T} \quad (4)$$

where,

Δn is the changes in the number of moles of substance, [kmol]

ΔP_{isoth} is the pressure drop under isothermal condition, (determined by the difference between the maximum and final pressure [kPa]

T is the gas temperature, [ºK]

The amount of substance is derived from the quotient, m/M, and consequently, the equation can be written as follows:

$$\Delta m = \frac{\Delta P_{isothermal} \times V \times M_R}{R \times T} \quad (5)$$

where,

Δm is the amount of substance, [g]

M_R is the molecular weight of the substance, [44 g-CO_2/mol]

The mass of carbon dioxide evolved during the incubation period from (w) g solids, can be calculated as follows:

$$CO_2 = \frac{\Delta P_{isothermal}(pa) \times 1\left(\frac{N}{\frac{m^2}{Pa}}\right) \times V(m^3) \times 44\left(\frac{g}{mole}\right)}{8.314\left(\frac{J}{mole.K}\right) \times 1\left(\frac{N.m}{J}\right) \times T(K) \times w(kg)} \quad (6)$$

where,

CO_2 is the evolved carbon dioxide [g-CO_2/kg-corn]

3.5. Experimental design and statistical analysis

The experiment was designed as randomized block design (RBD), the blocks being divided based on temperature (23, 35, and 45°C). Five levels of moisture content (12.9%, 14.8%, 17.0%, 18.8%, and 20.7% w.b.) were taken into account in triplicates to study the respiratory behavior of the corn (Table 3-1). Data were collected for the duration of 9 days at each temperature level. Cumulative pressure data were recorded and converted to respiration rate as described earlier. The results were analyzed using JMP® Pro software (version 11.0.0, SAS Institute Inc., Cary, NC). Two-Way ANOVA was used to analyze the impact of moisture content, storage temperature and storage time on the respiration rate of corn.

Table 3-1. Experimental Design.

Temperature (ºC)	Moisture Content (% w.b.)	Duration (d)	Replicates (-)
23	12.9	1 - 9	R1, R2, R3
	14.8	1 - 9	R1, R2, R3
	17.0	1 - 9	R1, R2, R3
	18.8	1 - 9	R1, R2, R3
	20.7	1 - 9	R1, R2, R3
35	12.9	1 - 9	R1, R2, R3
	14.8	1 - 9	R1, R2, R3
	17.0	1 - 9	R1, R2, R3
	18.8	1 - 9	R1, R2, R3
	20.7	1 - 9	R1, R2, R3
45	12.9	1 - 9	R1, R2, R3
	14.8	1 - 9	R1, R2, R3
	17.0	1 - 9	R1, R2, R3
	18.8	1 - 9	R1, R2, R3
	20.7	1 - 9	R1, R2, R3

CHAPTER 4

RESULTS AND DISCUSSION

4.1. Evaluation of the repeatability of pressure measurements

The repeatability of the pressure measurements was determined in triplicate for each treatment. Figure 4-1 shows an example of the adjacent pressure measurements and another example of the nonadjacent pressure measurement. One-way analysis of variance was performed to determine the significance among the means of the triplicate treatments. Majority of the triplicated pressure measurements (78.8% of the treatments) showed significant differences between their means ($p<0.05$). The significant differences between the means of the nonadjacent treatments may be attributed to the natural behavior of living organism. The bottle location inside the incubator, which has a thin glass door, may have some effect of the heat loss. Additionally, not all the living organisms consume the exact amount of oxygen or evolve the exact amount of carbon dioxide in the same environment. It was observed that some replicates have temperature differences of 1-2 degrees. As a result, however, 22.2% of the treatments showed insignificant differences between their means, it was decided to utilize the average pressure values among the three treatments for truthful results.

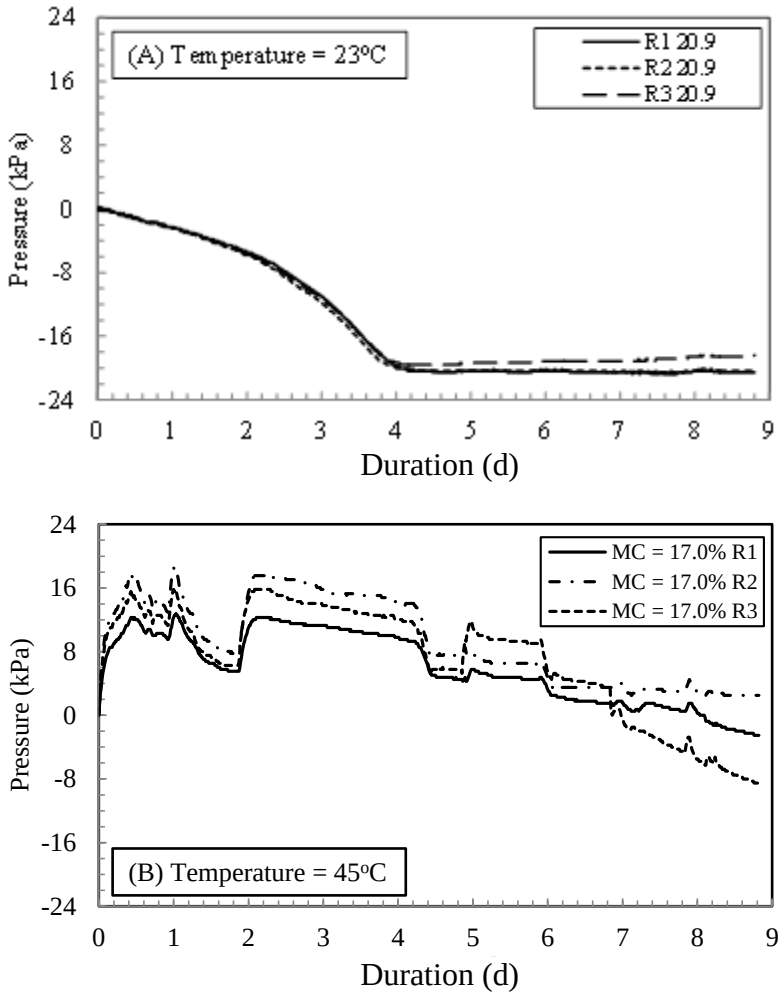

Figure 4-1. Temporal cumulative pressure showing examples of the three triplicate measurements at two temperature levels (23 and 45ºC).

R1, R2 and R3 are replicates 1, 2 and 3, respectively.

4.2. Temperature profiles

Biological activities, like respiration, are temperature dependent. Accordingly, the temperature profiles, in the pressure modules, were monitored and recorded every 15 minutes. The average values of temperatures were recorded during the steady state operation for the duration of the experiment of 9 days. Figure 4-2 shows the temporal profiles of the mean modules temperatures for the three predetermined temperature levels. This graph depicts three distinguished levels of temperature, namely low (23°C), medium (35°C), and high (45°C). Throughout the course of the three experimental runs, the temperature profiles were found to be relatively steady with some fluctuations in the high-temperature level. The temperature ranged between 41.1°C and 45.7°C under the high-temperature level. On the other hand, the temperature ranged between 32.8°C and 38.8°C and 21.7°C and 23.4°C under the medium and low-temperature levels, respectively. The high fluctuation of the temperatures during high-temperature experimental run of 45°C as compared to that of the low experimental run of 23°C could be attributed to the very short time power outage. There was always negligible difference between the room temperature and the module temperature at a low level since the room temperature was set to about 23°C. Conversely, the 22 degrees difference between the incubator temperature of 45°C at the high level and the room temperature resulted in a small temperature fluctuation. Some readings were slightly greater than the incubator temperature. This slight increase in the temperature could be attributed to the heat released from respiration activities as shown previously in equation 1.

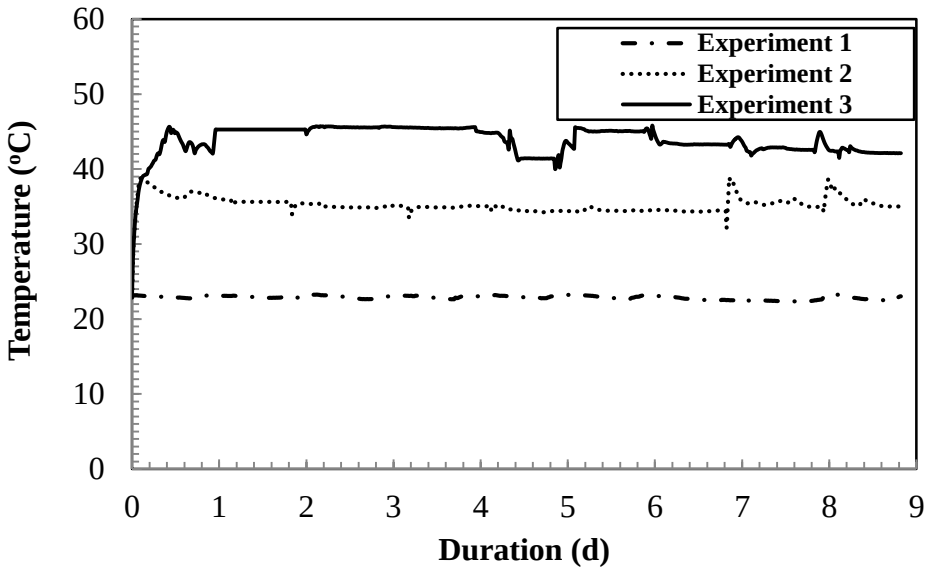

Figure 4-2. Temperature profile during the experimental runs

4.3. Effects of corn moisture content, storage temperature, and storage duration on cumulative pressure

Immediately after placing the pressure modules in the incubator, the temperature increased until it equilibrated with the incubator temperature resulting in a rise in the module pressure. This phenomenon is attributed to an increase in the air temperature subjected to a constant module volume. The pressure sensors communicated the pressure data to the computer. The gas pressure monitoring (GPM) software tabulated the pressure data and provided the cumulative pressure readings. Following the initial increase, the pressure started to drop and varied based on the corn moisture content and the incubator temperature as shown in Figure 4-3. Sodium hydroxide placed in the vials captured the produced CO_2 and thus the drop in the pressure was simply due to the oxygen consumption in the respiration process. The pressure in the control module (no corn) also decreased due to the absorption of carbon dioxide presented initially in the air. It can be seen that the control treatment did not undergo considerable pressure drop. The maximum value of the pressure drop was obtained for each temperature level. Subsequently, the pressure drop readings after every 24 h interval was found and subtracted from the maximum value of the cumulative pressure at the starting of the experiment. Afterward, the pressure drop readings of control treatment were subtracted from the cumulative pressure readings. This quantity was referred to as $\Delta P_{isothermal}$ and was used in the calculations of respiration rate. An increase in the pressure drop is attributed to the consumption of oxygen that exists in the air. It should be mentioned one more time that the respiration process released carbon dioxide, which was absorbed by sodium hydroxide pellets.

The pressure drop increased with increase in the corn moisture content under the three tested temperature levels. The maximum pressure drop of 22.9 kPa was observed under the medium temperature level of 35°C and the moisture content of 18.8%, which started on the fifth day until the end of the experimental run. On the contrary, the lowest pressure drop of 1.1 kPa was observed under the lowest temperature level of 23°C and the moisture content levels of 14.8%. On the ninth day, the pressure drop reached its maximum values at the medium temperature level of 35°C. Pressure drop levels under the high-temperature levels of 45°C were lower than that of the medium temperature level. The smallest recorded pressure drop levels were reported at the lowest temperature level of 23°C.

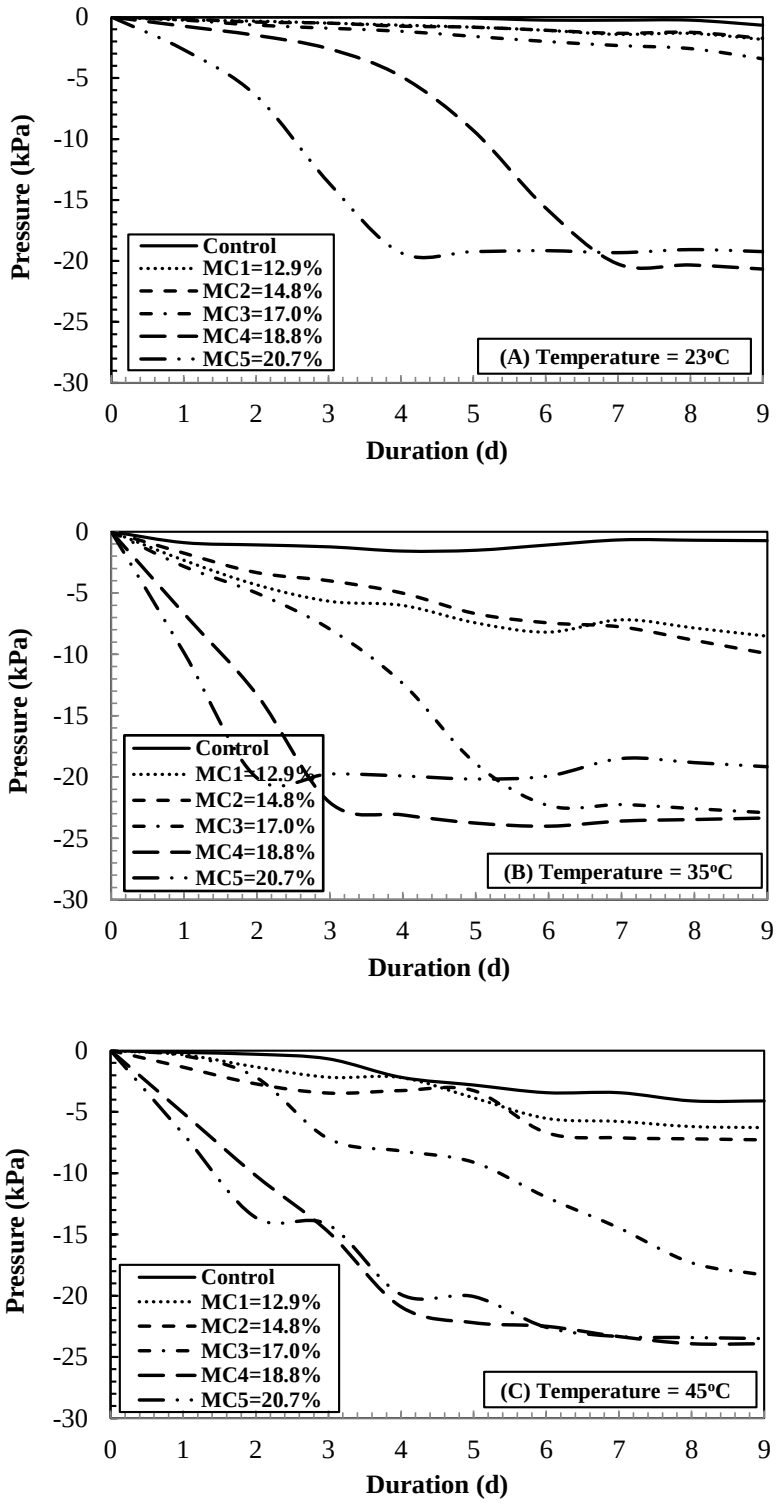

Figure 4-3. Temporal cumulative pressure drop as affected by corn initial moisture content and storage temperature

4.4. Effects of corn storage temperature, moisture content, and duration on cumulative respiration

Figure 4-4 shows the impact of corn moisture content, storage temperature, and storage duration on the cumulative respiration. The cumulative respiration is associated with the pressure drop in the module. As a result, there are some similarities between the effects of temperature, moisture content, and storage duration on the modules pressure drop values and their corresponding values of cumulative respiration. It is clear that there is a substantial effect of moisture content and temperature on the cumulative respiration. It is evident from the parabolic curves of the higher levels of moisture content under the three temperature levels; i.e.; low, medium, and high. Statistical analysis showed a significant effect of corn moisture content, storage temperature and time of the corn cumulative respiration ($P<0.0001$). Increased cumulative respiration was observed with increases in the temperature levels. The cumulative respiration reached 2.391, 2.625, and 2.219 g-CO_2/kg-corn after nine days at the moisture content of 18.8% and storage temperatures of 23, 35, and 45^{o}C, respectively. Similar trends were observed for all the studied moisture content levels where the highest cumulative respiration values were found under the medium temperature. The only exception to this observation was found at the highest level of moisture content (20.7%) and the longest storage duration. The cumulative respiration in this case reached 2.302, 2.175, and 2.078 g-CO_2/kg-corn (5.5% and 9.7% reduction) under the low, medium, and high-temperature levels, respectively. Table 4-1 shows the average cumulative respiration at various temperature, moisture content and storage duration levels. There is a significant difference between the average values of the three studied temperature levels; i.e.; low, medium and high. As mentioned previously, the cumulative

Table 4-1. The average values of cumulative respiration at various temperature, moisture content and storage duration levels

Parameter*	Number of observations	Mean ($g\text{-}CO_2/kg\text{-}corn$)	Grouping**
Temperature (^oC)			
35	45	1.417	A
45	45	0.953	B
23	45	0.688	C
Moisture Content (% w.b.)			
20.7	27	1.885	D
18.8	27	1.760	D
17.0	27	0.855	E
14.8	27	0.313	F
12.9	27	0.285	F
Time (d)			
9	15	1.381	G
8	15	1.358	G
7	15	1.326	G
6	15	1.268	GH
5	15	1.143	GH
4	15	1.003	HI
3	15	0.820	IJ
2	15	0.595	J
1	15	0.281	K

*Averages with the same letters are not significantly different from each other at $P<0.05$.
**Differences in means are tested within various levels of the parameter, but not among different parameters

respiration reached its maximum value at the medium temperature level. This trend had been observed in various studies on another type of grains (Lacey et al., 1994; Dillahunty et al., 2000). Dillahunty et al. (2000) reported a decline in cumulative respiration above 50^{o}C and under the moisture content of 25%. This phenomenon could be attributed to the fact that living organisms have an optimum temperature level that maximizes their respiration. In the present study, cumulative respiration peaked at the medium temperature level of 35°C.

Correspondingly, the corn cumulative respiration increased with increase in the moisture content levels. In most of the studied cases, and particularly under higher moisture content levels of 18.8, and 20.7%, the cumulative respiration curves increased sharply during the first 3-4 days. Subsequent, the cumulative respiration leveled off until the end of the experiment. The highest cumulative respiration of 2.625 g-CO_2/kg-corn was observed with the moisture content of 18.8% and the medium temperature level of 35^{o}C. Increasing and/or decreasing the moisture content negatively affected the cumulative respiration. Table 4-1 also shows that the highest average cumulative respiration was observed with the moisture content of 20.7% and followed by other lower moisture content treatments. No statistical difference was found between treatments of moisture content levels of 20.7% and 18.8% as well as moisture content levels of 14.8% and 12.9% ($p<0.05$). Huang et al. (2013) and Chidananda et al. (2014) reported that respiration rate increased when temperature and grain moisture content increased. Moog et al. (2010) reported that the temperature had a greater effect than moisture content on fungal susceptibility measurements during the storage of shelled corn samples. Gómez et al. (2014) mentioned that the high temperatures accelerate chemical reactions and respiration rates as well as they expedite the occurrence of the climacteric peak.

Additionally, the storage duration positively affected the cumulative respiration. Statistical analysis showed a significant effect of storage duration on commutation respiration (ANOVA, $p<0.0001$), with some significant differences between means. The cumulative respiration values were significantly different on day 1, 3 and 5 ($p<0.05$). Subsequently, there was no statistical difference in cumulative respiration as shown in Table 4-1. At the low-temperature level of 23°C, corn moisture content of 18.8% and 20.7% showed an increasing trend in the cumulative respiration until day 3 and 6, respectively. The maximum value of the cumulative respiration of 2.625 g-CO_2/kg-corn was observed on the ninth day, the moisture content level of 18.8% and the medium temperature level of 35ºC. All other treatments reported almost negligible respiration activity during the entire experimental period. It is noticeable that various treatments reached their maximum cumulative respiration on different days. Possible explanation of this is the variation in moisture content and storage temperature as stated previously. Additionally, it is clear that the cumulative respiration curves leveled off after they reached their maximum values. It may be attributed to the substantial drop in respiration activities as affected by the reduction in the oxygen concentration in the modules. Herner (1987) stated that oxygen depletion causes inhibition of respiration and prevents further production of carbon dioxide, which might support the results obtained in the present study.

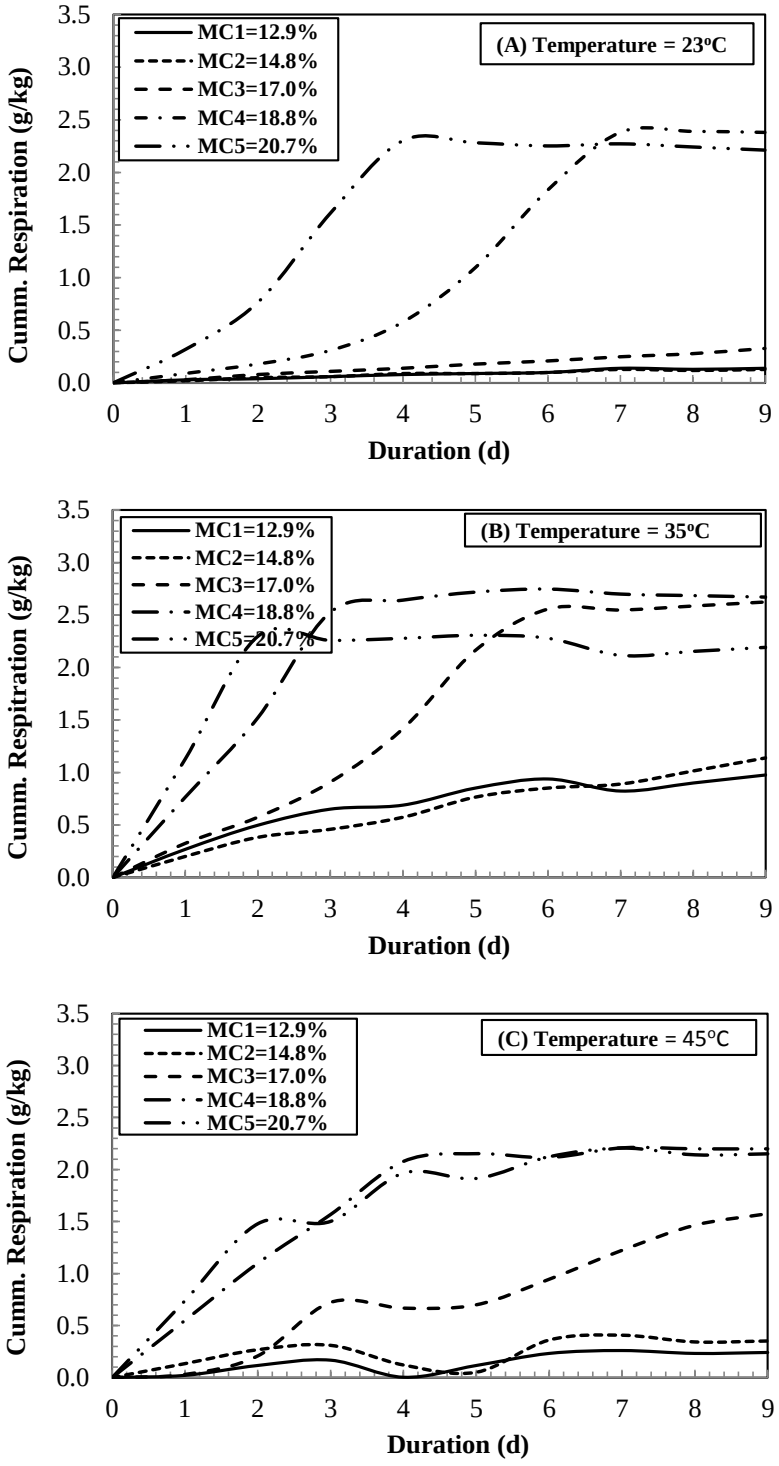

Figure 4-4. Temporal corn cumulative respiration as affected by initial moisture content and storage temperature

4.5. Effects of corn moisture content and storage temperature on respiration rate

Despite the importance of the determination of corn cumulative respiration, it is also crucial to determine the respiration rate and study its changes as affected by the storage duration. Respiration rate is a factor that may help in decision making during the programming of the system aeration. The respiration rate was calculated for each day by subtracting the cumulative respiration of a particular day from the cumulative respiration of the next day. Figure 4-5 illustrates the corn respiration rate as a function of storage temperature, moisture content, and storage duration. Respiration rate increased with the rise in the storage duration and then declined to almost negligible values. Corn moisture content of 20.7% showed the highest respiration rate as compared to the other studied moisture levels. Additionally, the increase of the storage temperature from 23°C to 35°C resulted in an increase in the respiration rate. Further increase in the storage temperature from 35°C to 45°C led to a decline in the respiration rate for all the studied moisture levels. The highest observed respiration rate value of 1.147 g-CO_2/kg-corn.d was observed with the moisture content of 20.7%, at the medium temperature level and on the second day. An imperative trend was observed while storing corn under the medium temperature level of 35°C. Respiration rates reached their maximum values of 0.199, 0.755, 0.987, 1.147 g-CO_2/kg-corn.d under the moisture contents of 14.8% (5^{th} day), 17.0% (5^{th} day), 18.8% (3^{rd} day), and 20.9% (2^{nd} day), respectively. It is clear that under the medium temperature level (35°C), the higher the moisture content, the faster the respiration rate reached its maximum value.

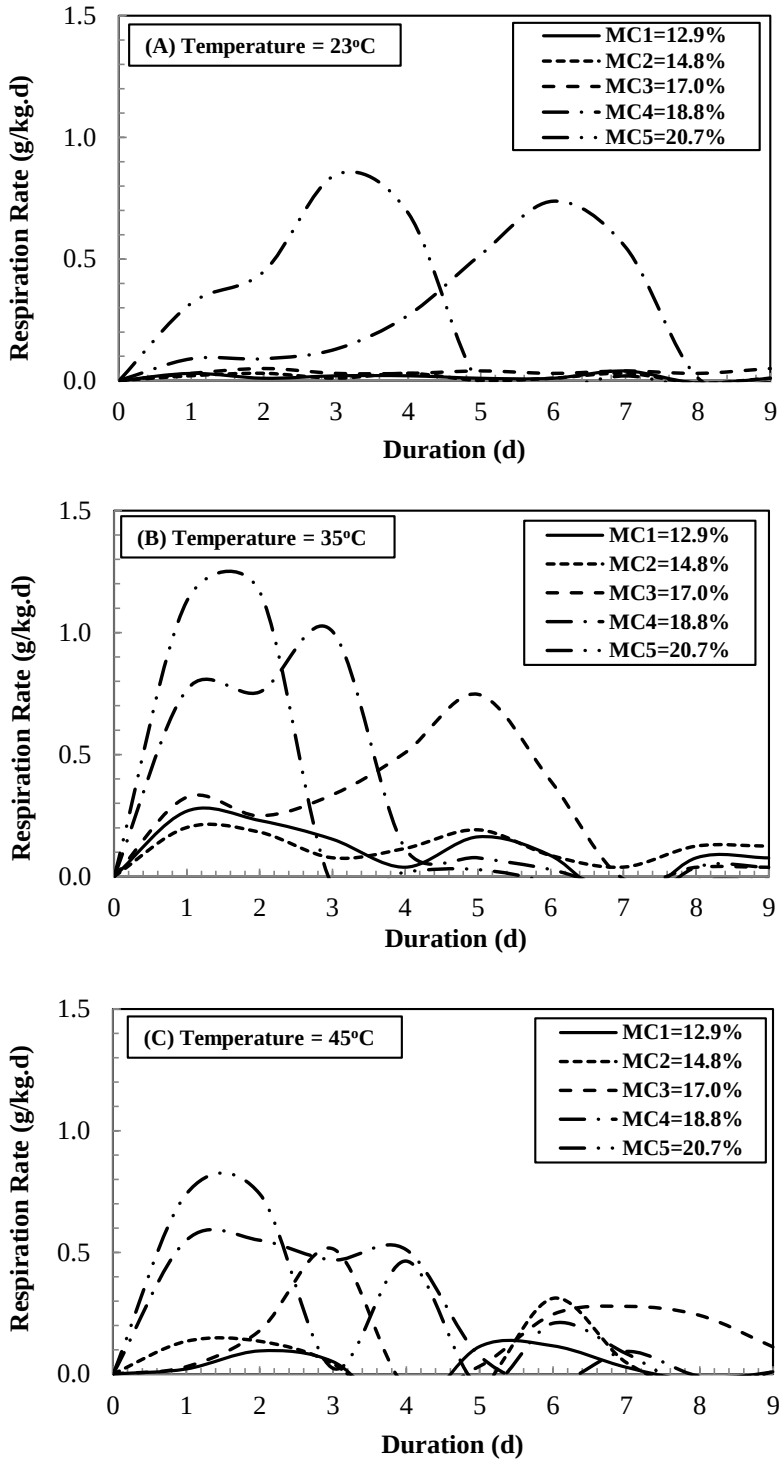

Figure 4-5. Corn respiration rate as affected by initial moisture content and storage temperature

The reason for the difference in timings for peak respiration of individual treatments is due to the difference in moisture levels, as moisture content is the responsible parameter, which expedites the grain respiration activity. Some similar trends were observed at the low and the high-temperature levels. It should be mentioned that under all the temperature levels and lower moisture content treatments, the respiration rate values were significantly lower than that of the higher moisture content levels. Dillahunty et al. (2000) also reported that respiration rate increase up to a certain period and then plateau at a given moisture content and temperature. Pronyk et al. (2004) cited that carbon dioxide production increased with the increase in storage time, moisture content, and temperature. Corn respiration rates observed in the present study, which ranged between 0.000 and 1.147 g-CO_2/kg-corn.d, were in good agreement with the published results. Huang et al. (2013) reported that corn respiration rates were almost negligible at storage temperatures of 10, 20 and 30°C and moisture content of 14.0%. Increasing the storage moisture content to 22.2%, increased corn respiration rates to 0.240, 0.720 and 1.128 g-CO_2/kg-corn.d under the storage temperatures of 10, 20 and 30°C, respectively.

4.6. Effects of corn moisture content and storage temperature on average respiration rate

This study revealed that the medium temperature of 35°C resulted in the maximum values of average respiration rate. Accordingly, it was essential to illustrate the average respiration rate as affected by the moisture content and storage temperature. The average respiration of corn at all temperature levels; i.e.; low, medium and high, increased up to a certain moisture level and then showed a small decline (Figure 4-6). This graph also established the fact that the medium storage temperature of 35°C resulted in the highest average respiration rate as compared with the low and high-temperature levels. The maximum observed average respiration

rate of 0.292 g-CO_2/kg-corn.d was found under the medium temperature level of 35°C and the moisture content level of 18.8%. The average values of respiration rate, under the lower temperature level of 23°C, were lower than the corresponding average respiration rates under the high-temperature level of 45°C. This observation was reversed once the moisture content level reached 18.8% and upward. A possible explanation for this trend is that grains need an individual optimum temperature and moisture levels to grow and disintegrate the matter. Beyond these levels, the organism metabolic activity suppressed resulting in decreased respiration. Jian et al. (2014) reported that non-living and living factors govern the rates of CO_2 production and O_2 consumption. These factors include moisture content, temperature, O_2 concentration, mechanical damage to seeds, microbial contamination, the condition, and period of the previous storage and infestation history by insects and microorganisms. The high rate of respiration of high moisture grain may mostly be caused by the respiration of microorganisms (Magan et al., 2004). The same grain under different storage conditions can be infected by various microorganisms (Pronyk et al., 2004).

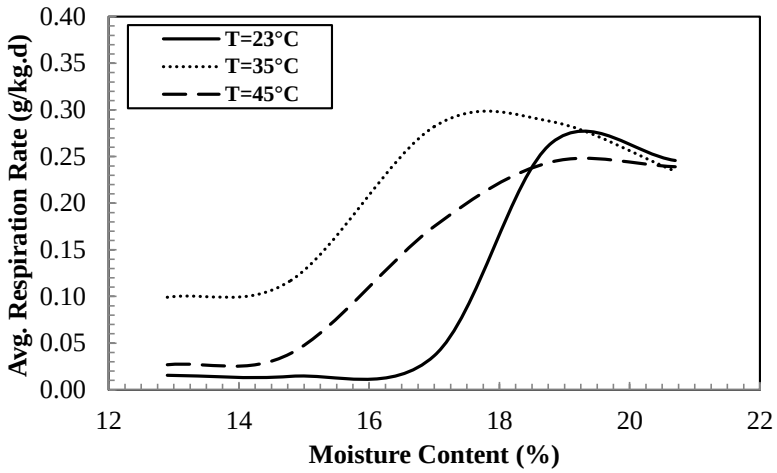

Figure 4-6. Corn average respiration rate as affected by initial moisture content under the low, medium, and high-temperature levels

4.7. Numerical modelling

The multiple regression technique was used to develop an empirical correlation for the determination of the corn cumulative respiration as a function of storage temperature, corn moisture content, and storage duration. The analysis of variance performed using JMP® Pro 11.2.0 on the cumulative respiration values determined that the second-degree polynomial model was the best fit for the data. The multiple linear regression resulted in the following expression:

$$Log_{10}(CC) = -1.8550 + 0.2692\,(T) - 0.4522\,(MC) - 0.0049\,(T)^2 - 0.0199\,(t)^2 + 0.0511\,(MC)\,(t)$$

$$[\text{Adjusted } R^2 = 0.80] \qquad (7)$$

where,

CC is the cumulative carbon dioxide produced in the system [g-CO_2/kg-corn]

T is the average storage temperature [°C]

MC is the initial corn moisture content [%]

t is the storage duration [days]

Waghmare et al. (2013) reported that mathematical modeling is used for prediction of respiration rate as a function of both time and temperature. They found that the temperature and the interaction of time and temperature had significant effects on respiration rate. Bern et al. (2002) developed a set of equations to predict CO_2 evolution of stored corn as a function of moisture content, temperature, and mechanical damage level. Their equations could be used to predict CO_2 evolution from shelled corn. These equations are valid under the moisture content range between 15% and 34%, temperature range between 0 and 49°C, and mechanical damage range between 2 and 41%. In the present study, the developed empirical equation is valid under the moisture content range between 12.9% and 20.7%, temperature range between 23 and 45°C, the storage duration of 9 days for rewetted corn.

CHAPTER 5

CONCLUSIONS

From the experimental work described in this research, several important conclusions can be drawn.

- The pressure sensor technique was found to be reliable and sensitive to measuring corn respiration rate at the tested parameters including storage temperature, moisture content and storage duration.
- Minimum of three replicates should be used to determine corn cumulative respiration.
- Increasing the corn initial moisture content and storage temperature to 18.8% and 35^{o}C, respectively, increased the cumulative respiration to its maximum value of 2.625 g-CO_2/kg-corn.
- The corn respiration rates reached their maximum values earlier under the highest moisture content levels.
- An empirical correlation between the logarithmic value of the corn cumulative respiration as a function of its initial moisture content, the storage temperature, and the storage duration was developed with adjusted coefficient of determination of 0.80.

CHAPTER 6

RECOMMENDED FUTURE WORK

From the results presented in this thesis, it is recommended that future respiration tests needed to be conducted:

- Respiration tests on other commodities including rice, soybean, grain sorghum and wheat.
- Additional testing using longer incubation times and different temperature and moisture conditions need to be performed.
- Corn respiration of freshly harvested corn exposed to natural air drying samples should be evaluated and compared with the re-wetted corn under the same moisture levels.
- Respiration rate of mold infected and non-infected corn samples should be tested and compared to determine the effects of mold infection on respiration rate.
- Respiration rate of infested and non-infested corn samples should be tested and compared to determine the effects of pest infestation on respiration rate.
- Respiration rate of mechanically damaged and whole corn samples should be tested and compared to determine the effects of percentage of broken corn on respiration rate.
- Use the pressure sensor technique to determine respiration rate of rice, a major commodity in Arkansas, and correlate the data with the dry matter loss as well as rice discoloration.

- Respiration rate of new breeding commodities need to be conducted using the pressure sensor technique to understand their respiration activists in just few days prior to drying them.
- Lab-scale respiration results need to be compared to the respiration data from continuous systems and a correlation need to be developed.

CHAPTER 7

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