EVALUATING DRY MATTER LOSS RATES OF 14 TO 22% MOISTURE CONTENT SOYBEANS AT 35°C USING A DYNAMIC GRAIN RESPIRATION MEASUREMENT SYSTEM

BY

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THESIS

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ABSTRACT

Soybeans are biologically active after harvest, continuing to respire during storage and processing. Soybean respiration rate (v_{CO_2}) and, thus, dry matter loss rate (v_{DML}) are affected by moisture content (w) and temperature (T). Knowledge of v_{CO_2} or v_{DML} is useful in developing maximum allowable storage time (MAST) guidelines for soybeans, which are currently sorely lacking for high w and T storage conditions. Therefore, in this study, v_{CO_2} and v_{DML} were measured for soybeans at a wide range of w (14 to 22 %) stored at 35°C. A dynamic grain respiration measurement system (D-GRMS) was used to measure grain deterioration rates of 14 to 22 % moisture content soybeans stored at 35ºC. Results showed that the pooled dry matter loss rates, $\left(v_{DML} \pm SE_{v_{DML}}\right)_p$ were 0.128 ± 0.001, 0.250 ± 0.004, and 0.253 ± 0.005 % d^{-1} for 14, 18, and 22 % moisture content soybeans, respectively. These corresponded to pooled respiration rates, $\left(v_{CO_2} \pm SE_{v_{CO_2}}\right)_p$ of 1.879 ± 0.028, 3.664 ± 0.064, and 3.708 ± 0.068 mg CO_2 $(kg\ d)^{-1}$, respectively. The time to reach 0.5% DML, $t_{0.5} \pm \sigma_{t_{0.5}}$, were also highly variable at 8.32 ± 2.89, 5.88 ± 1.47, and 3.83 ± 0.54 d for 14, 18, and 22 % moisture content soybeans, respectively, due to variable lag times before 0.05 % DML was reached. Using a minimum significant difference to be detected of $\delta = 4(v_{DML})_p$ of 0.0032 % d^{-1} from respiration tests with 18 % moisture content soybeans, a statistical power analysis showed that a minimum of four replications was needed for a $3w$ x $1T$ factorial experiment. The analysis of variance (ANOVA) results, however, showed that across the w tested, the minimum significant difference between treatments was δ = 0.066 % d^{-1} and a minimum of one replication was needed. It is recommended that future soybean respiration experiments proceed with at least four replications. The effects of w on v_{DML} were best described with an exponential question [$v_{DML} = \beta_1 \exp(\beta_2 w)$] with a mean relative error, MRE = 0.15 % d^{-1}; standard error of regression, SE_{reg} =0.02 % d^{-1}; F-statistic = 149.18, and an estimated coefficient of determination, R^2= 0.97. The D-GRMS, protocols, and statistical analyses of grain deterioration parameters presented in this study are useful for conducting robust grain respiration measurements in the future towards building a set of *MAST* guidelines for soybeans and other cereal or oilseed commodities.

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CHAPTER 1. INTRODUCTION

Grains are biologically active after harvest, continuing to respire during storage and postharvest processing. According to Mason et al. (1997), grain deterioration starts even before harvesting and continues after harvest, mainly due to physical, chemical and biological interactions between the grain and its environment. Grain moisture content (w, wet-basis) and storage temperature (T) are critical factors influencing the rate of grain deterioration (Murthy et al., 2003; Liu, 1997). Many researchers have shown that increases in w and T lead to increases in respiration and, hence, grain deterioration rates (Frankel et al., 1987; Lacey et al., 1994; Murthy et al., 2003; Liu et al., 2011).

To minimize deterioration, grains need to be stored under conditions that mitigate further physical, chemical and biological interactions with the environment and guarantee that the crop will not be impaired, for example, by fungal action (Kaleta and Górnicki, 2013). Modern grain trade is increasingly competitive and demands for food safety and quality are growing, which lead to intensified needs for safe grain storage (Ryniecki, 2005; Kaleta and Górnicki, 2013). Thus, grain deterioration is a problem that needs persistent attention to minimize losses that negatively affect grain processing qualities and decrease economic values (Hou and Chang, 2004). Development of adequate storage systems is an important factor in keeping grain for extended periods without losing weight, quality, value and increasing food safety and health risks (Chow, 1980). However, insufficient data on grain deterioration rates increases the challenges of developing guidelines for safe grain storage.

Safe grain storage is defined as the time it takes for the stored product to lose significant processing quality or market value. The American Society of Agricultural and Biological Engineers (ASABE) currently maintains an engineering standard (ASABE Standard D535, R2014) for safe storage of shelled corn based on a 0.5% dry matter loss (DML) threshold, which has been shown to be equivalent to the loss of one unit of market grade, i.e., from U.S. Grade No. 1 to No. 2. The U.S. grade system for grain quality ranges from grades No. 1 to No. 4 and is based on percentages of damaged, broken and split kernels, foreign materials, and discolored soybeans (USDA, 2007). The greater the percentage of these components in a sample, the lower the quality of the grain.

The approach of using DML as the basis for safe storage time guidelines has been proposed for other cereal grains, oilseeds, feed, and fiber (Rotz, 2005; Dadgar et al., 2009), including soybeans, but no other maximum allowable storage time (MAST) guidelines have been accepted as an industry standard. Before additional guidelines are developed, however, it is necessary to evaluate the effects of w and T on respiration and DML rates (v_{CO_2} and v_{DML}, respectively) and their respective variabilities to build a robust and consistent experimental procedure that enables reliable and repeatable results. Moreover, understanding behavior of deterioration rates over a wide range of w and T is key for the development of better and safer postharvest practices. Mathematical models of grain deterioration data from well-designed experiments, with adequate numbers of replications, should form the basis for MAST guidelines.

Therefore, the overall objective of this study was to determine v_{CO_2} and v_{DML} of soybeans, as well as the times to reach 0.5% DML ($t_{0.5}$), and their respective variabilities ($SE_{v_{CO_2}}, SE_{v_{DML}}$, and $\sigma_{t_{0.5}}$) at 14 to 22% moisture contents stored at 35°C. Soybeans are of great economic and social importance worldwide. The chosen experimental conditions represent typical harvest and storage conditions for low-latitude countries. For example, Sinop, Mato Grosso, Brazil (11.8608° S, 55.5095° W) is a region that produces 30% of soybeans in the country, which is equivalent to 9% of global soybean production (USDA, 2014).

The specific objectives of the thesis research described hereunto are to:

1. Determine the pooled respiration rate, $\left(v_{CO_2}\right)_p$, pooled dry matter loss rate, $(v_{DML})_p$, and time to reach 0.5% DML $(t_{0.5})$, and their respective variabilities [standard error of respiration rate, $\left(SE_{v_{CO_2}}\right)_p$, standard error of dry matter loss rate, $\left(SE_{v_{DML}}\right)_p$, and the standard deviation of the time to reach 0.5% DML $(\sigma_{t_{0.5}})$} of 14 to 22% moisture content soybeans stored at 35°C;
2. Determine the minimum number of replications (r_{min}) needed for a robust, statistically designed experiment to build a set of MAST guidelines for soybeans using variability estimates from tests with 18% moisture content soybeans; and
3. Develop an appropriate mathematical model describing the effect of w on v_{DML} at 35°C.

CHAPTER 2. LITERATURE REVIEW

2.1. Grain respiration and dry matter loss

Respiration is a metabolic process that breaks down complex materials, such as sugars, into carbon dioxide (CO_2), water, and energy. The process of respiration is represented by the following chemical equation, where glucose reacts with oxygen and generates carbon dioxide (CO_2), water and heat (Rees, 1982):

$$C_6H_{12}O_6 + 6O_2 \rightarrow 6CO_2 + 6\,H_2O + heat$$

The CO_2 produced during respiration can be used to estimate DML during postharvest processing:

$$DML\ (\%) = \left(\sum m_{CO_2}\right)_s \left(\frac{1\ mol\ C_6H_{12}O_6}{6\ mol\ CO_2}\right)\left(\frac{M_{C_6H_{12}O_6}}{M_{CO_2}}\right) \qquad [2.1]$$

where $\left(\sum m_{CO_2}\right)_s$ is the specific accumulated mass of respired CO_2 (gram per kilogram of dry matter), and $M_{C_6H_{12}O_6}$ and M_{CO_2} are the molar masses of glucose (180 g mol^{-1}) and carbon dioxide (44 g mol^{-1}), respectively.

The amount and rate of CO_2 evolved during the respiration have been used in the development of storage guidelines. Kaleta and Górnicki (2013) reviewed over 30 papers on respiration studies and showed the use of CO_2 and DML measurements and rates, thereof, as the basis for safe storage times of grains. Nearly a century ago, Bailey and Gurjar (1920) used a static grain respiration measurement system (S-GRMS) to estimate the safe storage time of wheat. Steele et al. (1969) were the first to demonstrate that a 0.5% DML corresponded to a depreciation of corn from U.S. Grade No. 1 to No. 2 and, with additional data from other researchers (Friday et al., 1989; Stroshine and Young, 1990; Wilcke et al., 1993; Al-Yahya et al., 1993; Bern et al., 2002), this threshold became the basis for MAST guidelines for shelled corn

(ASABE, R2014). The same approach was taken by Sukabdi (1979), who observed that 18% moisture content long grain rice stored at 29.5°C dropped from a U.S. Grade No. 1 to No. 2 when 0.5% DML was reached, while 22% medium grain rice, at the same T, dropped from U.S. Grade No. 1 to No. 2 when 0.25% DML was reached and even further to U.S. Grade No. 5 when 0.5% DML was reached. Hence, Sukabdi (1979) advocated for a 0.25 to 0.5% DML threshold as a basis for safe storage of rice of any variety. The choice of DML thresholds can be based also on germination losses. For example, for wheat, White et al. (1982) proposed a 0.04% DML threshold while others proposed a 0.085% DML threshold (Brook, 1987; Lacey et al., 1994). The discrepancy in thresholds stemmed from variabilities in estimated times when molding was visibly observed.

2.2. Effects of moisture content and temperature on respiration and dry matter loss rates

A host of factors – T, w, hybrids or varieties, mechanical damage during harvesting, poor handling, inadequate storage facilities – contribute to grain deterioration rates and, thus, safe storage times (Kader, 1988). Elevated values of w and T create a favorable environment for mold growth further aggravating deterioration (Christensen and Kaufmann, 1965). Thompson (1972) studied the effects of T and w on corn DML and respiration, as well as heat transfer into the grain bin and effects of continuous aeration. He concluded that DML doubled when airflow was reduced by half and with each 2% increase in w. Other researchers noted that w has a stronger effect on grain deterioration rates or $t_{0.5}$ than T (Steele et al., 1969; Dillahunty et al., 2000; Bern et al., 2002; Sorour and Uchino, 2004; Jian et al., 2014). For example, when the $t_{0.5}$ table in ASABE Standard D535 (R2014) and Steele et al. (1969) are analyzed, $t_{0.5}$ decreases linearly for every 5.5°C increase in T, but the rate of decrease is exponential for every 2% increase in w. Rice at 11.5 to 15% moisture content and 50°C exhibited v_{CO_2} below 10 mg (kg h)$^{-1}$, which increased five times to 50 mg CO_2 (kg h)$^{-1}$ when w was raised to 20%, and further increased to 80 mg CO_2 (kg h)$^{-1}$ when w reached 25% (Dillahunty et al., 2000). Wheat at 14 to 24% moisture content doubled in v_{CO_2} for every 10°C increase in T, and v_{CO_2} increased by at least 23% for every 1% increase in w (White et al., 1982). Spencer (1976) saw allowable storage time, based on a drop of one US grade quality, for 14% moisture content soybeans decrease from 75 d at 15.5°C to 25 d at 26.6°C; for 22% moisture content, allowable storage times decreased from 5 to 2 d when T increased from 15.5 to 26.6°C, respectively. The effects of w and T on germination loss rates of soybeans were studied by Alencar et al. (2006), where it was observed that total losses increased by 0.6% for every 10°C rise in storage T and nearly doubled for every 2% increase in w.

Grain varieties or hybrids also affect deterioration rates. For example, mold-resistant and mold-susceptible hybrids of corn, when stored at 20.5% moisture content and 26°C, took 14 and 10.5 d, respectively, to reach 0.5% DML (Friday et al., 1989). The v_{DML} of mold-susceptible corn (47.6×10^{-3} % d^{-1}) is 30% greater than the mold-resistant corn (36.4×10^{-3} % d^{-1}). Mechanically damaged soybeans from combine harvesting exhibited v_{DML} (36.4×10^{-3} % d^{-1}) that was 26.8% higher than hand-shelled soybeans (23.1×10^{-3} % d^{-1}), which corresponded to $t_{0.5}$ of 21.5 d instead of 17.0 d (Wilcke et al, 1993).

Mathematical models have been developed to predict $\sum m_{CO_2}, DML, v_{CO_2}, v_{DML}$ and $t_{0.5}$ (Table 2.1) dating back to 1972, when Thompson created a model for predicting CO_2 production during storage of corn (Model No. 1) based on the data obtained by Steele et al. (1969). This model has been extensively used by other researchers, even under different grains and storage conditions, for example, to predict CO_2 production of corn from 15 to 35% moisture content and 0 to 49°C by Bern et al. (2002) and to study the effects of grain damages on grain

Table 2.1. Summary of mathematical models available for predicting grain respiration rates, dry matter loss, and safe storage times. Variables are defined in the footnotes.

	Model No.	Mathematical model[1]	Grain	GRMS (S/D)[2]	Storage conditions: T (°C) and w (%)	References
Exponential or logarithmic functions	1	$\sum m_{CO_2} = \beta_1[exp(\beta_2 t - 1)] + \beta_3 t$ for every w and T treatment combination	corn	D	$10 \leq T \leq 50$ $20 \leq w \leq 25$	Thompson (1972) Stroshine and Yang (1990)
			soybeans	D	$15 \leq T \leq 30$ $18 \leq w \leq 26$	Sorour and Uchino (2004)
	2	$DML = 1 - \exp\{\beta_1 \exp[(T - 15.6) + \beta_3(0.14w)]\}$	rice	S	$10 \leq T \leq 40$ $12 \leq w \leq 21$	Atungulu et al. (2017)
	3	$v_{CO_2} = \beta_1[\exp(\beta_2 w)]$	rice	S	$T = 30$ $11 \leq w \leq 25$	Dillahunty et al. (2000)
	4	$t_{0.5} = \exp(\beta_1 + \beta_2 T + \beta_3 w + \beta_4 DML)$	wheat	D	$4 \leq T \leq 40$ $15 \leq w \leq 24$	Al-Yahya (2001)
	5	$t_m = \exp[\beta_1 - \beta_2 T - 2.71\phi]$	pea	S	$10 \leq T \leq 40$ $w = 18$	Dadgar e al. (2009)
	6	$\log\left(\sum m_{CO_2}\right) = -\beta_1 + \beta_2 T - \beta_3 w - \beta_4 T^2 - \beta_5 t^2 + \beta_6 wt$	corn	S	$23 \leq T \leq 45$ $13 \leq w \leq 21$	Ubhi and Sadaka (2015)
	7	$\log(DML) = \beta_1 + \beta_2 w + \beta_3 T + \beta_4 w^2 + \beta_5 wT$	wheat	S	$15 \leq T \leq 30$ $15 \leq w \leq 19$	Mylona et al. (2012)
	8	$\log\left(v_{CO_2}\right) = \beta_1 + \beta_2 T + \beta_3 t + \beta_4 t^2 + \beta_5 w$	rice	S	$10 \leq T \leq 40$ $14 \leq w \leq 24$	White et al. (1982)
	9	$\ln v_{CO_2} = \beta_1 + \beta_2 t + \beta_3 t^2 + \beta_4 w$	wheat	S	$15 \leq T \leq 35$ $15 \leq w \leq 19$	Karunakaran et al. (2001)

[1] Model variables: Dependent variables: accumulated mass of respired CO_2, $\sum m_{CO_2}$; accumulated dry matter loss, DML; respiration rate, v_{CO_2}; time to reach 0.5 or 0.5 or 1.0% DML, $t_{0.5}$ or $t_{1.0}$, respectively; time to reach visible molding, t_m. Independent variables: storage time, t; storage temperature, T; grain moisture content, w; relative humidity, ϕ; concentration of split kernels, C_{sk}. Regression coefficients: β_1 through β_6.

[2] Static (S) or dynamic (D) grain respiration measurement system (GRMS).

Table 2.1. Continued

	Model No.	Mathematical model[1]	Grain	GRMS (S/D)[2]	Storage conditions: T (°C) and w (%)	References
Quadratic or cubic functions	10	$DML = \beta_1 + \beta_2 T + \beta_3 w + \beta_4 T^2 + \beta_5 wT + \beta_6 w^2$	corn	S	$5 \leq T \leq 20$ $14 \leq w \leq 35$	Kaleta and Górnicki (2013)
	11	$t_{1.0} = \beta_1 + \beta_2 T + \beta_3 w + \beta_4 T^2 + \beta_5 wT + \beta_6 w^2$	corn	D	$1 \leq T \leq 24$ $15 \leq w \leq 30$	Brooker et al. (1974)
	12	$v_{CO_2} = \beta_1 t + \beta_2 t^2 + \beta_3 t^3$	soybean	D	$T = 26$ $19 \leq w \leq 21$	Rukunudin (1997)
	13	$t_{0.5} = \beta_1 C_{sk}^3 + \beta_2 C_{sk}^2 - \beta_3 C_{sk} + \beta_4$	corn	D	$T = 26$ $9 \leq w \leq 21$	Bern et al. (1999)

[1] Model variables: Dependent variables: accumulated mass of respired CO$_2$, $\sum m_{CO_2}$; accumulated dry matter loss, DML; respiration rate, v_{CO_2}; time to reach 0.5 or 0.5 or 1.0% DML, $t_{0.5}$ or $t_{1.0}$, respectively; time to reach visible molding, t_m. Independent variables: storage time, t; storage temperature, T; grain moisture content, w; relative humidity, ϕ; concentration of split kernels, C_{sk}. Regression coefficients: β_1 through β_6.

[2] Static (S) or dynamic (D) grain respiration measurement system (GRMS).

respiration by Stroshine and Yang (1990). The model developed by Thompson (1972) was applied to soybeans by Sorour and Uchino (2004) at discrete w and T treatment combinations. By doing so, the exponential models they built were for specific storage conditions in their studies and cannot be generalized or used for other conditions or grains.

Exponential models of DML (Model 2) and v_{CO_2} (Model 3) across a wide range of w and T were developed by Atungulu et al. (2017) and Dillahunty et al. (2000), respectively, for rice while others used the same approach to predict specific values of DML Models 4 and 5 (Al-Yahya, 1991; Dadgar et al., 2009). Other researchers, however, used a logarithmic (Models 6 to 9) (Ubhi and Sadaka, 2015; Mylona et al., 2012; White et al., 2001; and Karunakaran et al., 2001), quadratic (Models 10 and 11) (Kaleta and Górnicki, 2013; Brooker et al., 1974), or cubic functions (Models 12 and 13) (Rukunudin, 1997; Bern et al., 1999).

Why are there many model forms to describe the same grain deterioration behavior? It all depends on how narrow the w or T range used in a particular study. Consider the general shape of a linear, quadratic, cubic, or exponential function (Figure 2.1). Studies that are based on a narrow set of w and/or T may observe a linear DML behavior across these two parameters over time. Over a wide range of treatments, the nonlinear DML behavior becomes more apparent, especially when a lag period or visible molding is observed at the beginning or end of a respiration test, respectively. Regardless of whether a static or dynamic grain respiration measurement system (S- or D-GRMS) is used, there is no trend on which model function tends to be used to describe grain deterioration despite lower DML values reported for S-GRMS (Pereira da Silva et al., 2017).

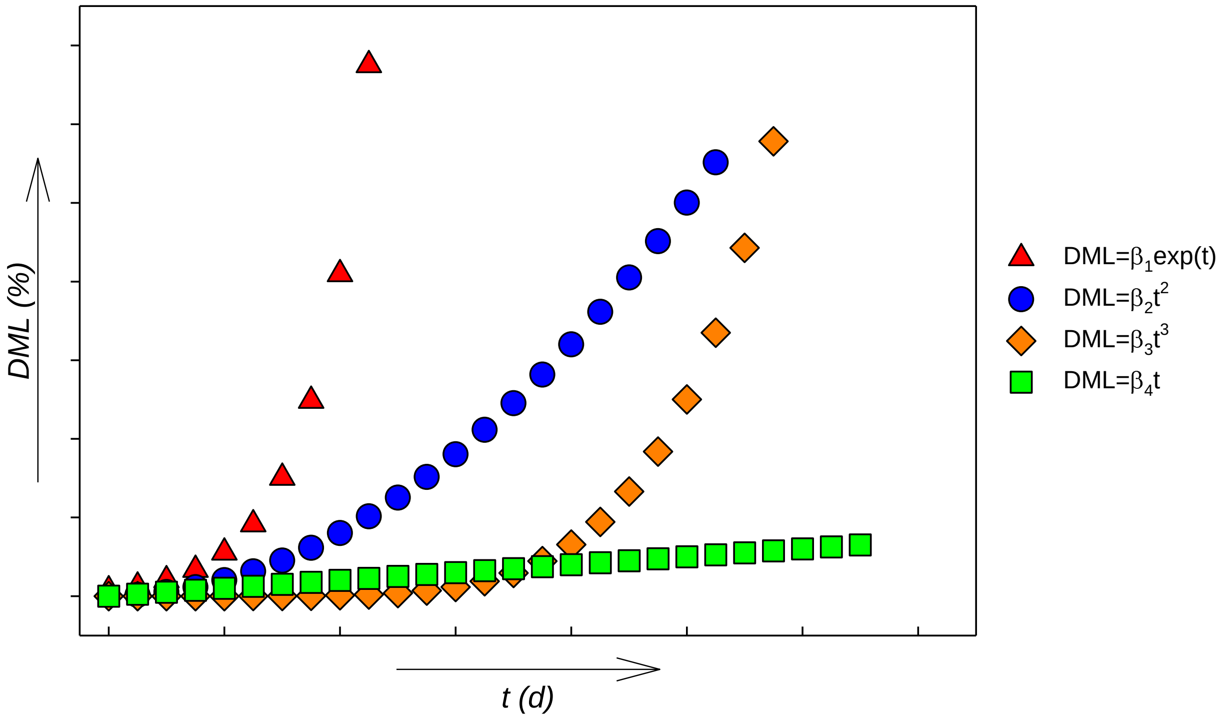


Figure 2.1. General shape of linear, quadratic, cubic and exponential functions used to describe grain deterioration.

If all the models listed in Table 2.1 are describing the same biological behavior, there should be some agreement amongst them. However, that is not the case. A comparison among models demonstrated that for equations designed to predict the same dependent variable ($v_{CO_2}, DML, t_{0.5}$ or $\sum m_{CO_2}$) values do not converge, even for very similar ranges of w and T. For example, the predictive model developed by White et al. (1982) and Dillahunty et al. (2000), using data from static systems for v_{CO_2} of rice, provides respiration rates of 6.19 mg CO_2 (kg d)$^{-1}$ and 0.51 mg CO_2 (kg d)$^{-1}$, respectively at 20% moisture content and 30°C. The discrepancy in these predictions may be explained by factors such as ranges of w and T tested, modeling (exponential, quadratic, or cubic), data collection system (static or dynamic) and CO_2 monitoring system (by absorption or use of a transducer or bench-scale instrument).

2.3. Systems used to measure grain respiration

Historically, two different types of measurement systems – static and dynamic – have been used to measure grain respiration. In S-GRMS, grain is placed in a sealed container, where the CO_2 concentration is monitored over time using a gas chromatograph or a sensor (White et al., 1982; Dadgar et al., 2009; Jian et al., 2014). This system is useful for small grain amounts and short storage periods. A D-GRMS, on the other hand, involves passing air through a bed of grain and determining the difference in CO_2 levels at the inlet and outlet over time (Ubhi and Sadaka, 2015; Gross et al., 2016). CO_2 levels can be determined in a variety of ways: gravimetrically or using a gas analyzer, such as a non-dispersive infrared (NDIR) sensor. Gravimetric methods involve passing the respired air through a CO_2 absorbent material and monitoring the incremental increase in mass of the absorbent over time.

Earlier respiration studies used ascarite extensively as a CO_2 absorbent (Steele et al., 1969; Fernandez et al., 1985). Ascarite is a combination of asbestos particles and sodium hydroxide, which can absorb CO_2 as much as 40% of its own weight (Al-Yahya, 1991). Al-Yahya et al. (1991) described the preparation of a vermiculite-potassium mixture to capture CO_2, as a replacement for ascarite. This mixture was prepared using vermiculite granules soaked 50% (w/v) in potassium hydroxide solution.

A medical grade soda lime material called Sodasorb® (Amron International, Vista, CA, USA) can be used as CO_2 absorbent in D-GRMS. Sodasorb® works following the same principles as ascarite and the vermiculite-potassium mixture, specifically by the following chemical reactions (Nuckols et al., 1985):

$$CO_2 + H_2O \leftrightarrow H_2CO_3$$

$$2H_2CO_3 + 2NaOH + 2KOH \rightarrow Na_2CO_3 + 4H_2O + K_2CO_3$$

$$2Ca(OH)_2 + Na_2CO_3 + K_2CO_3 \rightarrow 2CaCO_3 + 2NaOH + 2KOH$$

Water is needed to initiate the first chemical reaction, where CO_2 is captured in the Sodasorb® as carbonic acid (H_2CO_3) which reacts with embedded hydroxides to form more stable carbonates (Na_2CO_3 and K_2CO_3). In this process, water is produced as a byproduct (Nuckols et al., 1985). The carbonates react with calcium hydroxide, also embedded in the Sodasorb®, to form a more stable calcium carbonate ($CaCO_3$). Often, ethyl violet dye is added during manufacture of Sodasorb®. This indicator dye changes from white to purple as CO_2 is absorbed, but its efficacy diminishes as pH falls from 10.3, moisture content is lost, or the material is exposed to intense ultraviolet light. Over time, the indicator dye reverts from purple to white as a result of subsurface calcium hydroxide regenerating active hydroxide at the surface of a granule, causing a pH change at the surface. At this point, the Sodasorb® granules appear white even though the soda lime is nearly exhausted, and its CO_2 absorptive capacity is minimal. Nuckols et al. (1985) demonstrated that absorbent moisture content, flow rate and relative humidity of the incoming air are crucial factors for proper functioning of Sodasorb®. Too little moisture in the absorbent may prevent the absorption reactions to start; also, too little moisture in the incoming air may cause the absorbent to dry and compromise the absorbent efficiency. Moreover, gas carrying CO_2 needs to reside in the scrubber long enough to allow absorption process to occur.

2.4. Challenges in developing MAST guidelines

With several researchers having reported v_{CO_2} or $t_{0.5}$ for several grains and oilseeds stored under a variety of conditions (Table 2.2), it may be plausible to compile information from

Table 2.2. Previous studies on grain respiration, CO_2 measurement method, deterioration rates and number of replications used. Variables are defined in the footnotes.

Grain	CO_2 measurement[1]		Storage conditions[3]		Grain deterioration rates			Replications r	Reference
			w (%, w.b.)	T (°C)	v_{CO_2}[4] [mg (kg d)$^{-1}$]	$t_{0.5}$[4] (d)	v_{DML}[5] (10^{-3} % d^{-1})		
canola	S	GC	8 – 14	10 – 40	10.2 – 327.8		1.00 – 22.0	3	Jian et al. (2014)
	S	GC	10 – 14	25 – 30	172 – 500		10 – 30	1	Pronyk et al. (2004)
corn	D	Abs	17.5 – 30	10 – 48		1.5 – 100	5.0 – 333	2	Steele et al. (1969)
	D	Abs	22	15 – 25		10.9 – 14.1*	31.0 – 45.0	2	Al-Yahya (1991)
	D	GC	18 – 22	20		9.8 – 42.5*	12.0 – 51.0	3	Gupta et al. (1999)
	S	P	13 – 21	23 – 45	0.0 – 1147		0.00 – 0.08	3	Ubhi and Sadaka (2015)
	D	GC	21.5 – 22.5	20		0.95 – 1.7	0.53 – 0.29	3	Wilcke et al. (1993)
	D	Abs	22	26	10 – 120		1-8	1	Fernandez et al. (1985)
	S	Abs	18 – 22	20	3.75 – 13.75		0.3 – 0.9	5	Chitrakar et al. (2006)
	D	ABS	15 – 18	25	0 – 18		0 – 1	3	Reed et al. (2006)
pea	S	GC	18	10 – 40		14.0 –126.0	4.00 – 35.0	2	Dadgar et al. (2009)

[1] Systems were either static (S) or dynamic (D) and used a variety of methods to monitor CO_2: gas chromatography (GC), absorbent (Abs), pressure sensor (P), and infrared (IR) sensor.

[2] The system was operated as a semi-static system. Researchers replenished oxygen (O_2) during respiration tests by periodically opening grain-filled flasks to allow air exchange with the environment.

[3] Grain wet-basis moisture content (w) and temperature (T).

[4] Values indicated with a "*" were converted from hourly to daily rates.

[5] Values estimated based on respiration rate [$v_{DML} = v_{CO_2}(1\ mol\ C_6H_{12}O_6/6\ mol\ CO_2)\left(M_{C_6H_{12}O_6}/M_{CO_2}\right)$] or time to reach 0.5% DML ($v_{DML} = 0.5/t_{0.5}$).

Table 2.2. Continued

Grain	CO$_2$ measurement[1]		Storage conditions[3]		Grain deterioration rates			Replications r	Reference
			w (%, w.b.)	T (°C)	v_{CO_2}[4] [mg (kg d)$^{-1}$]	$t_{0.5}$[4] (d)	v_{DML}[5] (10^{-3} % d^{-1})		
rice	S	GC	11 – 25	30	192 – 2040*		13.0 – 140.0	3	Dillahunty et al. (2000)
soybeans	D	Abs	16 – 24	22 – 89	12.0 – 1450		0.10 – 100.0	1	Ramstad and Geddes (1942)
	D	Abs	9 – 21	26		10.0 – 26.0	20.0 – 50.0	3	Rukunudin et al. (2004)
	D	Abs	14 – 26	15 – 30		7.1 – 47.2*	10.0 – 70.0	3	Sorour and Uchino (2004)
	S	GC	23	15 – 35	46.6 – 269.2		3.0 – 20.0	3	Jian et al. (2014)
	S	IR	12.5	40 – 80		45 – 180	2.7 – 10	5	Hartmann Filho et al. (2016)
	D	Abs	12	25	600 – 2400		4 – 160	5	Mendes et al. (2009)
wheat	S	Abs	12 – 16	37	2.2 – 247.2		0.10 – 16.0	1	Bailey and Gurjar (1920)
	S[2]	GC	14 – 24	10 – 40		1 – 77	10.0 – 500	4	White et al. (1982)
	S	GC	15 – 19	15 – 35	10.0 – 829.0		10.0 – 56.0	2	Karunakaran et al. (2001)
	S	GC	14	15 – 35	36.7 – 214.2		3.0 – 15.0	3	Jian et al. (2014)
	S	GC	20.5 – 32	15 – 30	10 – 600		1 – 4	3	Mylona et al. (2012)

[1] Systems were either static (S) or dynamic (D) and used a variety of methods to monitor CO$_2$: gas chromatography (GC), absorbent (Abs), pressure sensor (P), and infrared (IR) sensor.

[2] The system was operated as a semi-static system. Researchers replenished oxygen (O$_2$) during respiration tests by periodically opening grain-filled flasks to allow air exchange with the environment.

[3] Grain wet-basis moisture content (w) and temperature (T).

[4] Values indicated with a "*" were converted from hourly to daily rates.

[5] Values estimated based on respiration rate [$v_{DML} = v_{CO_2}(1\ mol\ C_6H_{12}O_6/6\ mol\ CO_2)\left(M_{C_6H_{12}O_6}/M_{CO_2}\right)$] or time to reach 0.5% DML ($v_{DML} = 0.5/t_{0.5}$).

the literature to develop MAST guidelines for soybeans and other major cereal crops. However, doing so is not an easy task. First, for some commodities like canola, peas, rice and wheat, respiration studies have been conducted using S-GRMS only. S-GRMS typically yield lower estimates of v_{CO_2} than D-GRMS (Pereira da Silva et al., 2017) as a result of limited oxygen levels in the system, so measurements describe hermetic storage conditions that are atypical of aerated bulk storage systems that are more commonly used in practice. For corn and soybeans, both S- and D-GRMS have been used in grain respiration studies, in combination with a wide array of CO_2 measurement techniques with varying sensitivities and limits of detection to CO_2, which make direct comparisons of results from one study to another difficult.

Because of the duration required for grain respiration studies, most researchers elect to minimize the number of respiration tests by focusing on a narrow range of w or T. In most studies using D-GRMS, one factor (w or T) was kept constant while varying the other. In some cases, few replications are conducted. For example, Steele et al. (1969) varied w from 23 to 28% and kept the T constant at 18°C. Al-Yahya (1991) tested 22% moisture content corn while varying T from 15 to 25°C. Compared to respiration studies on corn, soybean studies had higher ranges of w and T tested, but were limited in replications. Ramstad and Geddes (1942) tested soybeans from 16 to 24% moisture content stored at 22 to 89°C, but conducted only one replication per treatment combination. Others kept replication numbers to two or three. Mendes et al. (2009) was able to conduct five replications for a soybean respiration tests at a single w (12%) and T (25°C).

The variability in test conditions and methodologies led to a wide array of reported grain deterioration rates. A survey of grain respiration studies since the 1920s shows most researchers reported grain v_{CO_2}or $t_{0.5}$ (Table 2.2). For direct comparisons to be made, reported v_{CO_2} or $t_{0.5}$ values were converted to v_{DML} as follows:

$$v_{DML} = v_{CO_2}\left(\frac{1\,mol\ C_6H_{12}O_6}{6\,mol\ CO_2}\right)\left(\frac{M_{C_6H_{12}O_6}}{M_{CO_2}}\right) \qquad [2.2]$$

or

$$v_{DML} = \frac{0.5}{t_{0.5}}. \qquad [2.3]$$

Jian et al. (2014) found that 14% moisture content canola at 40°C exhibited v_{DML} of 22 x 10^{-3} % d^{-1}, while Pronyk et al. (2004) observed v_{DML} of 30 x 10^{-3} % d^{-1} for 14% moisture content canola at a lower temperature, 35°C. Corn at 22% moisture content and stored at 20°C exhibited v_{DML} of 51 x 10^{-3} % d^{-1} (Gupta et al., 1999), which was 100 times higher than the 0.53 x 10^{-3} % d^{-1} that Wilcke et al. (1993) reported. Furthermore, corn at 22% moisture content level and 26°C showed an even lower v_{DML} (0.001 x 10^{-3}% d^{-1}) (Fernandez et al., 1985), albeit this measurement was based on a single replication. Studies on wheat showed discrepancies in v_{DML} estimates despite having similar storage conditions. White et al. (1982) reported wheat at 22% moisture content stored at 40°C having a v_{DML} of 500 x 10^{-3} % d^{-1}, while Karunakaran et al. (2001) observed 19% moisture content wheat at 35°C having a v_{DML} of 56 x 10^{-3} % d^{-1}.

There was a general agreement from Ramstad and Geddes (1942), Rukunudin et al. (2004), and Sorour and Uchino (2004) that soybeans stored in D-GRMS had increasing v_{DML} with increasing w and T. Mendes et al. (2009) found the same behavior but reported much higher values for v_{DML} despite storing the soybeans at lower T conditions. Divergence of v_{DML} estimates could stem from a number of reasons, including the inadequacy of replications and differences in accuracy and precision of methods to measure respired CO_2.

2.5. Number of replications in grain respiration studies

From Table 2.2., grain respiration studies were based often on experiments with one to five replications. To ensure an experiment is reliable, it is necessary to plan trials and correctly evaluate their precision, including understanding sources of variability between replications of treatments (Storck, et al., 2011). To increase the ability of an experiment to assist in detecting differences, the minimum number of replications (r_{min}) should based on standard deviation (σ), degrees of freedom (df), significance (α) and power (β) of test, and the difference desired to be detected between two replications (δ) (Cochran and Cox, 1957):

$$r_{min} = 2\left(\frac{\sigma}{\delta}\right)^2 \ (t_1 + t_2)^2 \tag{2.4}$$

Values of t_1 and t_2 are determined from the t-student distribution, using a significance level (α) and power (β). The value of α was set as 0.05 interval of significance, which indicates the probability of Type I error, i.e. the chance of falsely rejecting a null hypothesis when it is true. The value for β is the power of the experiment design and expresses the probability that the difference δ will be falsely detected as treatment effect (Type II error); $\beta = 0.8$ is considered a powerful experiment (Cochran and Cox, 1957). For a set of replications under similar conditions, an estimate of the pooled standard deviation (σ_p) can be applied to estimate the variability among a group of tests or variation within each replication (Zimmerman, 2004). When comparing treatment means, both σ and δ may be expressed as a function or percentage of the mean value μ. Thus, coefficient of variation ($CV = \sigma/\mu$) and $\delta = c\mu$ may be used in Equation 2.4, where $0 < c \leq 1$:

$$r_{min} = 2\left(\frac{CV}{\delta}\right)^2 (t_1 + t_2)^2 = 2\left(\frac{\sigma}{c\mu^2}\right)^2 (t_1 + t_2)^2. \tag{2.5}$$

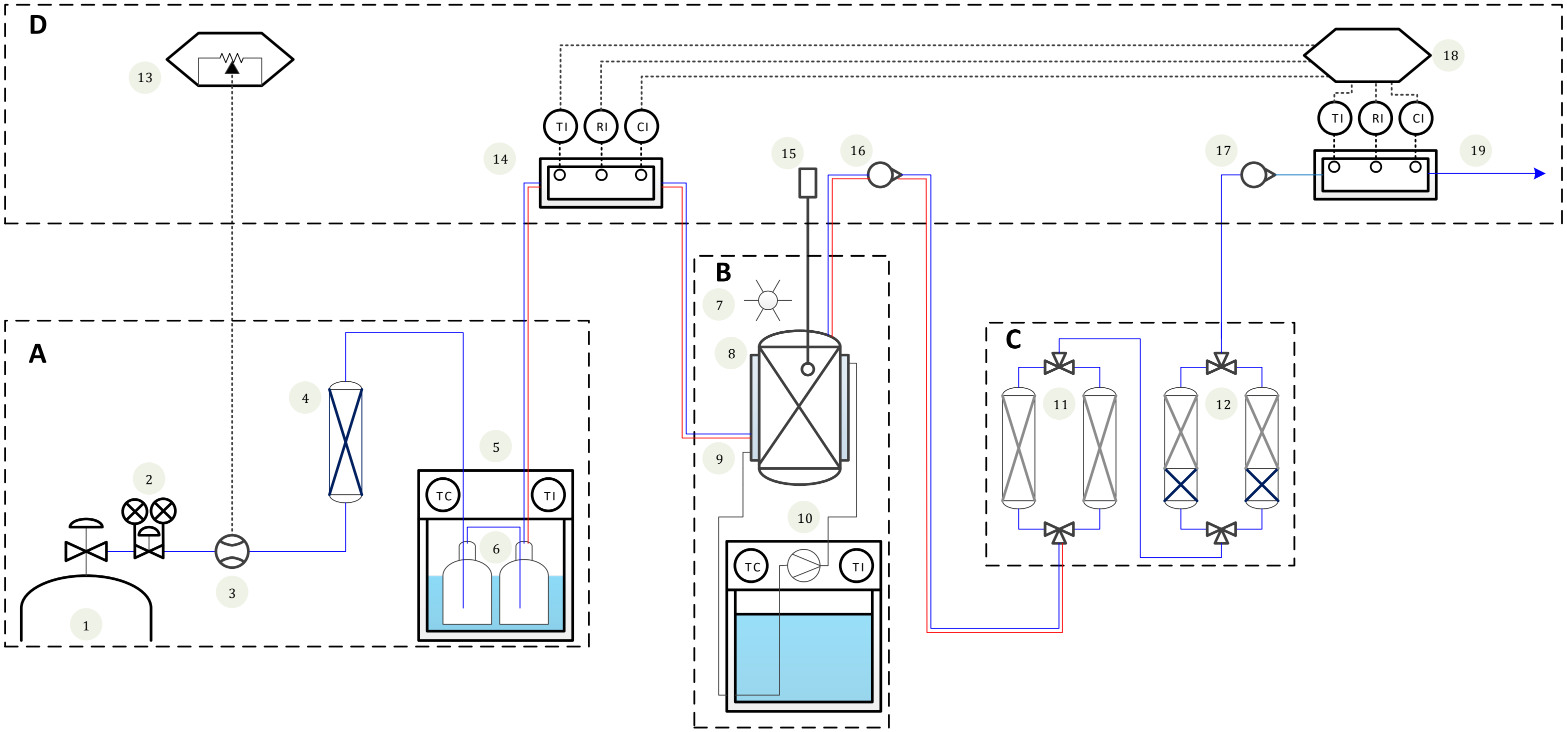
CHAPTER 3. MATERIALS AND METHODS

3.1. Dynamic grain respiration measurement system (D-GRMS)

A D-GRMS was developed and used in this study (Figure 3.1), based on the design and test protocols of Sood (2015). The system is divided into four parts: (a) air conditioning and flow management, (b) grain storage, (c) moisture and CO_2 absorption, and (d) instrumentation. Unless otherwise noted, system components were connected using Vincon Flexible PVC tubing (Part No. ABH02017, Saint-Gobain, Akron, OH, USA) using quick disconnect connectors and three-way valves (Catalog No. 60719, 60721 and 22259, U.S. Plastics, Lima, OH, USA), which were insulated with a 6.35 mm-thick pre-slit polyethylene pipe insulation on critical parts. All moisture content values are reported on a wet basis, unless specified otherwise.

3.1.1. Section A – Air conditioning and flow management.

Air was supplied using a compressed air tank (80% N_2, 20% O_2, with a CO_2 concentration (C_{CO_2}) of 400 ppm) whose flow rate (Q) was regulated at 500 ml min^{-1} using a precision mass flow controller (Model No. GFC17A, Aalborg, Orangeburg, NY, USA, accuracy ± 1.0% of full scale reading). The air was scrubbed of CO_2 by passing it through a 400 g bed of CO_2 absorbent material (Sodasorb®, Amron International Vista, CA, USA). Additional information regarding standard operating procedure (SOP) to setting up the air supply CO_2 scrubber is described in Document No. D-GRMS-001 in Appendix A. Temperature (T) and relative humidity (ϕ) of the air were controlled by passing the airstream through two bubblers (Part No. 50033, Red Sea, Houston, TX, USA) each placed in a glycerol-water solution contained in two 2 L plastic vacuum bottles (Catalog No. D1069702, US Plastics, Lima, OH, USA) to humidify the air stream to a relative humidity in equilibrium (ϕ_e) with the soybean



Section A – Air Conditioning & Flow Management

1. Compressed air tank
2. Pressure regulator
3. Mass flow controller (MFC)
4. Supply air CO_2 scrubber
5. Water bath A
6. Glycerol solution

Section B – Grain Storage

7. Light bulb
8. Respiration chamber (RC)
9. Heat tape
10. Water bath B

Section C – Absorption Columns

11. RC dehumidifier
12. RC CO_2 scrubber

Section D – Instrumentation

13. Microcontroller 1 - MFC
14. T, RH, and CO_2 sensors
15. Thermometer
16. Rotameter A
17. Rotameter B
18. T, RH, and CO_2 sensors
19. Microcontroller 2 – data acquisition

Abbreviation

TC, Temperature controller
TI, Temperature indicator
RI, Relative humidity indicator
CI, CO_2 concentration indicator

Blue line = flow path
Black line= instrumentation wiring
Red line = heat tape

Figure 3.1. Schematic of dynamic grain respiration measurement system (D-GRMS). Heat was applied to the flow tubes in Section B using a 100W light bulb and a 19.6 W m^{-1} heating tape to prevent condensation of the humidified or respired air streams.

moisture content ($w_{soy,1}$). The bottles were connected in series and immersed in a 35°C water bath (Model No. RTE7 NESLAB, Thermo Electron Corporation, Newington, NH, USA). For example, for a test with 18% moisture content soybeans at 35°C, a 36.3% (m/m) solution was prepared by diluting and mixing analytical grade glycerol (Product No. G33500, Fisher Scientific, Hampton, NH, USA) in deionized water for 30 min at 50°C using a hot plate (Model 11-100-49SH, Fisher Scientific, Dubuque, IA, USA). This corresponded to an ϕ_e of 88%. For tests with 14% and 22% moisture content soybeans at 35°C, a 51.47% and 29.52% (m/m) solution was used, respectively, corresponding to ϕ_e values of 80 and 92%, respectively. Additional information regarding preparation of glycerol-water solutions can be found in Appendix A (Document No. D-GRMS-002) and Sood (2015).

3.1.2. Section B – Grain storage

The conditioned airstream passed though the grain respiration chamber (RC) made of a sealed acrylic cylinder (10.2 cm ID x 40.6 cm height), which could hold 1800 g of soybeans. RC temperature, or grain storage T, was maintained using an external water jacket made of Vincon Flexible PVC tubing (Part No. ABH02017, Saint-Gobain, Akron, OH, USA) wrapped around the RC. Water at 35°C was recirculated through the jacket using a second water bath (Model 9102A11B, PolyScience, Niles, IL, USA). Grain storage T was visually monitored using a digital thermometer (Model No. 11050, DeltaTRAK, Pleasanton, CA, USA) located at the top of the column and inserted 7.5 cm deep into the grain bed. To minimize T fluctuations and condensation of incoming and exiting air streams, the RC was thermally insulated with a 6.35 mm-thick pre-slit polyethylene pipe insulation, wrapped with a heat tape (Model No. W51-6p, Raychem, Houston, TX, USA), and placed under a 100 W light bulb.

3.1.3. Section C – Moisture and CO_2 absorption

Air exiting the RC was passed through an RC dehumidifier made of 550 g bed of desiccant (Catalog No. 21001, WA Hammond Drierite Co., Ltd., Xenia, OH, USA) contained in a cylinder (Model No. 26800, W. A. Hammond Drierite Co., Xenia, OH, USA) to remove excess moisture from humidification and grain respiration. The 4-mesh desiccant contained a color indicator (i.e., blue when dry, pink when wet) that allowed for visual monitoring of the moisture removal process. The SOP on setting up RC CO_2 dehumidifiers is available as Document No. D-GRMS-003 in Appendix A.

After dehumidification, the air was passed through a RC CO_2 scrubber made of a layer of 150 g of CO_2 absorbent (Sodasorb®, Amron International, Vista, CA, USA) followed by a 300 g of 4-mesh desiccant (Catalog No. 21001, WA Hammond Drierite Co., Ltd., Xenia, OH, USA). The two layers of materials were contained in a cylinder (Model No. 26800, W. A. Hammond Drierite Co., Xenia, OH, USA), separated by a small plastic cylinder (2.5 cm ID x 1.5 cm height) with perforated disks at each end (40% open, 0.3 cm dia. holes) to mitigate diffusion of moisture from the CO_2 absorbent into the desiccant. The SOP on setting up RC CO_2 scrubbers is available as Document No. D-GRMS-004 in Appendix A, as well as results from testing their efficacy.

3.1.4. Section D – Instrumentation

A series of T and ϕ sensors (Model No. DHT11, WAVGAT, Caizhixing, China) and CO_2 nondispersive infrared (NDIR) sensor probes and transmitters (Model Nos. GMP222 and GMPG0N0, Vaisala, Boulder, CO, USA) were placed at (a) in between Sections A and B and (b) the exhaust of D-GRMS to verify the air stream were at the following conditions: $T = 35 \pm 2$ °C, $C_{CO_2} \leq 20$ ppm and $\phi = 79$, 89, or 92 ± 5 %RH for w = 14, 18, or 22% moisture content, respectively. All sensor readings were logged every 2 min onto a desktop computer using a microcontroller (ATmega2560, Arduino, Ivrea, Italy). Additional information regarding the circuitry and Arduino codes used to log T, ϕ, C_{CO_2} are included in Appendix A. The mass flow controller voltage input was finely adjusted using a digital potentiometer connected to a second microcontroller. Finally, two rotameters (Model No. MMA-4, Dwyer Instruments, Michigan City, IN, USA) were placed at the inlet and outlet of Section C to confirm Q downstream and assure the D-GRMS was airtight or leak-free.

3.2. Soybeans and sample preparation

Soybeans (Pioneer 28T33R, Pioneer Hi-Bred, Johnston, Iowa, USA) were harvested at approximately 15% wet basis moisture content from the Crop Sciences Research and Education Farm at the University of Illinois in Urbana, IL in October 2016 and stored in a grain bin where they were dried to 12-13%. On January 19, 2017, soybeans (327 kg or 12 bu) were removed from the bin, placed in plastic storage containers (18 gal capacity), and stored at 4°C until testing.

Prior to the start of each test, soybeans were manually mixed in the container and a 3 kg sample was removed (Figure 3.2). The sample was hand cleaned using sieves (Grainman 0.39 cm x 1.9 cm, Miami, FL, USA) to remove broken beans, or splits, and foreign material. The beans were cleaned in batches to ensure removal of all undesired material. After cleaning, the sample was poured into a thin layer on a metal tray and allowed to acclimate to room temperature (approximately 20 to 22°C) for 30 to 40 min. A handheld moisture meter (Model No. SW16060, John Deere, Moline, IL, USA) was used to estimate the initial moisture content of the sample ($\widehat{w}_{soy,0}$), which was used to calculate the amount of deionized water (m_{H_2O}) necessary to be added to adjust the moisture content of 2.4 kg ($m_{soy,0}$) of clean soybeans to 14, 18, or 22% ($\widehat{w}_{soy,1}$):

$$m_{H_2O} = m_{soy,0}\left(\frac{\widehat{w}_{soy,1} - \widehat{w}_{soy,0}}{100 - \widehat{w}_{soy,1}}\right) \qquad [3.1]$$

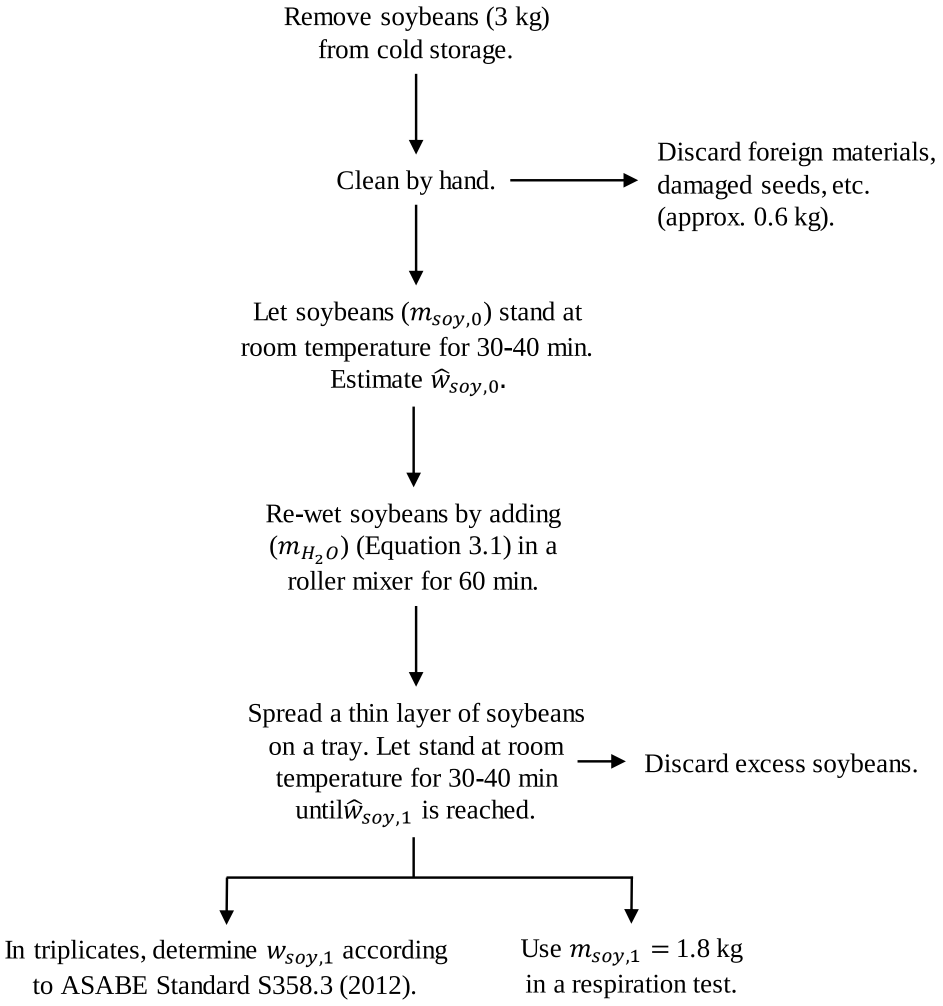


Figure 3.2. Preparation of soybeans for a respiration test involved hand-cleaning, re-wetting and determining its moisture content.

The 2.4 kg of clean soybeans were split into two batches of 1.2 kg each and placed into two round plastic jars (2 L capacity). Deionized water equal to $0.5m_{H_2O}$ according to Equation 3.1 was added to each jar. An extra 5 g of water was also added to each jar for good measure. The filled jars were placed on a roller mixer (Scilogex MX-T6-S, Rocky Hill, CT, USA), which was set at 60 rpm and left to run for 60 min. Afterward, the beans were poured as a thin layer on a metal tray and allowed to air-dry at room temperature for 30 to 40 min. $\hat{w}_{soy,1}$ was monitored using the handheld moisture meter every 10 min, until the desired moisture content was achieved.

After beans were rewetted, three subsamples (25 to 30 g each) were removed for gravimetric moisture determination ($w_{soy,1}$), in an oven at 103°C for 72 h, according to ASABE Standard S352.2 (R2017). A 400 g subsample was also removed, placed in a plastic bag, sealed, and stored at -20°C for future lipid oxidation tests (not included in this study). No initial samples were saved for the first objective of the thesis. Lastly, approximately 1800 g ($m_{soy,1}$) of rewetted soybeans were poured into the RC of D-GRMS for testing. Excess soybeans were discarded. The moisture content obtained gravimetrically ($w_{soy,1}$) was used to calculate the initial amount of dry matter ($m_{DM,1}$) present in the sample:

$$m_{DM,1} = m_{soy,1}\left(1 - \frac{w_{soy,1}}{100}\right) \quad [3.2]$$

The SOPs on preparing soybeans for a grain respiration test (Document No. D-GRMS-005) and gravimetric measurement of grain moisture content (Document No. D-GRMS-006) are available in Appendix A.

3.3. D-GRMS preparation and respiration test initiation

Two RC dehumidifiers and two RC CO_2 scrubbers were prepared and the initial masses of the scrubbers [$\left(m_{RC,A}\right)_t$ and $\left(m_{RC,B}\right)_t$ at $t = 0$] were determined using a digital scale (Model i3100, MyWeight, Phoenix, AZ). The dehumidifiers and scrubbers were placed into the D-GRMS using quick disconnect connectors and three-way valves, with airflow set to pass through RC dehumidifier A and RC CO_2 scrubber A. A soybean-filled RC was installed in the

D-GRMS. The air supply and all water baths were turned ON and allowed to run for 30 to 40 min. All sensor readings were checked to confirm they were within respiration test conditions ($T = 35 \pm 2$ °C, $C_{CO_2} \leq 20$ ppm and $\phi = 79$, 89, or 92 ± 5 %RH for w = 14, 18 and 22%, respectively). Adjustments to the water bath temperature or glycerol-water solution concentration were made accordingly. Once these conditions were met, a respiration test was started by recording the time. The SOP on running a grain respiration test (Document No. D-GRMS-007) is available in Appendix A.

3.4. Respired CO_2 measurements

The system was allowed to equilibrate for a period of 12 to 14 h. After this initial period, airflow was directed to RC CO_2 scrubber B so that RC CO_2 scrubber A could be removed from the system, weighed three times (with the scrubber turned 120° clockwise in between measurements), and corresponding date and time recorded. The average of three weight measurements $[(\overline{m_{RC}})_{A,t}]$ was computed. $[(\overline{m_{RC}})_{A,t}]$ increased over time as the mass of respired CO_2 was captured in the CO_2 absorbent material and accumulated:

$$\left(\sum m_{CO_2}\right)_A = \left(\overline{m_{RC,A}}\right)_t - \left(\overline{m_{RC,A}}\right)_0 \quad [3.3]$$

Afterwards, RC scrubber A was installed back in the D-GRMS. The respired CO_2 was allowed to accumulate in RC scrubber B for 2 h and the airflow was diverted back to RC scrubber A. The same weight measurement procedures were conducted and

$$\left(\sum m_{CO_2}\right)_B = \left(\overline{m_{RC,B}}\right)_t - \left(\overline{m_{RC,B}}\right)_0 \quad [3.4]$$

Hence, at any time t, the total accumulated mass of respired CO_2 was

$$\left(\sum m_{CO_2}\right)_{A+B} = \left(\sum m_{CO_2}\right)_A + \left(\sum m_{CO_2}\right)_B = \left(\sum m_{CO_2}\right)_S m_{DM,1} \quad [3.5]$$

which, when divided by the mass of dry matter in the soybeans ($m_{DM,1}$) was the specific accumulated mass of respired CO2, $\left(\sum m_{co_2}\right)_s$.

Respired CO2 measurements were taken approximately every 2 h thereafter, during daytime hours. No measurements were taken overnight. The system was equipped with two RC CO2 scrubbers so that respired air could flow continuously during periods when one of the RC CO2 scrubbers needed to be weighed. The system was also equipped with two RC dehumidifiers so that respiration testing could proceed for extended periods (12 to 15 d). When RC dehumidifier A had absorbed 75% of its capacity by visual observation that the bottom ¾ of the desiccant bed had turned from blue to pink color), airflow was diverted to the second dehumidifier. The first dehumidifier was then removed from the system, replenished with fresh desiccant, weighed for its new tare, and available for the next changeover in RC. All data were recorded in a spreadsheet (Appendix B) using Microsoft Excel (Version 2016, Microsoft Corporation, Redmond, WA, USA).

3.5. Ending a respiration test and shutting down D-GRMS

A respiration test proceeded until $\left(\sum m_{CO_2}\right)_s = 22$ g CO2 (kg dry beans)$^{-1}$ were absorbed in combination by both RC CO2 scrubbers, which is equivalent to 1.5% DML, or three times the 0.5% DML threshold typically recommended for safe storage of shelled corn (ASABE, R2014). At the end of a respiration test, airflow was shut off and water baths were turned off. The soybeans were poured onto a metal tray and manually mixed. Triplicate samples were taken to estimate $\widehat{w}_{soy,2}$ using the handheld moisture meter. Three samples (25 to 30 g each) were also removed for gravimetric moisture content ($w_{soy,2}$) determination (ASABE, R2017). All CO2 scrubbers, dehumidifiers, and RC were cleaned with soap and hot water, rinsed with ethanol, and dried for subsequent respiration tests.

3.6. Dry matter loss calculation and time parameters

The amount of DML at any time t was determined using the stoichiometric relationship between glucose and carbon dioxide in the respiration equation and $\left(\sum m_{co_2}\right)_s$ in Equation 2.1 and is included in Appendix B. To facilitate calculations of elapsed time during a respiration test, logged dates and times in the Gregorian calendar were converted to Julian date (JD):

$$JD = \overbrace{(YY)}^{\text{MM/DD/YYYY}}10^3 + D_j + \overbrace{\frac{hh}{24\,h\,d^{-1}} + \frac{mm}{1440\,\mathrm{min}\,d^{-1}}}^{\text{hh:mm}} \tag{3.6}$$

where the last two digits of the Gregorian year were multiplied by 10^3 and added to the total number of days since January 1 of the same year (D_j) and the fraction of day. The hours and minutes ($hh\!:mm$) were stated in military time, which were converted to fraction of day by dividing hh by 24 hours d^{-1} and mm by 1440 min d^{-1}.

Preliminary tests showed that respiration exhibited a “lag” period (t_{lag}) from the time soybeans were placed in D-GRMS to the time when an appreciable inflection point in DML vs. t curve could be detected (Figure 3.3). This lag phase may be a combination of the rewetted beans acclimating to their new environment and the respiration measurement system coming to equilibrium. Thus, the start time of grain respiration (t_{start}) was initially designated as the time at which DML reached 0.05% ($t_{0.05}$). Grain respiration tests were terminated when DML exceeded 1.5%, and the endpoint of the respiration test set at the time when 1.5% DML was reached ($t_{1.5}$). The lower limit is one order of magnitude below the 0.5% DML threshold used in MAST guidelines for shelled corn and the upper limit is three times this valuation threshold. Since it is not often convenient to obtain a CO_2 measurement at exactly when $\left(\sum m_{CO_2}\right)_s =$

0.733 g kg^{-1} (0.05% DML) and $\left(\sum m_{CO_2}\right)_s$ =22.0 g kg^{-1} (1.5% DML) are reached, $t_{0.05}$ and $t_{1.5}$ were estimated by linear interpolation using two respired CO$_2$ measurements – one immediately above and one immediately below these DML limits. Later, t_{start} was adjusted to the time to reach 0.10% DML ($t_{0.10}$).

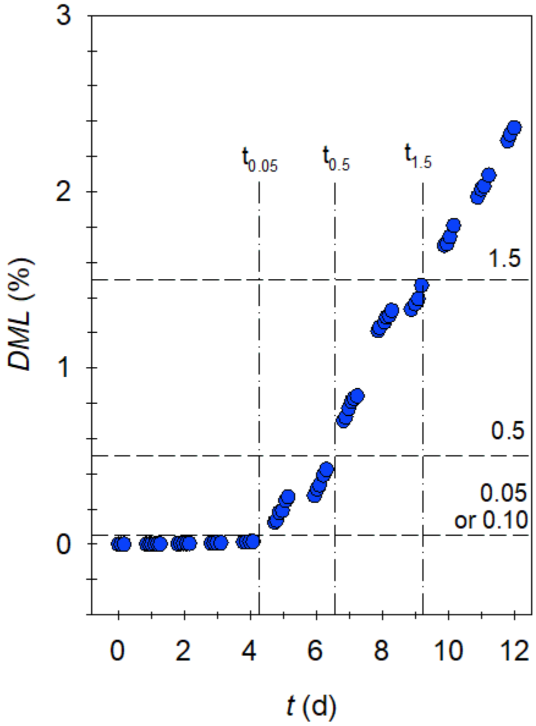


Figure 3.3. Preliminary tests indicated present of lag period. Start of a grain respiration test was set to time to reach 0.05 or 0.10% DML ($t_{0.05}$ or $t_{0,10}$, respectively) and ended when 1.5% DML was reached ($t_{1.5}$).

3.7. Statistical analyses

All code and outputs from statistical analyses from the Data Analysis ToolPak (Version 2016, Microsoft Excel, Microsoft Corporation, Redmond, WA, USA) and SAS Studio software, Version 4.2, Copyright ® 2017 SAS Institute Inc. are available in Appendix C. SAS and all other SAS Institute Inc. product or service names are registered trademarks or trademarks of SAS Institute Inc., Cary, NC, USA.

3.7.1. Soybean moisture contents

Initial soybean w estimated by handheld moisture meter ($\widehat{w}_{soy,1}$) were compared to gravimetric w measurements taken before the respiration test ($w_{soy,1}$) by calculating their difference $\left(\Delta w_{soy}\right)_1$ and checking if it is zero in an effort to evaluate the use of a handheld meter to estimate w and the efficacy of the rewetting procedure. Likewise, soybean moisture contents before ($w_{soy,1}$) and after ($w_{soy,2}$) the respiration test were compared by calculating their difference $\left(\Delta w_{soy}\right)_{1\rightarrow 2}$ was zero to evaluate the efficacy of the air conditioning mechanism of D-GRMS. Means comparisons were made using paired samples and a two-tailed Student's *t*-test to test the following hypotheses at $\alpha = 0.05$:

$$\textit{Hypotheses 1} \quad H_0\text{: } \overline{\left(\Delta w_{soy}\right)_1} = \frac{\Sigma\left(\widehat{w}_{soy,1} - w_{soy,1}\right)_k}{k} = 0 \qquad [3.7]$$

$$H_A\text{: } \overline{\left(\Delta w_{soy}\right)_1} \neq 0$$

$$\textit{Hypotheses 2} \quad H_0\text{: } \overline{\left(\Delta w_{soy}\right)_{1\rightarrow 2}} = \frac{\Sigma\left(w_{soy,1} - w_{soy,2}\right)_k}{k} = 0 \qquad [3.8]$$

$$H_A\text{: } \overline{\left(\Delta w_{soy}\right)_{1\rightarrow 2}} \neq 0$$

where is the *k* is the replication number.

3.7.2 Lag times and time to reach 0.5% dry matter loss

The first set of grain respiration tests conducted were for 18% moisture content soybeans, which included five replications. First, DML data were plotted against t, which were transformed to remove t_{lag} (Figure 3.3). The start time was adjusted by subtracting $t_{0.05}$ or $t_{0.10}$ to yield t' and DML was adjusted by subtracting from the time, t, either 0.05 or 0.10% DML to yield DML' (Figure 3.4). These transformations effectively repositioned the origin from $(0{,}0)$ to $(t_{0.05}, 0.05)$ or $(t_{0.10}, 0.10)$. These transformations were repeated with data for 14 and 20% moisture content soybeans. Estimates of t_{lag} and $t_{0.5}$, which includes t_{lag}, were compared across w treatments and between t_{start} values using PROC ANOVA function in SAS Studio software. Significance of differences were tested at $a = 0.05$.

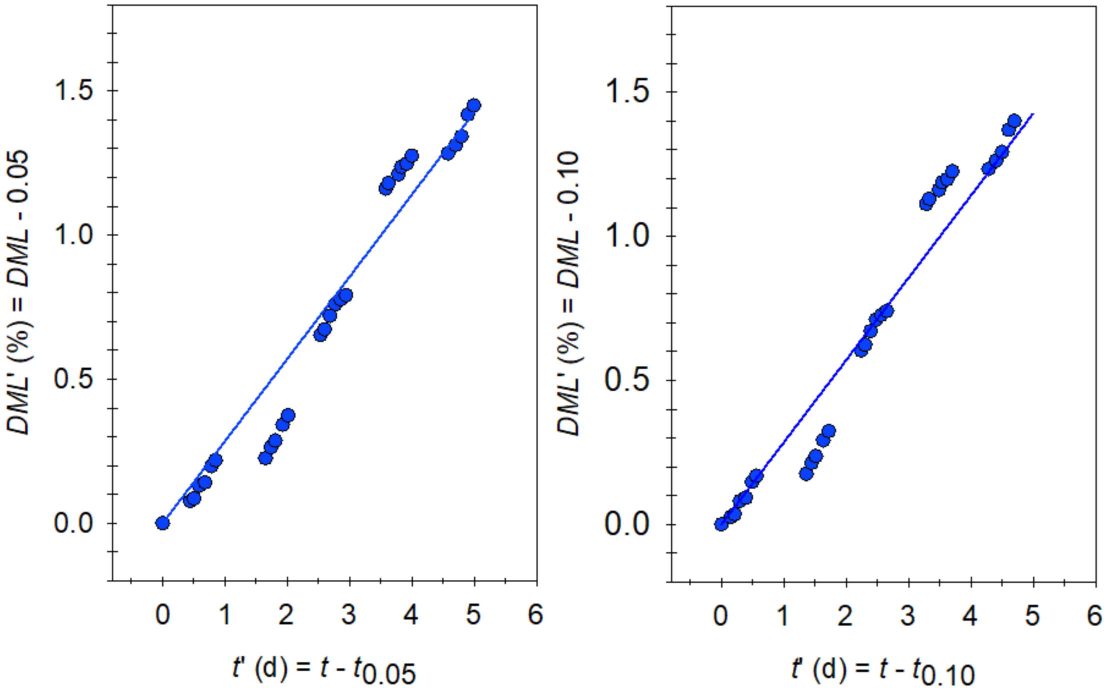


Figure 3.4. Dry matter loss (DML) and time (t) axes were adjusted to remove the lag period (t_{lag}).

3.7.3. Dry matter loss rates of 18% moisture content soybeans

Following the data transformation, a linear regression was performed using the following model and the linear regression option in the Data Analysis ToolPak in MS Excel:

$$DML' = v_{DML}t' + DML'_0 \qquad [3.9]$$

where the intercept (DML'_0) was set to zero. Outputs of the linear regression for each replication included:

- regression statistics – coefficient of determination (R^2), standard error of regression (SE_{reg}),
- analysis of variance (ANOVA) table, and

- estimate of the slope and its standard error ($v_{DML} \pm SE_{v_{DML}}$).

The overall DML rate and its standard error $\left(v_{DML} \pm SE_{v_{DML}}\right)_p$ for each w treatment were determined by pooling the data from all replications and conducting a linear regression using Data Analysis ToolPak. It was initially assumed that $\left(\sigma_{v_{DML}}\right)_p$, calculated as follows, may be a better estimate of the variability of v_{DML} estimates amongst replications:

$$\left(\sigma_{v_{DML}}\right)_p = \sqrt{\frac{\sum_{i=1}^{k}(n_i - 1)\left(SE_{v_{DML}}\right)_i^2}{\sum_{i=1}^{k}(n_i - 1)}} \qquad [3.10]$$

where n was the number of observations in a replication, $SE_{v_{DML}}$ was the standard error of the slope of regression, and the replicated respiration tests were indexed $i = 1, \dots, k = 5$. Hence, a comparison at α = 0.05 between $\left(SE_{v_{DML}}\right)_p$ and $\left(\sigma_{v_{DML}}\right)_p$ was performed, using PROC ANOVA in SAS Studio, in order to identify significant differences between these two values.

3.7.4. Statistical power and minimum number of replications

Using $\left(\sigma_{v_{DML}}\right)_p$ determined with $t_{start} = t_{0.05}$ from respiration tests with 18% moisture content soybeans, a statistical power analysis was conducted using a few assumptions. First, v_{DML} could be treated as a "mean" value representing n observations taken during a respiration test (*a.k.a.*, a population). As more replications were conducted, v_{DML} of each replication may be different but the standard deviation, or variance, of each population was the same. Therefore, the overall standard deviation of v_{DML} could be estimated by squaring, summing, and square rooting the individual population's standard deviations (Equation 3.10) to give a higher precision estimate of standard deviation than an individual population's.

Second, the value of $\left(\sigma_{v_{DML}}\right)_p$ was comparable among treatments. Finally, assuming a normally distributed response to a treatment, the size of δ between treatment means chosen in statistical power analysis is critical to determining r_{min}. For small values of δ, there is potential for a large overlap of response variables between treatments, resulting in a large r_{min} (Figure 3.5a). The overlap across treatment means decrease with increasing δ, as shown in Figures 3.5b and 3.5c.

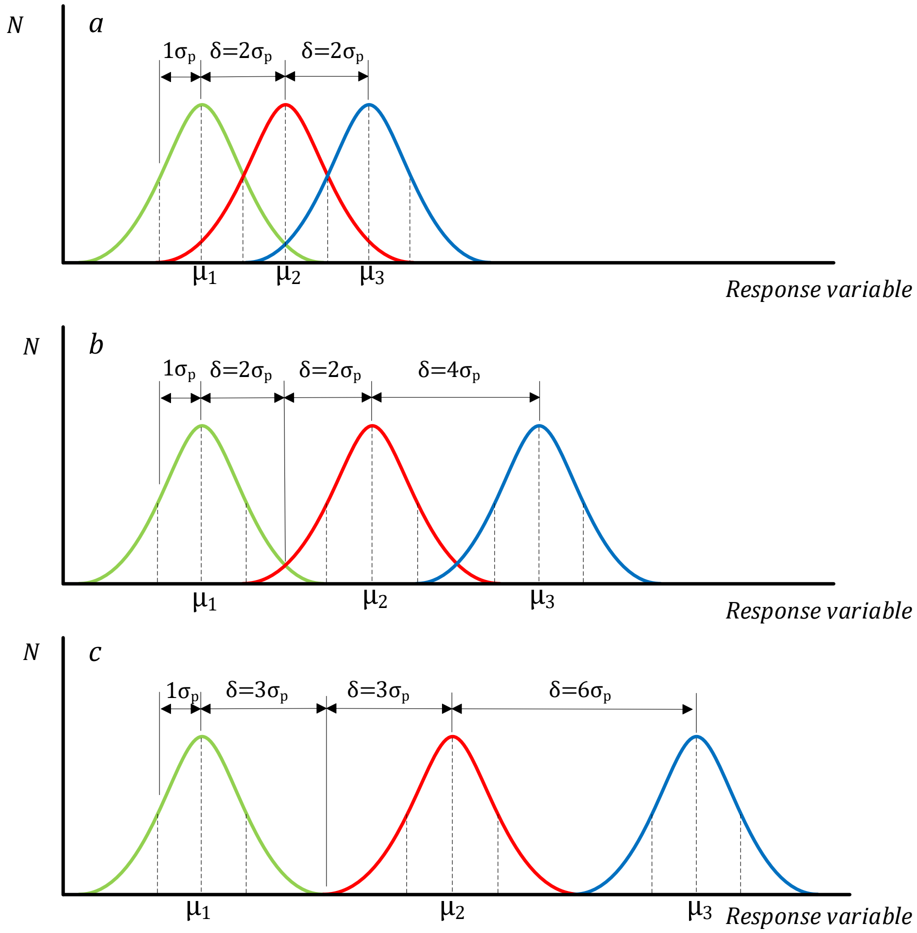


Figure 3.5. Effects of difference size (δ) used in statistical power analysis, assuming normally distributed responses around treatment means (μ) with a pooled standard deviation, σ_p. The subscripts 1, 2, and 3 represent treatment number.

The value for r_{min} was calculated two ways first, choosing a δ value and, second, using the coefficient of variance $\left[CV = \left(\sigma_{v_{DML}}\right)_p / (v_{DML})_p\right]$ in Equation 2.4. A value of $\delta = 4\left(\sigma_{v_{DML}}\right)_p$ was chosen since 95% of v_{DML} values are expected to surround each treatment mean, resulting in little overlap in v_{DML} among treatments. Since $\sigma \approx \left(\sigma_{v_{DML}}\right)_p$, the term $(\sigma/\delta) = ¼$ or 0.25. Values of t_1 and t_2 were determined from a two-tailed Student's *t*-test table,

$$t_1 = t(\alpha/2\,, df) \qquad [3.11]$$

$$t_2 = t[2(1-\beta), df] \qquad [3.12]$$

where $\alpha = 0.05$, $\beta = 0.8$. The number of degrees of freedom (df) is

$$df = (r-1)(j-1) \qquad [3.13]$$

where r is the number of replications and j is the total number of treatment combinations (e.g., three levels of w and one levels of T would give a $j = 3 \times 1 = 3$). Here, we see that calculating r_{min} is an iterative process that starts with an initial guess ($\hat{r}_{min}$) or estimate of r_{min}, and terminates when solution has converged (i.e., $\hat{r}_{min} = r_{min}$).

3.7.5. Dry matter loss rates of 14 and 22% moisture content soybeans

The set of statistical analyses described in Section 3.7.3 were repeated for a completely randomized 2w x 1T x r_{min} experiment using 14 and 22% moisture content soybeans using the r_{min} determined in Section 3.7.4. The estimate of r_{min} was re-evaluated using $\left(\sigma_{v_{DML}}\right)_p$ of 14% and 22% moisture content soybeans and the experiment was adjusted, accordingly. A one-way ANOVA and Tukey's multiple pairwise comparison of v_{DML} amongst w treatments and between t_{start} values were conducted using PROC ANOVA function in SAS Studio software. Significance of differences were tested at $a = 0.05$.

3.7.6. Respiration rates of 14 to 22% moisture content soybeans

All $\left(v_{DML} \pm SE_{v_{DML}}\right)_p$ values were converted to $\left(v_{CO_2} \pm SE_{v_{CO_2}}\right)_p$ using Equation 2.2 for easy comparison to grain deterioration rates reported in the literature.

3.7.7. Mathematical models of dry matter loss rate

Sorour and Uchino (2004) had the widest range of w (18 to 26%) and storage T (15 to 30^oC) experiment ever conducted using a D-GRMS for soybean respiration. The linear models they developed for predicting $\sum m_{co_2,s}$ over time fit their respiration data well ($R^2 > 0.99$). Hence, their models were used to regenerate their data and calculate v_{DML} according to procedures described in Section 3.7.3. Details on data regeneration and linear regressions to calculate v_{DML} are in Appendix C. The v_{DML} estimates from Sorour and Uchino's (2004) study and from this study were fitted to mathematical models based on Models 3, 8 and 9 (Table 2.1) to describe the effects of w on v_{DML}. Nonlinear modeling was accomplished using the PROC NLIN function in SAS Studio software and goodness-of-fit was based on mean relative error (MRE), standard (SE_{reg}), F-statistic, and estimated coefficient of determination ($\hat{R}^2$) of the nonlinear regression, as well as the random nature of the residual plots.

CHAPTER 4. RESULTS AND DISCUSSION

4.1. Soybean moisture contents

For soybeans classified in the 14% moisture content group, the average moisture content estimated with a handheld meter was 14.37%, which was no different than the gravimetric moisture content measurement of 14.33% ($p = 0.86$). The same trend was found with soybeans classified in the 18% moisture content group, where the average estimated, and gravimetric measurements were 18.36% and 18.47%, respectively ($p = 0.44$). However, these values were found to be different with 22% moisture content soybeans group, where the average gravimetric moisture measurement (21.90%) was much higher than the average estimated moisture content (21.60%) ($p = 0.01$). This significance was due to a 0.51% difference in one of the four replications. Also, the moisture content estimates via the handheld meter were consistently lower than the gravimetric moisture content measurements at 22%, but the trend was not found with 14 and 18% moisture content soybeans. These results suggested that, for moisture contents below 22%, the handheld meter may be used to estimate the moisture content at the start of a grain respiration test. Estimates, however may be as far off as 0.5% from the gravimetric measurements for higher soybean moisture contents, so caution must be exercised at using and relying solely on the handheld meter.

Likewise, the average moisture contents before (14.33%) and after (14.36%) grain respiration tests with 14% moisture content soybeans were not different ($p = 0.91$). The same was true for 18% moisture content soybeans (before, 18.47% and after, 18.22%, $p = 0.27$). However, differences were observed between the average moisture contents before (21.90%) and after (21.65%) grain respiration tests with 22% moisture content soybeans ($p = 0.04$). These results suggested that maintaining the moisture content via humidification during respiration tests may require using slightly more humid air.

Table 4.1. Comparison of estimated and actual soybean moisture contents (%) before and after replicated respiration tests. Variables are defined in the footnotes.

Treatment	Before respiration test		After respiration test	Absolute differences	
w^1	$\hat{w}_{soy,1} \pm \sigma_{\hat{w}_{soy,1}}$	$w_{soy,1} \pm \sigma_{w_{soy,1}}$	$w_{soy,2} \pm \sigma_{w_{soy,2}}$	$\left\|(\Delta w_{soy})_1\right\|$	$\left\|(\Delta w_{soy})_{1\rightarrow 2}\right\|$
14	14.23 ± 0.11	14.31 ± 0.002	14.57 ± 0.002	0.08	0.26
	14.67 ± 0.11	14.26 ± 0.002	14.57 ± 0.001	0.41	0.31
	14.43 ± 0.15	14.73 ± 0.000	14.51 ± 0.001	0.30	0.22
	14.13 ± 0.06	14.02 ± 0.001	13.79 ± 0.001	0.11	0.23
$\mu \pm \sigma =$	14.37 ± 0.24$^{d\ 2}$	14.33 ± 0.30^{dD}	14.36 ± 0.38^{D}		
18	18.27 ± 0.06	18.40 ± 0.001	17.78 ± 0.002	0.13	0.62
	18.63 ± 0.15	18.51 ± 0.002	17.88 ± 0.005	0.12	0.63
	18.13 ± 0.06	18.26 ± 0.001	18.82 ± 0.002	0.13	0.56
	18.60 ± 0.10	18.80 ± 0.001	18.50 ± 0.001	0.20	0.30
	18.17 ± 0.06	18.40 ± 0.001	18.11 ± 0.001	0.23	0.29
$\mu \pm \sigma =$	18.36 ± 0.24^{c}	18.47 ± 0.20^{cC}	18.22 ± 0.44^{C}		
22	21.57 ± 0.06	22.08 ± 0.001	21.80 ± 0.001	0.51	0.28
	21.67 ± 0.12	21.88 ± 0.003	21.47 ± 0.001	0.21	0.41
	21.67 ± 0.05	21.78 ± 0.002	21.67 ± 0.001	0.11	0.11
	21.53 ± 0.06	21.86 ± 0.001	21.66 ± 0.000	0.33	0.20
$\mu \pm \sigma =$	21.61 ± 0.07^{b}	21.90 ± 0.13^{aA}	21.65 ± 0.14^{B}		

[1] Moisture content (w) variables: estimated moisture content via handheld meter, $\hat{w}_{soy,1}$; gravimetric moisture content before grain respiration test, $w_{soy,1}$; gravimetric moisture content after grain respiration test, $w_{soy,2}$; difference between estimated and gravimetric moisture content before grain respiration test, $(\Delta w_{soy})_1$; and difference between moisture contents before and after a grain respiration test, $(\Delta w_{soy})_{1\rightarrow 2}$. All moisture contents are reported as mean ± standard deviation and in % wet-basis.

[2] Treatment means and standard deviations ($\mu \pm \sigma$ % w.b.) with the same letter are not different from each other. Lowercase letters were used to denote differences in $(\Delta w_{soy})_1$ while uppercase letters were used to denote differences in $(\Delta w_{soy})_{1\rightarrow 2}$.

4.2. Lag times and time to reach 0.5% dry matter loss

As previously mentioned, all soybeans tested exhibited a lag period prior to steadily losing dry matter during storage, with t_{lag} decreasing with increasing w (Table 4.2). When $t_{start} = t_{0.05}$, average t_{lag} values for 14, 18, and 22% moisture content soybeans were 4.34, 3.65, and 0.94 d. These values were not different when $t_{start} = t_{0.10}$, which due to the large standard deviation in each w. Across treatments, t_{lag} decreased with increasing w and were different from each other.

Table 4.2. Lag times of respiration tests with 14 to 22% moisture content soybeans in D-GRMS. Variables are defined in the footnotes.

Moisture content, w^1 (% w.b.)	t_{lag} (d)			
	When $t_{start} = t_{0.05}$	n	When $t_{start} = t_{0.10}$	n
14	1.79	9	2.24	13
	2.53	13	3.87	21
	7.11	36	7.39	36
	5.91	30	6.30	30
	$\mu \pm \sigma = 4.34 \pm 2.58^{aA}$ [2]		$\mu \pm \sigma = 4.95 \pm 2.33^{aA}$	
18	5.04	23	5.34	26
	4.28	22	4.58	22
	4.09	22	4.75	24
	0.79	2	0.86	3
	4.07	23	4.68	25
	$\mu \pm \sigma = 3.65 \pm 3.85^{aA}$		$\mu \pm \sigma = 4.04 \pm 1.81^{aA}$	
22	1.04	6	1.40	6
	0.90	5	1.32	5
	0.91	6	1.11	6
	0.90	6	1.51	6
	$\mu \pm \sigma = 0.94 \pm 0.07^{bB}$		$\mu \pm \sigma = 1.33 \pm 0.17^{bA}$	

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[1] Variables: moisture content, w (% w.b.); start of grain respiration test, t_{start} (d); time to reach 0.05 or 0.10% DML, $t_{0.05}$ or $t_{0.10}$ (d), respectively; lag time, t_{lag} (d); number of data points or observations in the lag period, n.

[2] Treatment means and standard deviations ($\mu \pm \sigma$) with the same letter are not different from each other. Lowercase letters denote differences within a column ($t_{start} = t_{0.05}$ or $t_{start} = t_{0.10}$); uppercase letters denote differences within a row (w treatments).

For all w tested, DML increased steadily over time and estimates of $t_{0.5}$ decreased with increasing w, after the lag period was removed (Table 4.3 and Figure 4.1). For both considerations ($t_{start} = t_{0.05}$ or $t_{start} = t_{0.10}$), estimates of $t_{0.5}$ were not different from each other at 18 and 22% moisture content. However, 14% moisture content soybeans presented estimate $t_{0.5}$ different from the ones for 18 and 22%.

Table 4.3. Time to reach 0.5% dry matter loss for 14 to 22% moisture content soybeans in D-GRMS. Variables are defined in the footnotes.

Moisture content, w^1 (% w.b.)	Including t_{lag} (d)	Excluding t_{lag} (d)	
		when $t_{start} = t_{0.05}$	when $t_{start} = t_{0.10}$
14	4.91	3.69	3.61
	7.34	5.24	4.26
	11.82	5.13	5.17
	8.80	3.21	3.14
$\mu \pm \sigma =$	8.22 ± 2.89^{aA}	4.32 ± 1.02^{aB}	4.04 ± 0.88^{aB}
18	6.59	1.66	1.47
	6.44	2.25	2.05
	6.64	2.79	2.36
	3.26	2.64	2.73
	6.50	2.54	2.12
$\mu \pm \sigma =$	5.88 ± 1.47^{bA}	2.38 ± 0.45^{bB}	2.15 ± 0.46^{bB}
22	3.32	2.45	2.25
	2.95	2.16	1.85
	2.56	1.82	1.79
	3.83	3.12	2.67
$\mu \pm \sigma =$	3.16 ± 0.54^{bA}	2.39 ± 0.55^{bA}	2.14 ± 0.41^{bB}

[1] Variables: moisture content, w (% w.b.); time to reach 0.5% DML, $t_{0.5}$ (d); start of grain respiration test, t_{start} (d); time to reach 0.05 or 0.10% DML, $t_{0.05}$ or $t_{0.10}$ (d), respectively; lag time, t_{lag} (d).

[2] Treatment means and standard deviations ($\mu \pm \sigma$) with the same letter are not different from each other. Lowercase letters denote differences within a column ($t_{start} = t_{0.05}$ or $t_{start} = t_{0.10}$); uppercase letters denote differences within a row (w treatments).

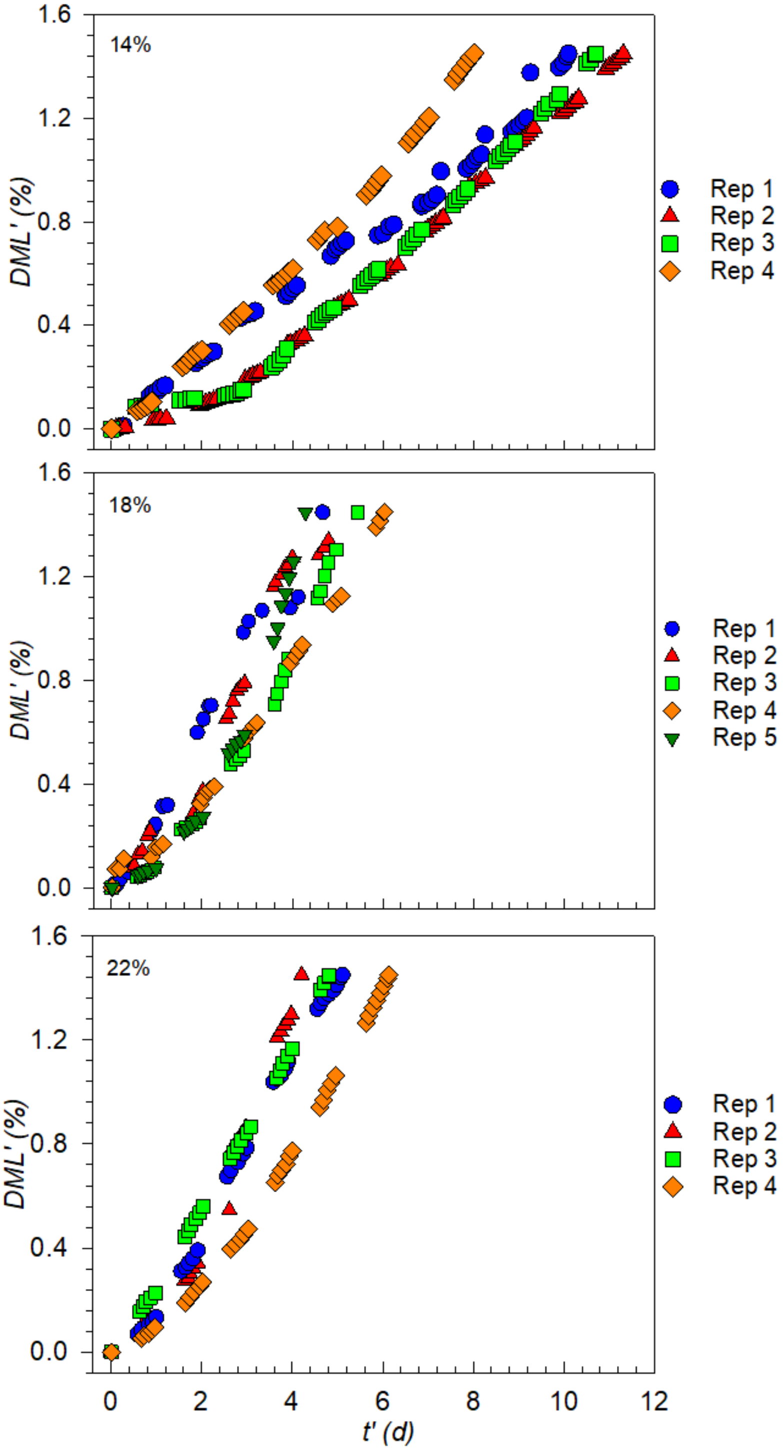

Figure 4.1. Dry matter loss (DML) of 14 to 22% moisture content soybeans at 35°C. The lag period prior to reaching 0.05% DML was removed.

4.3. Dry matter loss rates of 14 to 22% moisture content soybeans

Regardless of whether the start of a respiration test was set at $t_{0.05}$ or $t_{0.10}$, v_{DML} were linear and increased with increasing w (Table 4.4). Estimates of v_{DML} ranged from 0.117 to 0.169% d^{-1} for 14% moisture content soybeans; from 0.222 to 0.304% d^{-1} for 18% moisture content soybeans; and from 0.210 to 0.297% d^{-1} for 22% moisture content soybeans. The arithmetic mean v_{DML} value for 14% moisture content soybeans (denoted μ in Table 4.4) was different from those at 18 and 22% moisture content soybeans ($p = 0.001$ and 0.002, respectively) even though the average v_{DML} values for 18 and 22% moisture content soybeans were not different from each other ($p = 0.70$). Average v_{DML} values obtained with either t_{start} values were not different from each other (p = 0.85) with each w treatment.

The $SE_{v_{DML}}$ for each replication tended to increase with increasing w, as was expected, since high w conditions promote both grain respiration and mold growth, which could be highly variable across grain samples. The lowest $SE_{v_{DML}}$ values were observed with 14% moisture content soybeans, which also had the lowest v_{DML}. When DML data were pooled for each w treatment, resulting $(v_{DML})_p$ values were not different to the arithmetic mean v_{DML} values ($p = 0.793$), despite consistently being lower, and estimates of variability – $SE_{v_{DML}}$, $\left(SE_{v_{DML}}\right)_p$, and $\left(\sigma_{v_{DML}}\right)_p$ – were comparable with each other (Table 4.4). In fact, $\left(SE_{v_{DML}}\right)_p$ and $\left(\sigma_{v_{DML}}\right)_p$ were not different from each other ($p = 0.40$). However, the transformation of both t and DML that was made effectively shifted the origin (Section 3.7.2). In retrospect, a shift of only the time variable may be more informative as to the best choice for t_{start}.

Table 4.4. Dry matter loss rates of 14 to 22% moisture content soybeans. Variables are defined in the footnotes.

Moisture content, w (% w.b.)	$t_{start} = t_{0.05}$[1]				$t_{start} = t_{0.10}$			
	n	$v_{DML} \pm SE_{v_{DML}}$ (% d^{-1})	$\left(\sigma_{v_{DML}}\right)_p$ (% d^{-1})	$\left(v_{DML} \pm SE_{v_{DML}}\right)_p$ (% d^{-1})	n	$v_{DML} \pm SE_{v_{DML}}$ (% d^{-1})	$\left(\sigma_{v_{DML}}\right)_p$ (% d^{-1})	$\left(v_{DML} \pm SE_{v_{DML}}\right)_p$ (% d^{-1})
14	56	0.134 ± 0.001			52	0.136 ± 0.001		
	63	0.117 ± 0.002			55	0.134 ± 0.001		
	57	0.119 ± 0.002			57	0.117 ± 0.003		
	44	0.169 ± 0.001			44	0.171 ± 0.001		
	$\mu \pm \sigma =$	0.135 ± 0.024^{bA}	0.002	0.128 ± 0.001	$\mu \pm \sigma =$	0.140 ± 0.023^{bA}	0.002	0.134 ± 0.002
18	19	0.304 ± 0.006			16	0.319 ± 0.007		
	29	0.285 ± 0.007			29	0.297 ± 0.007		
	27	0.227 ± 0.008			25	0.262 ± 0.007		
	30	0.222 ± 0.004			29	0.213 ± 0.005		
	24	0.256 ± 0.014			22	0.305 ± 0.014		
	$\mu \pm \sigma =$	0.259 ± 0.036^{aA}	0.008	0.250 ± 0.004	$\mu \pm \sigma =$	0.279 ± 0.042^{aA}	0.008	0.263 ± 0.005
22	21	0.297 ± 0.012			21	0.322 ± 0.012		
	29	0.277 ± 0.005			29	0.299 ± 0.003		
	27	0.291 ± 0.002			27	0.295 ± 0.002		
	35	0.210 ± 0.005			35	0.232 ± 0.004		
	$\mu \pm \sigma =$	0.269 ± 0.040^{aA}	0.005	0.253 ± 0.005	$\mu \pm \sigma =$	0.287 ± 0.039^{aA}	0.003	0.273 ± 0.004

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[1] Variables: moisture content, w (% w.b.); start of grain respiration test, t_{start} (d); time to reach 0.05 or 0.10% DML, $t_{0.05}$ or $t_{0.10}$ (d), respectively; number of data points or observations used in the regression, n; per replication, DML rate and its standard error, $v_{DML} \pm SE_{v_{DML}}$ (% d^{-1}); pooled standard deviation, $\left(\sigma_{v_{DML}}\right)_p$ (% d^{-1}); and overall pooled DML rate and its standard error, $\left(v_{DML} \pm SE_{v_{DML}}\right)_p$ (% d^{-1}).

[2] Treatment means and standard deviations ($\mu \pm \sigma$) with the same letter are not different from each other. Lowercase letters denote differences within a column (t_{start}); uppercase letters denote differences within a row (w treatments).

Overall, v_{DML} values obtained in this study were higher than those reported in the literature, which ranged from 0.003 to 0.050 % d^{-1} (Table 2.2), owing to a variety of factors – storage conditions tested, accuracy and precision of CO_2 measurement method, soybean cultivar, etc. Previous studies focused on temperate conditions typical in North America and Europe rather than the tropical conditions in Brazil and other soybean-producing regions in low-latitude countries. Corresponding $\left(v_{CO_2} \pm SE_{v_{CO_2}}\right)_p$ and $t_{0.5} \pm \sigma_{t_{0.5}}$ (including t_{lag}) to $(v_{DML} \pm SE_{DML})_p$ values obtained in this study are presented in Table 4.5.

Table 4.5. Grain deterioration rates for 14 to 22% moisture content soybeans stored at 35°C. Variables are defined in the footnotes.

Moisture content, w^1 (% w.b.)	Respiration rate [mg CO_2 (kg d)$^{-1}$]	Time to 0.5% DML (d)	DML rate (10^{-3} % d^{-1})	Replications, r
	$\left(v_{CO_2} \pm SE_{v_{CO_2}}\right)_p$	$t_{0.5} \pm \sigma_{t_{0.5}}$ ^{3}	$(v_{DML} \pm SE_{DML})_p$	
14	1.879 ± 0.028	8.32 ± 2.89	128 ± 1	4
18	3.664 ± 0.064	5.88 ± 1.47	250 ± 4	5
22	3.708 ± 0.068	3.16 ± 0.54	253 ± 5	4

[1] Variables: moisture content, w (% w.b.); respiration rate and its standard error, calculated from a pooled data set, $\left(v_{CO_2} \pm SE_{v_{CO_2}}\right)_p$ [mg CO_2 (kg d^{-1})]; time to reach 0.5% DML and its standard deviation, calculated with the lag period (d); DML rate and its standard error, calculated from a pooled data set, $v_{DML} \pm SE_{v_{DML}}$ (10^{-3} % d^{-1}); number of replications, r.

[2] Respiration measurements were conducted using a dynamic grain respiration measurement system (D-GRMS) outfitted with a CO_2 absorbent (Sodasorb®).

[3] Estimates include lag period. Excluding the lag period, $t_{0.5} \pm \sigma_{t_{0.5}}$ estimates are 4.32 ± 1.02, 2.38 ± 0.45, and 2.39 ± 0.55 d, with $t_{start} = t_{0.05}\ DML$; and 4.04 ± 0.88, 2.14 ± 0.46, 2.14 ± 0.41, with $t_{start} = t_{0.10}\ DML$; for 14, 18, and 22% moisture content soybeans, respectively.

4.4. Minimum number of replications

In general, the number of treatment combinations (j) increased, the number of replications required decreased proportionally. As the value of j becomes smaller, it is more difficult to detect a significant difference between treatment combinations. Therefore, one can save labor by increasing treatment combinations rather than the number of replications to detect a difference δ. Table 4.6 illustrates this idea for a factorial experiment with three factors. For example, for a 2 × 1 × 1 factorial treatment combinations (j), 25 replications are necessary. On the other hand, for a 3 × 3 × 3 combination, only one replication is needed. However, this strategy may only be used if $\left(\sigma_{v_{DML}}\right)_p$ remains the same, as additional replications and respiration tests are completed.

Table 4.6. Minimum number of replications for experiments with a minimum significant difference of 0.032 d^{-1}. Variables are defined in the footnotes.

No. of levels in a factorial experiment			j^1	df	t_1	t_2	k^2	r_{min}
Factor 1	Factor 2	Factor 3						
2	1	1	2 × 1 × 1 = 2	1	12.70	1.37	4	25
3	1	1	3	2	4.30	1.06	4	4
2	2	1	4	3	3.18	0.97	4	3
3	2	1	6	5	2.57	0.92	3	2
2	2	2	8	7	2.36	0.89	3	2
3	3	1	9	8	2.31	0.89	3	2
3	2	2	12	11	2.20	0.87	3	2
3	2	3	18	17	2.11	0.86	3	2
3	3	3	27	130	1.97	0.84	1	1

[1] Variables: number of treatment combinations, j; number of degrees of freedom, df; values from Student's t-distribution tables, t_1 and t_2; number of iterations, k; and minimum number of replications, r_{min}.

[2] Statistical power analysis constants used were δ = 0.032 % d^{-1}, $\alpha = 0.05$, $\beta = 0.8$, and an initial guess, $\hat{r} = 6$.

In this study, using the $\left(\sigma_{v_{DML}}\right)_p$ value of 0.008 % d^{-1} obtained from the five replicated tests with 18% moisture content, a $\delta = 4\left(\sigma_{v_{DML}}\right)_p = 0.032$ % d^{-1} was used to find the number of replications necessary to assess the effects of $3w \times 1T$ on v_{DML} (Table 4.7). The iterative process yielded a minimum of four replications were needed. After four replications each were conducted for 14 and 22% moisture content soybeans, r_{min} was recalculated using their respective $(\sigma_{v_{DML}})_p$ values, which were lower than that for 18%. Hence, resulting r_{min} values were lower and no additional replications were needed.

Table 4.7. Minimum number of replications needed in this study.

Moisture content, w (% w.b.)	Pooled standard deviation, $(\sigma_{v_{DML}})_p$ (% d^{-1})	Significant difference, $\delta = 4(\sigma_{v_{DML}})_p$ (% d^{-1})	Minimum number of replications, r_{min}
14	0.002	0.008	2
18	0.008	0.032	4
22	0.005	0.020	3
		0.066[1]	1

[1] Minimum significant difference obtained from PROC ANOVA output from analyzing dry matter loss rates across moisture content treatments considering balanced sample sizes for all treatments. Fourth replication at 18% moisture content removed as possible outlier.

4.5. Mathematical models of dry matter loss rate

The DML data collected in this study were higher than those reported by Rukunudin (1997) and Sorour and Uchino (2004) (Figure 4.2). Over time, DML data for 14 to 22% moisture content soybeans appeared exponential, likely due to the high storage temperature (35ºC). Similarly, data from 22% moisture content soybeans stored at 26ºC reported by Rukunudin (1997) and those reported for 18 to 26% moisture content soybeans stored at 30°C by Sorour and Uchino (2004) were nonlinear over time, but comparable in magnitude to those observed in this study.

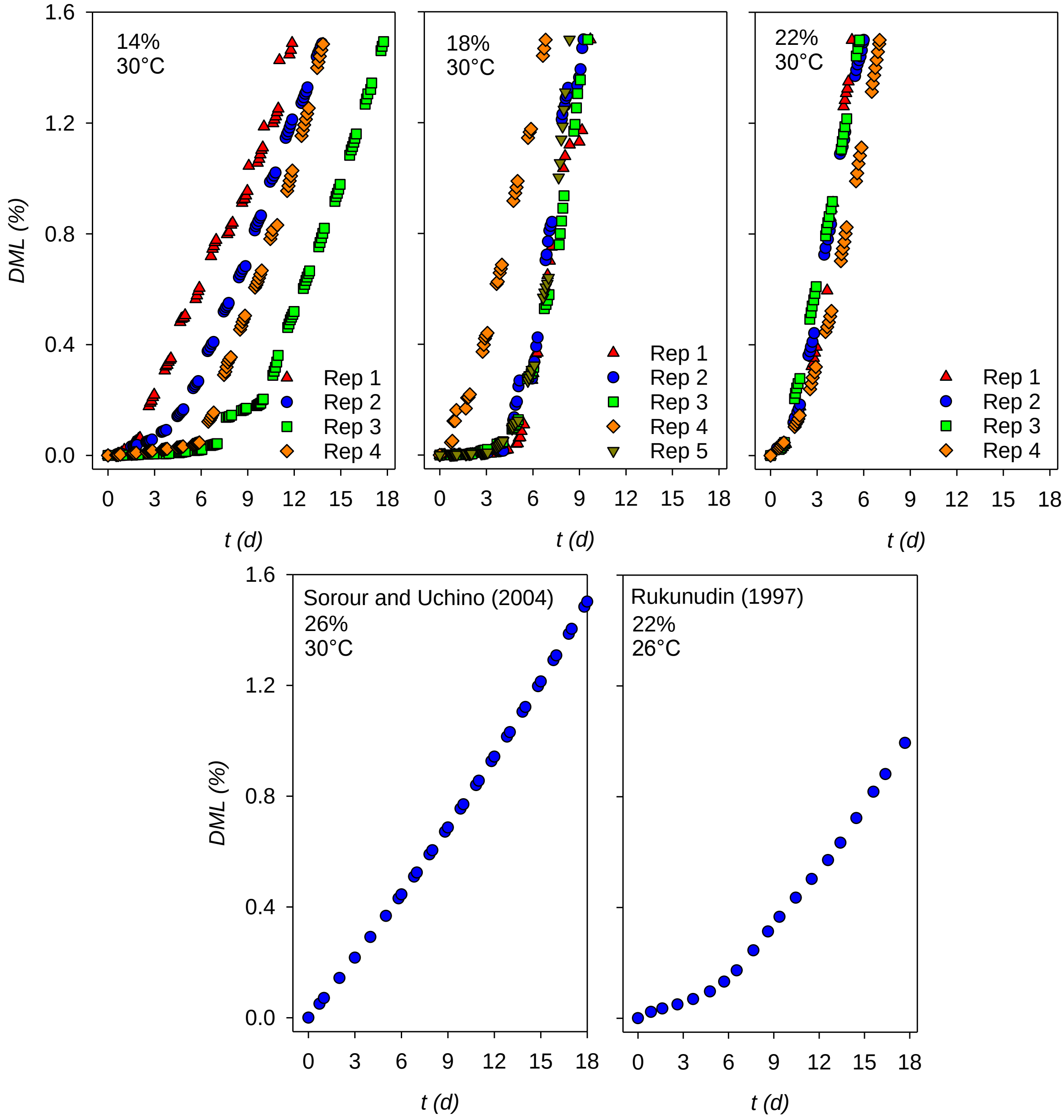

Figure 4.2. Comparison of dry matter loss data of soybeans collected in this study to those reported by Rukunudin (1997) and Sorour and Uchino (2004).

The v_{DML} values observed in this study were higher, but comparable, to those obtained by Sorour and Uchino (2004) (Figure 4.3). Over the range of w tested, v_{DML} appears to reach an asymptote at 22% moisture content, while v_{DML} appears unbounded based on Sorour and

Uchino's (2004) data. From Table 2.1, the following models have been proposed previously to describe w, T and t effects on v_{CO_2}:

Model No. 3 $$v_{CO_2} = \beta_1 \exp(\beta_2 w)$$

Model No. 8 $$\log v_{CO_2} = \beta_1 + \beta_2 T + \beta_3 t + \beta_4 t^2 + \beta_5 w$$

Model No. 9 $$\ln v_{CO_2} = \beta_1 + \beta_2 t + \beta_3 t^2 + \beta_4 w$$

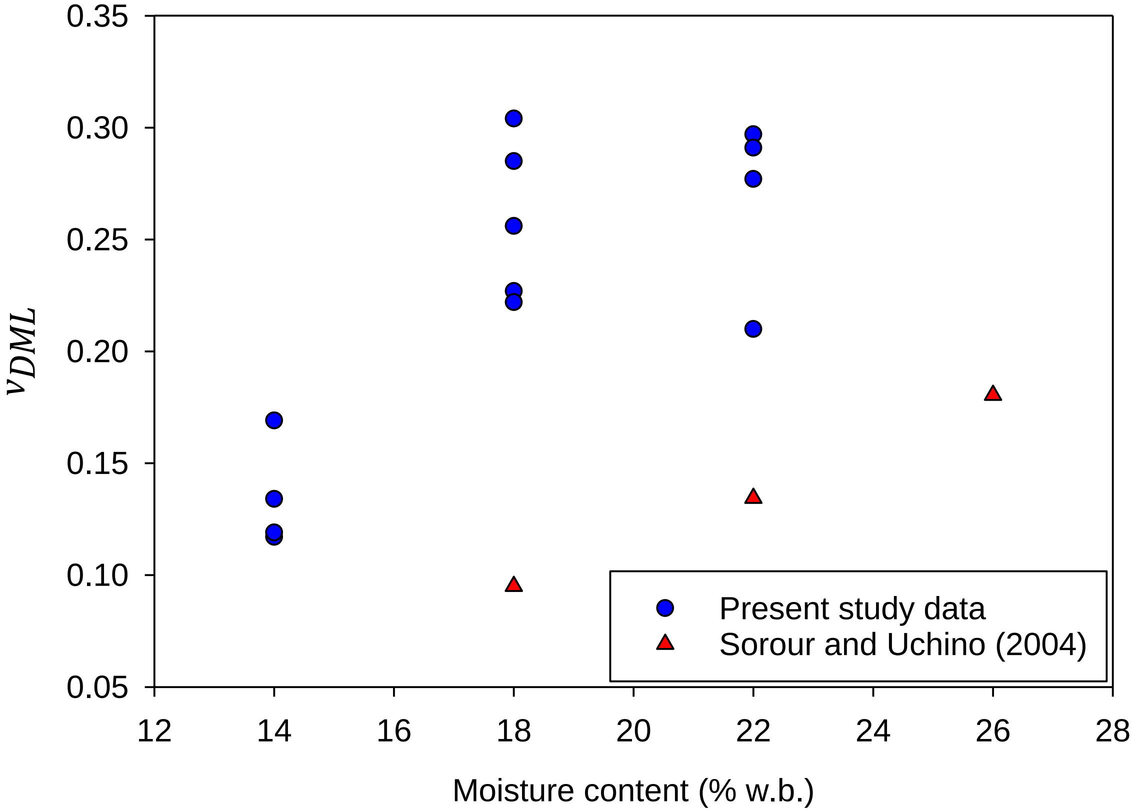

Figure 4.3. Effects of soybean moisture content on dry matter loss rates obtained in this study and Sorour and Uchino's (2004) study.

From Equation 2.2, v_{DML} is a multiple of v_{CO_2}, so v_{DML} can be substituted easily into the above equations and simplified as follows, since T was held constant in this study:

$$v_{DML} = \beta_1 \exp(\beta_2 w) \quad [4.1]$$

$$\log v_{DML} = \beta_1 + \beta_2 w \rightarrow v_{DML} = 10^{\beta_1 + \beta_2 w} \quad [4.2]$$

$$\ln v_{DML} = \beta_1 + \beta_2 w \rightarrow v_{DML} = \exp(\beta_1 + \beta_2 w) = \beta_1 \exp(1 + \beta_2 w) \quad [4.3]$$

Equations 4.1 through 4.3 are very similar and, thus, v_{DML} data were fitted to Equations 4.1 and 4.3 only. Results showed that both exponential models present a good prediction for v_{DML} (R^2 =0.96 and 0.97) in the range of w used in this study.

Table 4.8. Nonlinear models of the effects of soybean moisture content on dry matter loss rates.

Mathematical model: $v_{DML} = \beta_1 \exp(\beta_2 w)^1$							
v_{DML} vs. w	Regression coefficients		MRE	SE_{reg}	F	$\hat{R}^2$	Residual plot
	$\beta_1 \pm \sigma_{\beta_1}$	$\beta_2 \pm \sigma_{\beta_2}$					
Sorour and Uchino (2004)	0.019 ± 0.009	0.090 ± 0.020	0.16	0.02	149.18	0.97	random
This study	0.064 ± 0.024	0.068 ± 0.024	0.15	0.02	149.31	0.96	random

Mathematical model: $v_{DML} = \beta_1 \exp(1 + \beta_3 w)$							
v_{DML} vs. w	Regression coefficients		MRE	SE	F	$\hat{R}^2$	Residual plot
	$\beta_1 \pm \sigma_{\beta_1}$	$\beta_3 \pm \sigma_{\beta_3}$					
Sorour and Uchino (2004)	0.007 ± 0.003	0.090 ± 0.020	0.16	0.02	149.18	0.97	random
This study	0.024 ± 0.009	0.068 ± 0.019	0.15	0.02	149.31	0.96	random

CHAPTER 5. CONCLUSIONS AND FUTURE WORK

A dynamic grain respiration measurement system (D-GRMS) was used to measure grain deterioration rates of 14 to 22% moisture content soybeans stored at 35ºC. Overall, v_{CO2}, v_{DML} and $t_{0.5}$ results reported in this thesis research were higher than those previously reported in the literature because of the method of CO_2 measurement used and soybean sample size. Most of the previous grain respiration studies were conducted using a static grain respiration measurement system (S-GRMS) which tends to predict a lower deterioration rate because of the limited oxygen supply for respiration. Nevertheless, v_{CO2}, v_{DML} and $t_{0.5}$ results were comparable to results from soybean respiration studies that utilized D-GRMS, albeit at lower storage temperatures (Rukunudin, 1997; Mendes et al., 2009). The quantification of the variability of v_{DML}, in the form of $\left(SE_{v_{DML}}\right)_p$ and $\left(\sigma_{v_{DML}}\right)_p$, was presented to show its utility in determining minimum significant difference to be detected in a statistical power analysis so that future grain respiration studies may be conducted in a robust manner. Finally, v_{DML} tended to increase exponentially with increasing w, based on data with 18 to 26% moisture content soybeans from Sorour and Uchino’s (2004) study, but the trend was asymptotic between 18 and 22% moisture content soybeans in this study.

For future work, it is recommended that more grain respiration data are collected for a wider range of w and T that would be typical and useful for soybean producing regions in low-latitude countries. It would also be beneficial to find the correlation between DML and market grade. In this study, $t_{0.5}$ was determined but no data were presented to show its correlation to market grade or quality factors, such as germination or lipid oxidation rates. Finally, the D-GRMS, protocols, and statistical analyses of grain deterioration parameters presented in this study may be used with other cereal or oilseed commodities. For example, all wheat respiration studies described in the literature have been conducted with S-GRMS, which provide important information for hermetic storage systems. However, since wheat is typically stored in bulk, aerated systems, respiration data collected in D-GRMS would be more appropriate towards the development a MAST guidelines for wheat crops.

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APPENDIX A. DYNAMIC GRAIN RESPIRATION MEASUREMENT SYSTEM (D-GRMS)

A.1. D-GRMS Instrumentation

A.1.1. Fine adjustment of volumetric flow rate

Fine adjustment of the voltage delivered to the mass flow controller (Figure 3.1., Item 3) was adjusted using a digital potentiometer driven by a microcontroller (ATmega2560, Arduino, Ivrea, Italy) and the flow rate was displayed using an LCD digital display (Figure A.1).

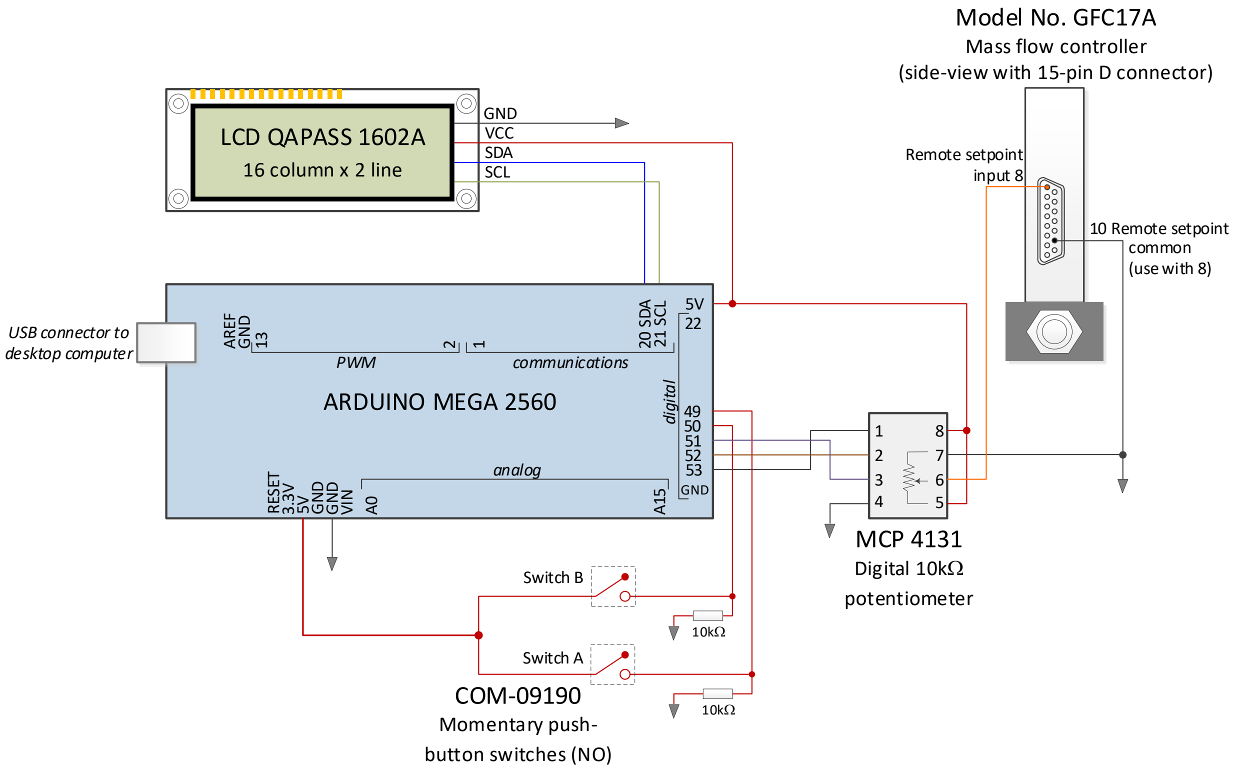

Figure A.1. Diagram of the circuit for fine adjustment of volumetric flow rate. All components share a common ground.

The mass flow control program (listed below) utilized the open-source software by Arduino (IDE Version 1.5.5r2, Arduino, Ivrea, Italy) and 3 supporting libraries: SPI, LiquidCrystal_I2C, Wire. DigitalRead command was used to collect the information from the momentary switches connected to the Pin 49 and 50 respectively, which were used to change the internal resistance of the digital potentiometer, resulting in a change of the voltage across this element. The voltage was then supplied to the remote control circuitry of the MFC which translated to flow rate level. The flow was kept constant with constant input voltage. The LCD connected to the SCL and SDA ports displayed the flow and the voltage in the MFC.

Mass flow control program:

```
#include <SPI.h>
#include <Wire.h>
#include <LiquidCrystal_I2C.h>
// Set the LCD address to 0x27 for a 16 chars and 2 line display
LiquidCrystal_I2C lcd(0x27, 16, 2); //LCD I2C address
byte address = 0x00;
int CS= 10; //chip select pin connection
const int buttonPin_A = 50;    // pin assignment of switch A
const int buttonPin_B = 49;    // pin assignment of switch B
const int ledPin =  13;        // digital port for LED pin
int buttonState_A = 0;         // variable for reading switch A status
int buttonState_B = 0;         // variable for reading switch A status
int count=0;                   //start counting from zero

//creating characters for LCD
uint8_t bar0_13[8] =  {B00000 , B00000 , B00000 , B00000 , B00000 , B00000 ,
B00000 ,};
uint8_t bar1_13[8] =  {B00000 , B00000 , B00000 , B00000 , B00000 , B00000 ,
B11111 ,};
uint8_t bar2_13[8] =  {B00000 , B00000 , B00000 , B00000 , B00000 , B11111 ,
B11111 ,};
uint8_t bar3_13[8] =  {B00000 , B00000 , B00000 , B00000 , B11111 , B11111 ,
B11111 ,};
uint8_t bar4_13[8] =  {B00000 , B00000 , B00000 , B11111 , B11111 , B11111 ,
B11111 ,};
uint8_t bar5_13[8] =  {B00000 , B00000 , B11111 , B11111 , B11111 , B11111 ,
B11111 ,};
uint8_t bar6_13[8] =  {B00000 , B11111 , B11111 , B11111 , B11111 , B11111 ,
B11111 ,};
uint8_t bar7_13[8] =  {B11111 , B11111 , B11111 , B11111 , B11111 , B11111 ,
B11111 ,};

uint8_t arrowup[8] =  {B00100 , B01110 , B10101 , B00100 , B00100 , B00100 ,
B00100 ,};
uint8_t arrowdown[8] = {B00100 , B00100 , B00100 , B00100 , B10101 , B01110 ,
B00100 ,};

//setup loop, runs once
void setup()
  {

  Serial.begin(9600);
```

```
  // initialize the LED pin as an output:
  pinMode(ledPin, OUTPUT);
  // initialize the pushbutton switch pin as an input:
  pinMode(buttonPin_A, INPUT);//read information from switch A as high
                        or low voltage.
  pinMode(buttonPin_B, INPUT);// read information from switch B as
                              high or low voltage.
  pinMode (CS, OUTPUT); // defining the output pin on the digital
                              potentiometer
  SPI.begin(); //initializing the communication protocol with digital
                 potentiometer
  lcd.begin(); //starts LCD display

//creating characters for
  lcd.createChar(0,bar0_13);
  lcd.createChar(1,bar1_13);
  lcd.createChar(2,bar2_13);
  lcd.createChar(3,bar3_13);
  lcd.createChar(4,bar4_13);
  lcd.createChar(5,bar5_13);
  lcd.createChar(6,bar6_13);
  lcd.createChar(7,bar7_13);
  lcd.home();

//starting main loop
}
void loop()
  {
  //delay(1000);
  // read the state of the pushbutton value:
  buttonState_A = digitalRead(buttonPin_A); //read switch A
  buttonState_B = digitalRead(buttonPin_B); //read switch B
Serial.print("  Count: ");Serial.println(count);

  if (buttonState_A == HIGH) //when switch A status is HIGH, increment
                              one count and increase the voltage that
                              leads to increase of 0.01 L/m.
    {
    Serial.println(" ");
    if(count<128)
      {
      lcd.createChar(0, arrowup);
      lcd.home();
      lcd.setCursor(11, 0);
      lcd.write(0);
      count++;
      delay(200);
      lcd.setCursor(11, 0);
      lcd.print(" ");
      }
    digitalWrite(ledPin, HIGH);
    }
  else
    if (buttonState_B == HIGH) //when switch B status is HIGH, increment
                                    one count and increase the voltage that
                                    leads to increase of 0.01 L/m.
```

```
      {
      Serial.println(" ");
    if(count>0)
      {
      lcd.createChar(0, arrowdown);
      lcd.home();
      lcd.setCursor(11, 0);
      lcd.write(0);
      count--;
      delay(200);
      lcd.setCursor(11, 0);
      lcd.print(" ");
      }
      // turn LED on:
      digitalWrite(ledPin, HIGH);
      lcd.clear();
      }
  else
    {
    Serial.println(0); //in case of error, no voltage, the screen will be
turned off
    // turn LED off:
    digitalWrite(ledPin, LOW);
    }
    if(count>=128)
      {
      lcd.backlight();
      lcd.setCursor(13, 0);
      lcd.print("MAX ");
      //delay(200);
      //lcd.clear();
      }
    else
      {
      if(count<=0)
        {
        lcd.backlight();
        lcd.setCursor(12, 0);
        lcd.print(" MIN ");
        //delay(200);
        //lcd.clear();
        }
      if(count>0 && count <128)
        {
        lcd.backlight();
        lcd.setCursor(12, 0);
        lcd.print("    ");
        lcd.setCursor(12, 0);
        //lcd.write(int((count-1)*(7.0/128)));

      if(round(7*(count+1)/128)<1)
          {
          lcd.setCursor(12, 0);
          lcd.print(" ");
          }
        else
          {
```

```
          lcd.write(round(7*(count+1)/128));
          }

        Serial.print("%: ");
        Serial.print(round(7*(count+1)/128));

        lcd.setCursor(13, 0);
        lcd.print(((count*100)/128));
        lcd.setCursor(15, 0);
        lcd.print("%");
        /*lcd.write(int(count*(7.0/128)));
        lcd.setCursor(14, 0);
        lcd.write(int(count*(7.0/128)));
        lcd.setCursor(15, 0);
        lcd.write(int(count*(7.0/128)));*/
        }
      }
    digitalPotWrite(count);
    lcd.backlight();
    lcd.setCursor(0, 0);
    lcd.print("PWR: ");
    lcd.print(count*(5.0/128));
    Serial.print("PWR: ");Serial.print((count*(5.0/128))); //prints the flow
                                                                      value
    lcd.setCursor(0, 1);
    lcd.print("Flow Range: ");
    lcd.print((count*(5.0/128))/2.5);
    Serial.print("  Flow Range: ");Serial.print((count*(5.0/128))/2.5);
    //delay(100);
    //lcd.clear();
  }

int digitalPotWrite(int value)
  {
  digitalWrite(CS, LOW); //status of digital potentiometer low
  SPI.transfer(address); //SPI communication
  SPI.transfer(value);
  digitalWrite(CS, HIGH); //status of digital potentiometer high
  }
```

A.1.2. Temperature, relative humidity and CO_2 monitoring

T, ϕ, and C_{CO_2} were monitored immediately after humidification and at the exhaust (Figure 3.1). Monitoring after humidification ensured that the airstream entering the *RC* was at $T = 35 \pm 2$ºC, $\phi = \phi_{set} \pm 5$ %RH, and $C_{CO_2} \leq 200$ ppm, where the setpoint $\phi_{set} = 79$, 89, and 92% for $w = 14$, 18, and 22% moisture soybeans, respecitvely. At the exhaust, it was critical that $\phi = 0$ %RH and $C_{CO_2} = 0$ ppm, to ensure all moisture and CO_2 were absorbed in the *RC* dehumidifiers and *RC* CO$_2$ scrubbers, respectively. T, ϕ, and C_{CO_2} measurements were logged every 2 min onto a desktop computer using an ATmega microcontroller (Figure A.2).

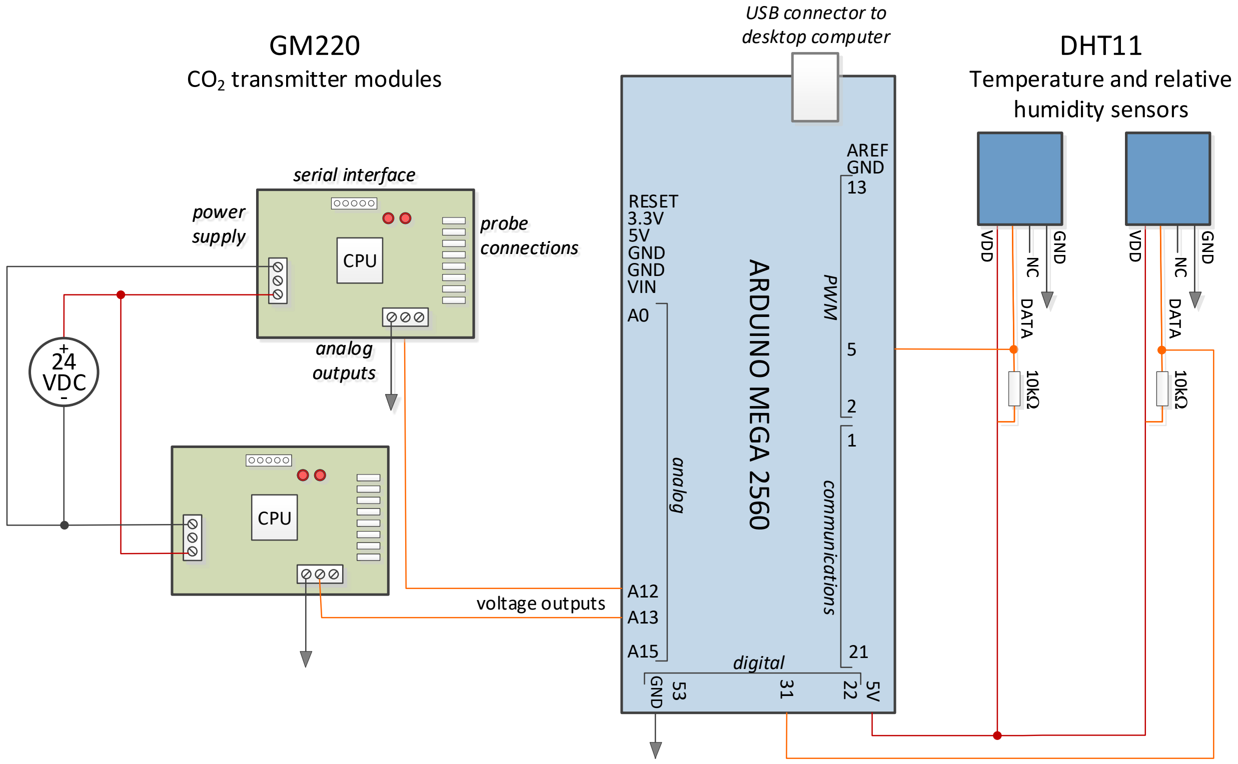

Figure A.2. Circuit diagram of the power supply, sensors, and data acquisition system for monitoring temperature, relative humidity, and CO$_2$ concentration in D-GRMS.

The data acquisition program (listed below) utilized the open source software for Arduino and 3 supporting libraries: SPI, DHT, Wire. The Wire library allowed communication between I2C devices. The DHT library provided communication between the humidity and temperature sensor and the microcontroller. Inside the void loop, initially the program checked if the sensors were working and, in case a problem was identified, an error message was printed. From pins 5 and 31 on the microcontroller, DHT library translated information into humidity (%) and temperature (°C). From pins 12 and 13 on the microcontroller, the command readAnalog provided the CO_2 concentration reading from the Vaisala sensors. The program printed the readings from all the sensors on a serial monitor screen.

Data acquisition program code:

```
#include <SPI.h>
#include <DHT.h>
#include <Wire.h>

//defining DHT 1
dht DHT1;
#define DHT11_PIN 5
//defining DHT 2
dht DHT2;
#define DHT11_2_PIN 31

//starting serial

void setup()
{
  Serial.begin(9600);
//defining the order of values displayed

  Serial.print("Set \tHumidity_1 (%)\tTemperature_1 (C) CO2_1 (ppm)");
  Serial.print("||\t");
  Serial.println("Set \tHumidity_2 (%) \tTemperature_2 (C) \tCO2_2 (ppm)");
}

//mains loop
void loop()
{
// READ DATA
  int chk1 = DHT1.read11(DHT11_PIN); //check if first Set of sensors is
working
  int chk2 = DHT2.read11(DHT11_2_PIN);//check if second Set of sensors is
working
//DHT1 check and reading
  switch (chk1)
    {
    case DHTLIB_OK:
            //Serial.print("OK,\t");
            break;
    case DHTLIB_ERROR_CHECKSUM:
            Serial.print("Checksum error,\t");
            break;
    case DHTLIB_ERROR_TIMEOUT:
            Serial.print("Time out error,\t");
            break;
    case DHTLIB_ERROR_CONNECT:
        Serial.print("Connect error,\t");
        break;
    case DHTLIB_ERROR_ACK_L:
        Serial.print("Ack Low error,\t");
        break;
```

```
    case DHTLIB_ERROR_ACK_H:
        Serial.print("Ack High error,\t");
        break;
    default:
                Serial.print("Unknown error,\t");
                break;
    }
//DHT 2 check and reading
 switch (chk2)
  {
    case DHTLIB_OK:
                //Serial.print("OK,\t");
                break;
    case DHTLIB_ERROR_CHECKSUM:
                Serial.print("Checksum error,\t");
                break;
    case DHTLIB_ERROR_TIMEOUT:
                Serial.print("Time out error,\t");
                break;
    case DHTLIB_ERROR_CONNECT:
        Serial.print("Connect error,\t");
        break;
    case DHTLIB_ERROR_ACK_L:
        Serial.print("Ack Low error,\t");
        break;
    case DHTLIB_ERROR_ACK_H:
        Serial.print("Ack High error,\t");
        break;
    default:
                Serial.print("Unknown error,\t");
                break;
    }
//Vaisala 1 reading
  int VAI1 = 0;
  #define VAI1pin 12
  VAI1 = map(analogRead(VAI1pin),0,1023,0,12200);[31]
//Vaisala 2 reading
 int VAI2 = 0;
  #define VAI2pin 13
  VAI2 = map(analogRead(VAI2pin),0,1023,0,12200);
// DISPLAY DATA
  Serial.print("SET1\t");
  Serial.print(DHT1.humidity, 1);
  Serial.print("\t");
  Serial.print(DHT1.temperature, 1);
  Serial.print("\t");
  Serial.print(VAI1);
  Serial.print("\t");
  Serial.print("||\t");
    Serial.print("SET2\t");
  Serial.print(DHT2.humidity, 1);
  Serial.print("\t");
  Serial.print(DHT2.temperature, 1);
  Serial.print("\t");
  Serial.println(VAI2);
  delay(2000);
}
```

A.2. Standard Operating Procedures (SOPs)

University of Illinois at Urbana-Champaign	**Title: Setting up air supply scrubber**
Effective date: 01 October 2017	Document ID: D-GRMS-001
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for setting up the air supply scrubber for the dynamic grain respiration measurement system (D-GRMS) located in Burnsides Research Laboratory.

2.0. SCOPE

This SOP describes how to prepare the air supply scrubber with Sodasorb® and clean and store the unit after each use.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. Drierite Laboratory Air and Gas Drying Unit (Product No. 26800, W.A. Hammond Drierite Co., Ltd., Xenia, OH, USA) - The unit includes 1 molded polycarbonate column, 1 polycarbonate cap fitting (screw-top) with an o-ring gasket, 2 desiccant supports or perforated metal disks, 1 coil spring, and 1 wrench.
4.2.Vincon Flexible PVC tubing – 6.35 mm (0.25 in) ID (Part No. ABH02017, Saint-Gobain, Akron, OH, USA) or similar material with equivalent resistance.
4.3.Connectors – In-line hose barbs, non-spill coupling insert (Product No. 60721, U.S. Plastics, Lima, OH, USA) or similar quick-disconnect coupler.
4.4. CO_2 absorbent – Sodasorb® (Product No. SODA-SORB-HP, Amron International, Vista, CA, USA) or similar material with equivalent absorbing capacity and particle size (e.g., granular).

5.0. PROCEDURES

5.1. Start with a clean and dry column.
5.2. Check that the tubing attached to the column is secure and, on each end, a quick-disconnect coupler insert is securely attached.
5.3. Place the first perforated metal disk in the bottom of the column to create a plenum.
5.4. Weigh 550 g Sodasorb®. Document the exact mass of Sodasorb® used in datasheet.
5.5. Carefully pour the Sodasorb® into the column.
5.6. Place the second perforated metal disk on top of the Sodasorb®, followed by the coil spring.
5.7. Cover the filled column using the screw-top cap. Tighten cap using the supplied wrench.

5.8. Weigh the filled column three times, rotating the column 120º in between measurements. Record each weight and their averages.

5.9. Place the filled column in between the mass flow controller and the first glycerol-water reservoir of D-GRMS. Connect tubing using quick-disconnects couple

6.0. PROCEDURE: CLEANING AND REPORTING

6.1. After a grain respiration test, repeat Step 5.8.

6.2. Dispose of spent Sodasorb® following chemical disposal guidelines by the UIUC Division of Research Safety.

6.3. Wash the column, metal disks, coil spring, and cap with warm soapy water and let dry at room temperature.

6.4. Check for scratches, cracks, and other defects in all components that would cause gas to leak in/out of the column.

6.5. Store clean and dry units in a cabinet at room temperature.

6.6. Record any issues with preparing, cleaning, and inspecting the column. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. When cracks or defects are found in the columns, notify the supervisor immediately. Do not use damaged columns in future grain respiration tests.

7.2. The supervisor will take further corrective actions which may include repairing or replacing damaged materials.

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.

8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Preparing glycerol-water solutions**
Effective date: 01 October 2017	Document ID: D-GRMS-002
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for preparing glycerol-water solutions for the dynamic grain respiration measurement system (D-GRMS) located in Burnsides Research Laboratory.

2.0. SCOPE

This SOP describes how to prepare glycerol-water solutions used to control the humidity of the airstream during a grain respiration test.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

- 4.1. Plastic vacuum bottles – Heavy duty HDPE bottles with 83 mm cap, 2 L capacity (Product No. D1069702 Saint Gobain Performance, Akron, OH, USA) or similar vacuum bottle with the same volume capacity.
- 4.2. Digital precision balance or scale – Ranges from 0 to 3100 g with 0.01 g resolution (Model iBalance i3100, MyWeigh, Phoenix, AZ, USA) or similar device with the same range and resolution.
- 4.3. Stirring hot plate – Temperature range 30 to 540ºC and magnetic stirrer speed 60 to 1200 rpm (Model No. G33500, Fisher Scientific, Hampton, NH, USA) or similar device that could heat solution to 50ºC and stir at 100-200 rpm.
- 4.4. Magnetic stir bar and remover tool – Octagonal with flat surfaces, 12.7 mm (0.5 in) long (Product No. S717737, Fisher Scientific, Hampton, NH, USA) or similar device.
- 4.5. Parafilm – 10.2 cm (4 in) wide (Product No. S37440, Fisher Scientific, Hampton, NH, USA) or similar material.
- 4.6. Glass beakers –two 3000 ml beaker
- 4.7. Glass bottle – 4.4 L capacity.
- 4.8. Glycerol – Certified ACS grade (Fisher Scientific, Hampton, NH, USA).
- 4.9. Deionized water

5.0. PROCEDURES

- 5.1. Use the table and equations in D-GRMS-002a to calculate individual masses of glycerol and water needed in a 2 L mixture to deliver desired relative humidity at the temperature of the grain respiration test.
- 5.2. Weigh the amount of glycerol and water, according to results from Step 5.1, into separate 3000 ml beakers.
- 5.3. Carefully pour the glycerol into the 4.4 L glass bottle.

5.4. Rinse the glycerol beaker with a portion of the deionized water weighed out in Step 5.2. Transfer rinse solution to the vacuum bottle. Repeat 2-3 more times to transfer all of the glycerol into vacuum bottle.
5.5. Pour any remaining water from Step 5.2 into the 4.4 L glass bottle.
5.6. Gently drop the magnetic stir bar into the mixture.
5.7. Seal the bottle with parafilm to prevent water loss due to evaporation.
5.8. Place mixture on the stirring hot plate. Carefully set the temperature to 50°C and stir speed to 100-200 rpm.
5.9. Let solution mix and warm up for 30 min.
5.10. Remove mixture from hot plate and let it cool to room temperature.
5.11. Pour mixture in equal parts into two vacuum bottles.
5.12. Connect the bottles in series, submerge them in water bath “A”, and let them reach the set temperature prior to starting a grain respiration test.
5.13. Check the resulting relative humidity and adjust by adding glycerol in small increments (100-1000 μl at a time) to decrease humidity or by adding water in small increments (100-1000 μl at a time) to increase humidity.

6.0. PROCEDURE: FINAL STEPS AND REPORTING

6.1. Record the date when a fresh glycerol-water solution was made.
6.2. Glycerol-water solutions may only be re-used once. Store solutions for re-use at 4°C.
6.3. Prior to re-use, check for molds or off-odors and test the resulting relative humidity.
6.4. A solution that has been used for two grain respiration tests should be discarded by pouring it down the drain with copious amounts of water.
6.5. Vacuum bottles and beakers should be washed with warm soapy water and let dry at room temperature.
6.6. Check for scratches, cracks and other defects in vacuum bottles that could cause gas to leak in/out of the bottle prior to each use.
6.7. Record any issues with preparing and re-using solutions. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. If the resulting relative humidity is below the desired humidity, calculate the amount of water needed to dilute the solution using information in D-GRMS-004a. Adjust accordingly by adding ½ the amount of water needed to each vacuum bottle.
7.2. Discard any glycerol-water solution that shows signs of molding or emits off-odors.
7.3. When cracks or defects are found in the bottles, notify the supervisor immediately. Do not use damaged bottles in future respiration tests.
7.4. The supervisor will take further corrective actions which may include repairing or replacing damaged materials.

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.
8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Table and equations for making glycerol-water solutions**
Effective date: 01 October 2017	Document ID: D-GRMS-002a
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP provides a table and equations used to calculate the relative proportions of glycerol and water to make the mixture used for humidification in the dynamic grain respiration measurement system (D-GRMS) located in Burnsides Research Laboratory.

2.0. SCOPE

This SOP provides glycerol-water concentrations for soybean respiration tests involving 12 to 22% moisture content soybeans to be stored at 25 to 45°C.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. Calculator or spreadsheet

4.2. Laboratory notebook and pen to record calculations

5.0. PROCEDURES

5.1. The following equations should be used to calculate the specific gravity (SG) and concentration ($C_{C_3H_8O_3}$) of glycerol-water solutions:

$SG = (-0.189\phi_{set} = 19.9)^{0.0806}$, where ϕ_{set} is the setpoint relative humidity in D-GRMS and is equal to ϕ_e of the soybeans.

$C_{C_3H_8O_3} = 383(SG - 1)$ (% mass glycerol concentration)

5.2. The total mass (M_{soln}) and volume (V_{soln}) of the solution are dependent on the mass and/or volume of the glycerol ($V_{C_3H_8O_3}$) and water (V_{H_2O}) to be mixed:

$M_{soln} = V_{H_2O}\left(100\rho_{H_2O}\right)\left(100 - C_{C_3H_8O_3}\right)$, where the density of water is ρ_{H_2O} = 1 g mL^{-1}.

$V_{C_3H_8O_3} = \frac{C_{C_3H_8O_3}M_{soln}}{100\rho_{C_3H_8O_3}}$, where $\rho_{C_3H_8O_3}$= 1.262 g mL^{-1}.

5.3. The table on the following page has been developed to ease calculations.

5.4.

6.0. PROCEDURES: FINAL STEPS AND REPORTING

6.1. Record calculations with Step 6.1 in D-GRMS-002.

7.0. CORRECTIVE ACTION

7.1. Record calculations with Step 7.1 in D-GRMS-002.

w (% w.b.)	T (°C)	ϕ_e (%)	SG	$C_{C_3H_8O_3}$ (%)	V_{H_2O} (mL)	M_{soln} (g)	$V_{C_3H_8O_3}$ (mL)	V_{soln} (mL)
12	25	65	1.18	68.09	1301	4076.85	2199.56	3500.56
	30	66	1.18	67.18	1335	4067.05	2164.86	3499.86
	35	68	1.17	65.28	1403	4041.32	2090.59	3493.59
14	25	77	1.14	55.41	1767	3963.13	1740.20	3507.20
	30	78	1.14	54.14	1810	3947.16	1693.47	3503.47
	35	79	1.13	52.83	1855	3932.66	1646.33	3501.33
18	25	87	1.11	40.27	2280	3817.21	1218.07	3498.07
	30	88	1.10	38.36	2345	3804.14	1156.22	3501.22
	35	89	1.09	36.34	2410	3785.64	1090.05	3500.03
22	25	91	1.08	31.93	2550	3746.40	948.02	3498.02
	30	92	1.08	29.52	2630	3731.32	872.68	3502.68
	35	93	1.07	21.10	2750	3763.16	802.82	3494.82

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 1 October 2017.

8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Setting up RC CO_2 dehumidifiers**
Effective date: 01 October 2017	Document ID: D-GRMS-003
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for setting up the respiration chamber (RC) dehumidifiers for the dynamic grain respiration measurement system (D-GRMS) located in Burnsides Research Laboratory.

2.0. SCOPE

This SOP describes how to prepare two RC dehumidifiers, clean, and store the unit after each use.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. Drierite® Laboratory Air and Gas Drying Units (Product No. 26800, W.A. Hammond Drierite Co., Ltd., Xenia, OH, USA) – Two units are needed. Each unit includes 1 molded polycarbonate column, 1 polycarbonate cap fitting (screw-top) with an o-ring gasket, 2 desiccant supports or perforated metal disks, 1 coil spring, and 1 wrench.
4.2.Vincon Flexible PVC tubing – 6.35 mm (0.25 in) ID (Part No. ABH02017, Saint-Gobain, Akron, OH, USA) or similar material with equivalent resistance.
4.3. Connectors – In-line hose barbs, non-spill coupling body and insert (Product Nos. 60719 and 60721, U.S. Plastics, Lima, OH, USA) or similar quick-disconnect coupler.
4.4. Valves – Three-way PVC ball valves with 6.35mm hosebarb end connectors (Product No. 22264, U.S. Plastics, Lima, OH, USA).
4.5.Desiccant – Drierite®, 8 mesh, with indicator (Product No. 26802, W.A. Hammond Drierite Co., Ltd., Xenia, OH, USA) or similar material.

5.0. PROCEDURES

5.1. Start with a clean and dry column.
5.2. Check that the tubing attached to the column are secure and, on each end, a quick-disconnect coupler body is securely attached.
5.3. Place the first perforated metal disk in the bottom of the column to create a plenum.
5.4.Weigh 500 g desiccant and record measurement in datasheet.
5.5. Carefully pour the desiccant into the column.
5.6.Place the second perforated metal disk on top of the desiccant, followed by the coil spring.

5.7. Cover the filled column using the screw-top cap. Tighten cap using the supplied wrench.

5.8. Weigh the filled column three times, rotating the column 120º in between measurements. Record each weight and their averages.

5.9. Repeat Steps 5.1 to 5.9 to make a second RC scrubber column.

5.10. Install the two RC dehumidifiers in parallel in the D-GRMS immediately following the RC using two valves and set up the valves so that airstream passes through the first RC dehumidifier (designated as "A"; the second scrubber is designated as "B").

6.0. PROCEDURE: CLEANING AND REPORTING

6.1. After a grain respiration test, repeat Step 5.8.

6.2. Regenerate spent desiccant at 210ºC for 1 h.

6.3. Wash the columns, metal disks, coil springs, caps, and separators with warm soapy water. Be sure to scrub any traces of mold. Let dry at room temperature.

6.4. Check for scratches, cracks, and other defects in all components that would cause gas to leak in/out of the column.

6.5. Store clean and dry units in a cabinet at room temperature.

6.6. Record any issues with preparing, cleaning, and inspecting the column. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. When cracks or defects are found in the columns, Sodasorb®, and desiccant, notify the supervisor immediately. Do not use damaged columns, Sodasorb®, or desiccant future grain respiration tests.

7.2. The supervisor will take further corrective actions which may include repairing or replacing damaged materials.

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.

8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Setting up RC CO_2 scrubbers**
Effective date: 01 October 2017	Document ID: D-GRMS-004
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for setting up the RC CO_2 scrubbers for the dynamic grain respiration measurement system (D-GRMS) located in Burnsides Research Laboratory.

2.0. SCOPE

This SOP describes how to prepare two RC CO_2 scrubbers, clean, and store the units after each use.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. Drierite® Laboratory Air and Gas Drying Units (Product No. 26800, W.A. Hammond Drierite Co., Ltd., Xenia, OH, USA) – Two units are needed. Each unit includes 1 molded polycarbonate column, 1 polycarbonate cap fitting (screw-top) with an o-ring gasket, 2 desiccant supports or perforated metal disks, 1 coil spring, and 1 wrench.

4.2. Separators – Each separator is custom-made of a plastic cylinder (2.5 cm ID x 1.5 height) with a plastic perforated disk (40% open, 0.3 cm dia. holes) on each end. One separator per column is needed for a total of two separators.

4.3. Oven - Convection oven (Product No. VWR-U50, VWR, Radnor, PA, USA), set at 103°C, or similar equipment.

8.3. Vincon Flexible PVC tubing – 6.35 mm (0.25 in) ID (Part No. ABH02017, Saint-Gobain, Akron, OH, USA) or similar material with equivalent resistance.

4.4. Connectors – In-line hose barbs, non-spill coupling body and insert (Product Nos. 60719 and 60721, U.S. Plastics, Lima, OH, USA) or similar quick-disconnect coupler.

4.5. Valves – Three-way PVC ball valves with 6.35mm hosebarb end connectors (Product No. 22264, U.S. Plastics, Lima, OH, USA).

4.6. CO_2 absorbent – Sodasorb® (Product No. SODA-SORB-HP, Amron International, Vista, CA, USA) or similar material with equivalent absorbing capacity and particle size (e.g., granular).

4.7. Desiccant – Drierite®, 8 mesh, with indicator (Product No. 26802, W.A. Hammond Drierite Co., Ltd., Xenia, OH, USA) or similar material.

5.0. PROCEDURES

5.1. Start with a clean and dry column.
5.2. Check that the tubing attached to the column are secure and, on each end, a quick-disconnect coupler body is securely attached.
5.3. Place the first perforated metal disk in the bottom of the column to create a plenum.
5.4. Weigh 150 g Sodasorb® and record measurement in datasheet.
5.5. Weigh 300 g desiccant and record measurement in datasheet.
5.6. Carefully pour the Sodasorb® into the column.
5.7. Place one separator on top of the Sodasorb®.
5.8. Fill the remaining height of the column with the desiccant.
5.9. Place the second perforated disk on top of the desiccant, followed by the coil spring.
5.10. Cover the filled column using the screw-top cap. Tighten cap using a wrench.
5.11. Weigh the filled column three times, rotating the column 120º in between measurements. Record each weight and their averages.
5.12. Repeat Steps 5.1 to 5.11 to make a second RC scrubber column.
5.13. Install the two RC scrubbers in parallel in the D-GRMS using two valves and set up the valves so that airstream passes through the first RC scrubber (designated as "A"; the second scrubber is designated as "B").

6.0. PROCEDURE: CLEANING AND REPORTING

6.1. After a grain respiration test, repeat Step 5.11.
6.2. Regenerate spent desiccant at 210ºC for 1 h.
6.3.Dispose of spent Sodasorb® following chemical disposal guidelines by the UIUC Division of Research Safety.
6.4. Wash the columns, metal disks, coil springs, caps, and separators with warm soapy water. Be sure to scrub any traces of mold. Let dry at room temperature.
6.5. Check for scratches, cracks, and other defects in all components that would cause gas to leak in/out of the column.
6.6. Store clean and dry units in a cabinet at room temperature.
6.7. Record any issues with preparing, cleaning, and inspecting the column. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. When cracks or defects are found in the columns, Sodasorb®, and desiccant, notify the supervisor immediately. Do not use damaged columns, Sodasorb®, or desiccant future grain respiration tests.
7.2. The supervisor will take further corrective actions which may include repairing or replacing damaged materials.

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.
8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Preparing soybeans for a grain respiration test**
Effective date: 01 October 2017	Document ID: D-GRMS-005
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for preparing soybeans for a grain respiration test.

2.0. SCOPE

This SOP describes how to clean and re-wet soybeans for a grain respiration test.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components, preparing samples, and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. Aluminum grain sieve – 33 cm ID with slotted screens of 0.39 cm x 1.9 cm to remove foreign materials and splits (Grainman 0.39 cm x 1.9 cm, Miami, FL, USA).
4.2. Handheld digital moisture meter (Model No. SW16060 Moisture Check, John Deere Co., City, State, USA) or similar device.
4.3. Digital precision balance or scale – Ranges from 0 to 3100 g with 0.01 g resolution (Model iBalance i3100, MyWeigh, Phoenix, AZ, USA) or similar device with the same range and resolution.
4.4. Roller mixer (Model No. Scilogex MX-T6-S, Rocky Hill, CT, USA) or similar device.
4.5. Plastic bottle – Nalgene 2 L capacity bottle (Model No. 2202-0005, U.S. Plastics, Lima Ohio, OH, USA) and wrapped with 2-3 rubber bands or similar device.
4.6. Glass beaker, 100 ml capacity
4.7. Plastic funnel – 32 oz. Polyethylene Funnel 6-3/8" Dia. x 7-1/8"H, (Model No. 832WN, U.S. Plastics, Lima Ohio, OH, USA).
4.8. Aluminum tray – 92x62x3 cm
4.9. Timer – Digital compact timer (Product No. 5806, Taylor, Oak Brooks, IL).
4.10. Soybeans (Pioneer 28T33R) – 12-14% (w.b.) moisture content and stored at 4°C.
4.11. Deionized water

5.0. PROCEDURES

5.1. Remove approximately 3 kg of soybeans from storage.
5.2. Clean by hand using sieves to remove foreign materials, split, broken, and damaged seeds.
5.3. Weigh the clean soybeans and record measurement ($m_{soy,0}$).
5.4. Let the soybeans stand at room temperature for 30-40 min.
5.5. In triplicates, estimate the soybean moisture content using the handheld digital

moisture meter and record the average measurement ($\widehat{w}_{soy,0}$).

5.6. Calculate the amount of water (m_{H_2O}) needed to achieve desired moisture content ($\widehat{w}_{soy,1}$) using the following equation:

$$m_{H_2O} = m_{soy,0}\left(\frac{\widehat{w}_{soy,1} - \widehat{w}_{soy,0}}{100 - \widehat{w}_{soy,1}}\right)$$

5.7. Weigh out the amount of water needed, and 10 g extra, using a glass beaker and digital scale.

5.8.Carefully transfer clean soybeans into plastic bottle and, using a plastic funnel, add deionized water according to clean soybeans.

5.9. Set filled bottle on its side on the roller mixer. Set mixer to 60 rpm and let the soybeans mix with water for 60 min at room temperature.

5.10. Spread a thin layer of soybeans on aluminum trays and let stand at room temperature to air-dry until $\widehat{w}_{soy,1}$ is reached, according to the digital handheld moisture meter. When $\widehat{w}_{soy,1}$ is reached, record an average moisture measurement using three samples.

5.11. Mix the air-dried soybeans by hand on the tray and remove three samples (25-30 g each) for a gravimetric moisture content measurement (see Document ID: D-GRMS-006).

6.0. PROCEDURE: FINAL STEPS AND REPORTING

6.1. Record all weight measurements and calculations in an electronic datasheet designated for the specific grain respiration test.

6.2. Set aside 400 g of excess soybeans for future soybean quality tests.

6.3. Discard all foreign materials, split, broken, damaged, and any remaining excess soybeans.

6.4. Check for molding and other defects in the soybeans after retrieval from storage.

6.5. Record any issues with cleaning and re-wetting soybeans. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. When mold or defects are found in soybeans retrieved from storage, notify the supervisor immediately. Do not use any soybean with visible molds or emit off-odors in grain respiration tests.

7.2. The supervisor will take further corrective actions which may include discarding soybeans from storage.

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.

8.2. Step 4.1 revised to include the use of two screens to clean soybeans.

8.3. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Gravimetric measurement of grain moisture content**
Effective date: 01 October 2017	Document ID: D-GRMS-006
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for determining soybean moisture content according to ASABE Standard S352.2 (R2017).

2.0. SCOPE

This SOP describes how to determine moisture content of soybeans before and after each grain respiration test.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components, preparing samples, and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. Convection oven (Product No. VWR-U50, VWR, Radnor, PA, USA), set at 103°C, or similar equipment.
4.2. Digital precision balance or scale – Ranges from 0 to 3100 y g with 0.01 g resolution (Model iBalance i3100, MyWeigh, Phoenix, AZ, USA) or similar device with the same range and resolution.
4.3.Desiccator cabinet with silica gel desiccant.
4.4. Aluminum weighing or moisture dishes – 4 oz utility cup full curl (Product No. 42330, Pactiv, Lake Forest, IL, USA), or similar materials.
4.5. Timer – Digital compact timer (Product No. 5806, Taylor, Oak Brooks, IL).
4.6. Soybeans (Pioneer 28T33R) – 12-14% (w.b.) moisture content and stored at 4°C.

5.0. PROCEDURES

5.1. Label three aluminum moisture dishes and label each with a unique ID. For example, “Test 1A-1a, Test 1A-2a, and Test 1A-3a” labels are for three samples for Test 1. The uppercase letters denote whether samples were taken (A) before and (B) after a grain respiration test. Lowercase letters denote whether samples were filled with (a) wet or (b) oven-dried soybeans.
5.2. Tare the empty digital scale. Place dish on the scale and record its mass (e.g., m_{1A}, m_{2A}, m_{3A}).
5.3. Without touching or moving the dish, gently pour one of the re-wetted soybean samples (see Document ID: D-GRMS-005, Step 5.11). Record the total mass of the dish with wet soybeans (e.g., $m_{1A-1a}, m_{1A-2a}, m_{1A-3a}$). Carefully remove dish with wet soybeans from the scale.
5.4. Repeat Steps 5.2 and 5.3 for the two remaining re-wetted soybean samples.

5.5. Place the three filled dishes on an aluminum tray and dry the beans in a convection oven set at 103°C for 72 h.

5.6. Remove the tray from the oven and cool in a desiccator cabinet with active desiccant for 20-30 min.

5.7. Carefully remove from cabinet and quickly weigh each dish's dry weight (e.g., $m_{1A-1b}, m_{1A-2b}, m_{1A-3b}$).

5.8. For sample 1, calculate the following:

mass of wet soybeans: $m_{soy,1a} = m_{1A-1a} - m_{1A}$
mass of dry matter: $m_{DM,1} = m_{1A-1a} - m_{1A-1b}$
mass of moisture removed: $m_{H_2O,1} = m_{1A-1a} - m_{1A} - m_{DM,1}$
moisture content of soybean sample: $w_{soy,1a} = \frac{m_{H_2O,1}}{m_{soy,1a}}$

5.9. Repeat Step 5.8 for the other two soybean samples.

5.10. Calculate the average moisture content of soybeans taken before a grain respiration test:

$$\overline{w}_{soy,1} = \frac{w_{soy,1a} + w_{soy,2a} + w_{soy,3a}}{3}$$

5.11. To determine average moisture content of samples taken after a grain respiration test, repeat Steps 5.1 to 5.10 taking care to label samples appropriately.

6.0. PROCEDURE: FINAL STEPS AND REPORTING

6.1. Record all weight measurements and calculations in an electronic datasheet designated for the specific grain respiration test.

6.2. Record any issues with determining moisture content of soybeans gravimetrically. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. Pay attention to calibration, drift, bias, etc. issues with the digital scale. Be sure to use the same digital scale for all weight measurements throughout a grain respiration study (not just individual tests or experiments). When scale issues arise, notify the supervisor immediately.

7.2. The supervisor will take further corrective actions which may include re-calibrating or replacing the digital scale.

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.

8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

University of Illinois at Urbana-Champaign	**Title: Running a grain respiration test**
Effective date: 01 October 2017	Document ID: D-GRMS-007
Written by: L.R. Trevisan	Approved by: R.S. Gates (supervisor)

1.0. PURPOSE

This SOP explains the protocol for running a grain respiration test.

2.0. SCOPE

This SOP describes how to set-up D-GRMS prior to starting a test, collect and measure respired CO_2 by the soybeans, calculate dry matter loss over time, end the test, and clean up.

3.0. RESPONSIBILITY

The supervisor will be responsible for training the personnel on proper use of D-GRMS and its components, preparing samples, and implementing the protocol/procedure.

4.0. MATERIALS AND EQUIPMENT

4.1. D-GRMS with air conditioning and flow management, grain storage, moisture and CO_2 absorption, and instrumentation sections (see Figure 3.1).

4.2. Air supply CO_2 scrubber (qty = 1) prepared according to Document No. D-GRMS-001.

4.3. RC CO_2 scrubbers (qty = 2) prepared according to Document No. D-GRMS-002.

4.4. RC dehumidifiers (qty = 2) prepared according to Document No. D-GRMS-003.

4.5. Glycerol-water solution (qty = 4 L) prepared according to Document No. D-GRMS-004.

4.6. Clean, re-wetted soybeans (qty = 1.8 kg) prepared according to Document No. D-GRMS-005.

4.7. Digital precision balance or scale – Ranges from 0 to 3100 g with 0.01 g resolution (Model iBalance i3100, MyWeigh, Phoenix, AZ, USA) or similar device with the same range and resolution.

4.8. Power supply – Regulated power supply 24V (Model No. PSR-2/24, EMCO, Allen, TX, USA) for mass flow controller and Vaisala sensors.

4.9. Power supply (110 V_{ac}) for water baths, lamp bulb, heat tape.

4.10. Glass beaker, 3000 ml capacity

4.11. Aluminum tray, 92 x 62 x 3 cm.

5.0. PROCEDURES

5.1. Turn ON all system components of the D-GRMS. Allow water baths to reach test temperatures.

5.2. Open electronic datasheet template and save the worksheet using a unique name that denotes the specific grain respiration test (e.g., 14%-35C-rep1).

5.3. Install air supply CO_2 scrubber, RC CO_2 scrubbers, RC dehumidifiers, and glycerol-water solutions in the D-GRMS following their respective SOPs.

5.4. Weigh 1.8 kg of clean, re-wetted soybeans. Record actual weight ($m_{soy,1}$) in the datasheet.

5.5. Using the moisture content of the soybeans ($w_{soy,1}$), estimate the mass of dry solids of the soybeans:

$$m_{DM,1} = m_{soy,1}\left(1 - \frac{w_{soy,1}}{100}\right)$$

5.6. Carefully pour beans into the RC. Cover the RC, secure the lid, and install it in the D-GRMS.

5.7. Turn ON pressure regulator valve of the air supply. Check flow rate as indicated on the LCD display of the Arduino-based fine adjustment of the mass flow controller. If $Q \neq 0.50 \pm 0.02$ L min^{-1}, adjust by tuning the digital potentiometer accordingly by pressing the buttons on the top of the controller.

5.8. Check all components, tubing, and insulation of the assembled D-GRMS for any cracks, loose connections, or other defects that could allow gas to leak in/out of the system during a test.

5.9. For 10 min, let the compressed air flow through the entire system to flush out air and CO_2 present in the tubing initially.

5.10. Turn ON the serial monitor of the Arduino software to start recording temperature, relative humidity, and CO_2 levels at the inlet of the respiration chamber (RC) and at the exhaust of the D-GRMS.

5.11. After 10 min, check the temperature, relative humidity, and CO_2 levels. If temperature and relative humidity are off their limits, adjust water bath thermostats and glycerol-water solution concentration, respectively. If CO_2 levels are off their limits, abort the test by turning OFF all system components.

5.12. Record the time when desired test conditions have been achieved.

5.13. Let the system run for 12-14 h undisturbed. During this period, the soybeans are acclimating to their new storage environment.

5.14. Quickly divert airflow from RC CO_2 scrubber A to RC CO_2 scrubber B.

5.15. Detach scrubber A from D-GRMS. Determine its average weight from three measurements, taking care to rotate the scrubber 120° in between measurements. Record all measurements and calculations in an electronic datasheet, along with the time of measurement.

5.16. Install scrubber A back into D-GRMS.

5.17. After 2 h, divert airflow from scrubber B to scrubber A.

5.18. Repeat Steps 5.14 and 5.15 with scrubber B.

5.19. Conduct weight measurements every 2 h during the daytime, alternating between the two scrubbers each time.

5.20. All weight measurements represent the accumulated respired CO_2 in the scrubbers. Normalize each weight measurement to $m_{DM,1}$.

5.21. When 22 g CO_2 (kg dry beans)$^{-1}$ is reached, terminate the grain respiration test.

6.0. PROCEDURE: FINAL STEPS AND REPORTING

6.1. Record all weight measurements, time of measurements, and calculations in an electronic datasheet designated for the specific grain respiration test.

6.2. At the end of each test, turn OFF all system components and remove the lid of RC.

6.3. Place a 3000 L glass beaker on a digital scale. Tare the weight.

6.4. Gently transfer soybeans into the beaker to determine and record its weight.

6.5. Spread the soybeans onto an aluminum tray. Mix manually by hand and retrieve three samples (25-30 g each).

6.6. Determine the moisture content of the soybeans gravimetrically following procedures outlined in Document No. D-GRMS-0006.

6.7. Set aside 400 g of soybeans for future quality tests. Discard remaining soybeans following disposal guidelines by the UIUC Division of Research Safety.

6.8. Record any issues encountered during a grain respiration test. If there are any issues, see corrective action (Section 7.0).

7.0. CORRECTIVE ACTION

7.1. Pay attention to calibration, drift, bias, etc. issues with the digital scale. Be sure to use the same digital scale for all weight measurements throughout a grain respiration study (not just individual tests or experiments). When scale issues arise, notify the supervisor immediately.

7.2. Pay attention to all sensor readings. If temperature, relative humidity and flow rate stray from their limits, make adjustments accordingly. Note adjustments in the electronic datasheets and notify supervisor immediately. If CO_2 levels go off limits, abort grain respiration test and notify supervisor immediately.

7.3. The supervisor will take further corrective actions which may include testing for leaks and repairing or replacing system components,

8.0. CHANGES FROM PREVIOUS VERSION

8.1. SOP drafted on 01 July 2016.

8.2. Reviewed, revised, and approved by supervisor on 01 October 2017.

A.3. Preliminary tests: Performance of respiration chamber (RC) CO_2 scrubbers

Materials and Methods

A series of preliminary tests was conducted to test the effect of CO_2 loading rate ($\hat{v}_{CO_2}$) and moisture content of the CO_2 absorbent (Sodasorb®, w_{abs}) on the performance of RC CO_2 scrubbers used in the D-GRMS, specifically, on their breakthrough times (t_b). To vary w_{abs}, Sodasorb® was exposed to a desiccant (Catalog No. 21001, WA Hammond Drierite Co., Ltd., Xenia, OH, USA) for 0 to 5 days. At the end of the drying period, a 30 g subsample of the Sodasorb® was assessed for w_{abs} by drying in a convection oven at 103°C for 24 h. Afterwards, a range of RC CO_2 scrubbers with varying w_{abs} was prepared and tested.

A series of certified tanks of CO_2 (0.1, 1, or 10% CO_2, Aigas, Danville, IL, USA) was coupled to the mass flow controller and used to deliver CO_2 at known $\hat{v}_{CO_2}$ to an RC CO_2 scrubber for a period of time t (Figure A.3). T, ϕ, and C_{CO_2} were monitored at the exhaust to ensure T was uniform throughout the testing period and ϕ and C_{CO_2} were zero, indicating all CO_2 and moisture produced by the Sodasorb® during the absorption process were retained in the scrubber.

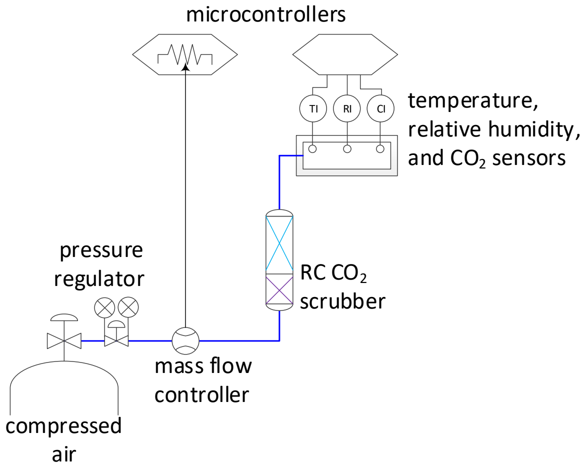

Figure A.3. Experiment setup in testing performance of an RC CO_2 scrubber. Instrumentation details are provided in Figures A.1 and A.2.

With a constant and known $\hat{v}_{CO_2}$ during testing period t, the theoretical accumulated mass of CO_2 to be retained in the scrubber could be determined:

$$\sum \widehat{m}_{CO_2} = \hat{v}_{CO_2} t$$

The initial mass of the scrubber was recorded. Any changes to it would be attributable to the actual $\sum m_{CO_2}$ in the scrubber. Hence, the CO_2 gas supply was turned OFF periodically during testing so that the scrubber could be removed and weighed using a digital scale (Model i3100, MyWeight, Phoenix, AZ). Both t and $\sum m_{CO_2}$ were recorded in an electronic datasheet. After each weighing, the scrubber was put back in place and testing resumed. Tests ended when the scrubber reached t_b or 8 h had passed since the start of a test.

Results were analyzed by comparing $\sum m_{CO_2}$ to $\sum \widehat{m}_{CO_2}$ and performing a linear regression using the Data Analysis ToolPak in MS Excel (Version 2016, Microsoft Corporation, Redmond, Washington, USA). Slope and intercept values that were not statistically different from unity and zero, respectively, indicated good agreement between $\sum m_{CO_2}$ and $\sum \widehat{m}_{CO_2}$.

Results and discussion

The RC CO_2 scrubbers were tested at loading rates $\hat{v}_{CO_2}$ of 0.1 to 23.7 g CO_2 h^{-1}. The slopes, $\sum m_{CO_2}/\sum \widehat{m}_{CO_2}$, of the calibration curves were nearly unity for $\hat{v}_{CO_2} < 10$ g CO_2 h^{-1} and intercepts were nearly zero, indicating a high efficacy for the scrubber (Table A.1). As $\hat{v}_{CO_2}$ increased from 10 g CO_2 h^{-1}, slopes decreased from 1.0 to 0.90 and intercepts became significantly different from zero, indicating failure of the RC CO_2 scrubber to capture, or absorb, all incoming CO_2. As $\hat{v}_{CO_2}$ increased, t_b decreased. The efficacy of the RC CO_2 scrubber was also tested for $1.83\% \leq w_{abs} \leq 12.04\%$. Results showed that when $w_{abs} \leq 10\%$, $\sum m_{CO_2}/\sum \widehat{m}_{CO_2} \neq 1$ and intercept was non-zero (Table A.1). Hence, w_{abs} must be maintained above 10% to function properly.

Table A.1. Results from testing performance of RC CO_2 scrubbers

Test No.	Test Inputs					Test Results		
	w_{abs}	C_{CO_2}		Q	$\hat{v}_{CO_2}$	Slope	Intercept	R^2
	(%)	(%)	(ppm)	(L min^{-1})	g CO_2 h^{-1}	$\sum m_{CO_2} : \sum \widehat{m}_{CO_2}$		
1	11.44	1.0	10000	0.5	0.6	0.9815	0.0679	0.99
2	9.83	1.0	10000	1.0	1.2	0.9630	0.1692	0.99
3	11.12	1.0	10000	0.5	0.6	1.0112	0.0346	0.99
4	9.92	1.0	10000	0.5	0.6	0.9820	0.0182	0.99
5	9.88	1.0	10000	0.5	0.6	1.0143	0.0556	0.99
6	9.85	1.0	10000	0.5	0.6	1.0182	0.0030	0.99
7	12.04	1.0	10000	0.5	0.6	0.9840	0.0121	0.99
8	10.73	1.0	10000	0.2	0.2	0.8504	0.0436	0.99
9	10.84	1.0	10000	0.1	0.1	1.3097	0.0424	0.98
10	8.40	1.0	10000	1.0	11.9	0.6009	7.1861	0.90
11	11.97	1.0	10000	1.0	11.9	0.7609	0.3512	0.98
12	11.76	10	1000000	1.0	23.7	0.8437	1.0421	0.99
13	10.83	10	1000000	1.3	15.4	0.6992	2.6435	0.97
14	10.18	10	1000000	1.5	17.8	0.6473	3.4833	0.91
15	9.52	10	1000000	1.5	17.8	0.3861	5.1276	0.89
16	6.75	0.04	450	0.6	0.1	0.8775	0.0183	0.99
17	1.83	0.04	450	0.6	0.1	0.5606	0.1799	0.95

APPENDIX B. SOYBEAN RESPIRATION DATA AT 35°C

Table B.1. Soybeans (14%) – Replication No. 1

Start date and time:	05/15/2017 16:50	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	05/27/2017 17:10	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	12	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	14.23 ± 0.1154	m_{soy} (g) =	1785.69
$\overline{w_{soy,1}}$ (%) =	14.31 ± 0.0019	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	255.55
$\overline{w_{soy,2}}$ (%) =	14.57 ± 0.0024	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1530.14

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t'[1] [d]	DML'[1] [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	932.39	949.88			0.00	0.00	0.00		
0.64	932.50		0.11		0.11	0.07	0.00		
0.72		949.90		0.02	0.13	0.08	0.01		
0.84	932.54		0.04		0.17	0.11	0.01		
0.88		949.95		0.05	0.22	0.15	0.01		
1.00	932.63		0.09		0.31	0.20	0.01		
1.05		950.08		0.13	0.44	0.29	0.02		
1.64	933.18		0.55		0.99	0.65	0.04		
1.72		950.14		0.06	1.05	0.68	0.05		
1.80	933.27		0.09		1.14	0.74	0.05	0.01	0.00
1.89		950.19		0.06	1.19	0.78	0.05	0.10	0.00
1.97	933.40		0.13		1.33	0.87	0.06	0.17	0.00
2.06		950.28		0.09	1.41	0.92	0.06	0.26	0.01
2.63	935.97		2.57		3.98	2.60	0.18	0.84	0.12
2.72		950.56		0.28	4.26	2.78	0.19	0.92	0.14
2.80	936.12		0.15		4.41	2.88	0.20	1.01	0.14
2.88		950.87		0.31	4.72	3.08	0.21	1.09	0.16
2.98	936.31		0.19		4.91	3.21	0.22	1.18	0.16
3.65		952.83		1.96	6.87	4.49	0.31	1.86	0.25
3.72	936.64		0.33		7.20	4.70	0.32	1.92	0.27
3.81		952.95		0.12	7.32	4.78	0.33	2.02	0.27
3.89	936.89		0.25		7.57	4.95	0.34	2.10	0.28
3.98		953.11		0.16	7.73	5.05	0.34	2.19	0.29
4.05	937.01		0.12		7.85	5.13	0.35	2.26	0.30
4.64		956.07		2.95	10.80	7.06	0.48	2.85	0.43
4.73	937.26		0.25		11.06	7.23	0.49	2.93	0.44

Table B.1. Continued

4.80		956.12		0.05	11.11	7.26	0.50	3.01	0.44
4.88	937.31		0.05		11.16	7.30	0.50	3.09	0.44
4.97		956.31		0.19	11.35	7.42	0.51	3.17	0.45
5.65	938.62		1.31		12.66	8.27	0.56	3.85	0.51
5.72		956.60		0.29	12.95	8.46	0.58	3.93	0.52
5.81	938.97		0.35		13.30	8.69	0.59	4.01	0.54
5.88		956.87		0.27	13.57	8.87	0.60	4.09	0.55
6.63	941.53		2.56		16.13	10.54	0.72	4.84	0.66
6.73		957.47		0.60	16.73	10.93	0.75	4.94	0.69
6.80	941.74		0.21		16.94	11.07	0.75	5.01	0.70
6.88		957.80		0.33	17.27	11.29	0.77	5.09	0.72
6.97	941.92		0.18		17.45	11.41	0.78	5.17	0.72
7.67		958.27		0.46	17.92	11.71	0.80	5.88	0.74
7.80	942.10		0.18		18.10	11.83	0.81	6.01	0.75
7.92		958.85		0.58	18.68	12.21	0.83	6.13	0.78
8.03	942.26		0.16		18.84	12.31	0.84	6.23	0.78
8.64		960.46		1.61	20.45	13.36	0.91	6.85	0.86
8.64	942.52		0.26		20.71	13.53	0.92	6.85	0.87
8.80		960.52		0.06	20.77	13.57	0.93	7.01	0.87
8.88	942.80		0.28		21.05	13.76	0.94	7.09	0.88
8.98		960.89		0.37	21.42	14.00	0.95	7.18	0.90
9.07	944.84		2.04		23.46	15.33	1.05	7.27	0.99
9.63		961.13		0.24	23.70	15.49	1.06	7.84	1.00
9.73	945.15		0.31		24.01	15.69	1.07	7.94	1.02
9.81		961.50		0.37	24.38	15.94	1.09	8.01	1.03
9.88	945.50		0.35		24.74	16.17	1.10	8.09	1.05
9.97		961.70		0.20	24.94	16.30	1.11	8.17	1.06
10.05	947.21		1.70		26.64	17.41	1.19	8.25	1.13
10.63		961.97		0.26	26.90	17.58	1.20	8.84	1.14
10.72	947.51		0.30		27.21	17.78	1.21	8.92	1.16
10.81		962.22		0.25	27.46	17.95	1.22	9.02	1.17
10.90	947.84		0.33		27.79	18.16	1.24	9.10	1.18
10.97		962.52		0.30	28.09	18.36	1.25	9.18	1.20
11.05	951.77		3.92		32.02	20.92	1.43	9.26	1.37
11.68		962.98		0.46	32.48	21.23	1.45	9.89	1.39
11.78	952.13		0.36		32.84	21.46	1.46	9.98	1.41
11.86		963.55		0.56	33.41	21.83	1.49	10.06	1.43
11.92	952.66		0.53		33.93	22.18	1.51	10.10	1.45

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.2. Soybeans (14%) – Replication No. 2

Start date and time:	06/14/2017 21:23	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	06/29/2017 09:30	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	15	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	14.67 ± 0.1155	m_{soy} (g) =	1809.91
$\overline{w_{soy,1}}$ (%) =	14.26 ± 0.0015	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	258.10
$\overline{w_{soy,2}}$ (%) =	14.57 ± 0.0009	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1551.81

t [d]	$(\overline{m_{RC}})_*$ [g] $*= A,t$	$*= B,t$	$\left(\sum m_{CO_2}\right)_*$ [g] $*= A$	$*= B$	$*= A+B$	$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
0.00	980.82	986.49			0.00	0.00	0.00		
0.55	980.89		0.07		0.07	0.05	0.00		
0.63		986.49		0.00	0.07	0.05	0.00		
0.71	980.90		0.01		0.08	0.05	0.00		
0.80		986.50		0.01	0.09	0.06	0.00		
0.87	980.90		0.00		0.09	0.06	0.00		
0.92		986.51		0.01	0.10	0.06	0.00		
1.49	981.52		0.62		0.72	0.46	0.03		
1.62		986.53		0.02	0.74	0.47	0.03		
1.69	981.56		0.04		0.78	0.50	0.03		
1.79		986.57		0.04	0.82	0.53	0.04		
1.86	981.58		0.02		0.84	0.54	0.04		
2.47		986.83		0.26	1.10	0.71	0.05		
2.55	981.63		0.04		1.15	0.74	0.05	0.01	0.00
2.63		986.87		0.04	1.19	0.76	0.05	0.09	0.00
2.72	981.64		0.02		1.20	0.78	0.05	0.19	0.00
2.83		986.95		0.08	1.29	0.83	0.06	0.30	0.00
3.45	982.28		0.64		1.92	1.24	0.08	0.92	0.03
3.54		987.00		0.05	1.97	1.27	0.09	1.01	0.04
3.61	982.32		0.04		2.01	1.30	0.09	1.08	0.04
3.74		987.06		0.06	2.07	1.34	0.09	1.21	0.04
4.47	983.46		1.14		3.21	2.07	0.14	1.94	0.09
4.53		987.17		0.10	3.32	2.14	0.15	1.99	0.10
4.62	983.57		0.11		3.43	2.21	0.15	2.08	0.10
4.69		987.29		0.12	3.55	2.29	0.16	2.16	0.11
4.79	983.69		0.12		3.67	2.36	0.16	2.26	0.11
4.86		987.39		0.10	3.77	2.43	0.17	2.32	0.12
5.48	985.43		1.74		5.51	3.55	0.24	2.95	0.19

Table B.2. Continued

5.58		987.56		0.17	5.68	3.66	0.25	3.05	0.20
5.65	985.59		0.15		5.83	3.76	0.26	3.12	0.21
5.75		987.71		0.15	5.98	3.86	0.26	3.22	0.21
5.82	985.69		0.11		6.09	3.92	0.27	3.29	0.22
6.42		990.18		2.47	8.56	5.52	0.38	3.89	0.33
6.48	985.82		0.13		8.69	5.60	0.38	3.95	0.33
6.61		990.40		0.22	8.91	5.74	0.39	4.08	0.34
6.69	986.06		0.24		9.15	5.90	0.40	4.16	0.35
6.80		990.55		0.15	9.30	5.99	0.41	4.26	0.36
7.45	988.57		2.52		11.82	7.61	0.52	4.92	0.47
7.54		990.73		0.18	12.00	7.73	0.53	5.00	0.48
7.61	988.75		0.18		12.17	7.84	0.53	5.07	0.48
7.71		990.87		0.13	12.31	7.93	0.54	5.17	0.49
7.78	988.96		0.21		12.52	8.07	0.55	5.24	0.50
8.44		992.97		2.11	14.62	9.42	0.64	5.91	0.59
8.53	989.20		0.24		14.86	9.58	0.65	5.99	0.60
8.62		993.22		0.24	15.11	9.73	0.66	6.08	0.61
8.70	989.41		0.21		15.32	9.87	0.67	6.16	0.62
8.86		993.43		0.22	15.54	10.01	0.68	6.32	0.63
9.46	992.36		2.95		18.48	11.91	0.81	6.92	0.76
9.53		993.77		0.34	18.82	12.13	0.83	6.99	0.78
9.61	992.55		0.19		19.01	12.25	0.84	7.07	0.78
9.70		994.02		0.25	19.26	12.41	0.85	7.16	0.80
9.80	992.80		0.25		19.52	12.58	0.86	7.26	0.81
9.86		994.21		0.19	19.70	12.70	0.87	7.33	0.82
10.44	995.58		2.78		22.48	14.49	0.99	7.91	0.94
10.58		994.46		0.25	22.73	14.65	1.00	8.05	0.95
10.69	995.83		0.25		22.98	14.81	1.01	8.16	0.96
10.80		994.71		0.25	23.23	14.97	1.02	8.26	0.97
11.45	998.68		2.85		26.08	16.81	1.15	8.91	1.10
11.54		995.01		0.30	26.38	17.00	1.16	9.00	1.11
11.62	998.91		0.23		26.61	17.15	1.17	9.08	1.12
11.70		995.33		0.31	26.93	17.35	1.18	9.16	1.13
11.78	999.22		0.31		27.24	17.55	1.20	9.24	1.15
11.87		995.68		0.35	27.59	17.78	1.21	9.34	1.16
12.46	1000.6		1.35		28.94	18.65	1.27	9.93	1.22
12.54		995.89		0.21	29.15	18.79	1.28	10.01	1.23
12.62	1000.8		0.26		29.42	18.96	1.29	10.08	1.24
12.71		996.18		0.29	29.71	19.15	1.31	10.17	1.26

Table B.2. Continued

12.78	1001.0		0.18		29.89	19.26	1.31	10.25	1.26
12.86		996.52		0.34	30.23	19.48	1.33	10.32	1.28
13.45	1003.6		2.53		32.76	21.11	1.44	10.92	1.39
13.53		996.81		0.29	33.05	21.30	1.45	11.00	1.40
13.62	1003.8		0.23		33.28	21.45	1.46	11.09	1.41
13.71		997.13		0.32	33.60	21.65	1.48	11.18	1.42
13.79	1004.0		0.24		33.84	21.80	1.49	11.26	1.43
14.50		1001.3		4.19	38.02	24.50	1.67	11.31	1.45

[1]Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.3. Soybeans (14%) – Replication No. 3

Start date and time:	08/18/2017 18:15	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	09/05/2017 14:18	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	18	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	14.43 ± 0.1528	m_{soy} (g) =	1798.33
$\overline{w_{soy,1}}$ (%) =	14.73 ± 0.0002	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	264.84
$\overline{w_{soy,2}}$ (%) =	14.51 ± 0.0007	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1533.49

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$* = A,t$	$* = B,t$	$* = A$	$* = B$	$* = A + B$				
0.00	933.38	932.70			0.00	0.00	0.00		
0.64	933.38		0.00		0.00	0.00	0.00		
0.73		932.70		0.00	0.00	0.00	0.00		
0.80	933.38		0.00		0.00	0.00	0.00		
0.89		932.70		0.01	0.01	0.00	0.00		
0.98	933.39		0.01		0.01	0.01	0.00		
1.58		932.71		0.01	0.02	0.01	0.00		
1.66	933.42		0.03		0.05	0.03	0.00		
1.75		932.71		0.00	0.05	0.03	0.00		
1.83	933.43		0.01		0.06	0.04	0.00		
1.97		932.71		0.00	0.06	0.04	0.00		
2.58	933.48		0.05		0.10	0.07	0.00		
2.66		932.71		0.00	0.11	0.07	0.00		
2.84	933.48		0.00		0.11	0.07	0.00		
2.95		932.72		0.01	0.12	0.08	0.01		
3.58	933.49		0.01		0.12	0.08	0.01		

Table B.3. Continued

3.75		932.72		0.00	0.13	0.08	0.01		
3.92	933.52		0.03		0.16	0.10	0.01		
4.59		932.77		0.05	0.21	0.14	0.01		
4.67	933.55		0.03		0.24	0.16	0.01		
4.76		932.78		0.01	0.25	0.17	0.01		
4.84	933.58		0.03		0.29	0.19	0.01		
4.93		932.80		0.02	0.30	0.20	0.01		
5.02	933.60		0.02		0.32	0.21	0.01		
5.60		932.87		0.07	0.39	0.25	0.02		
5.74	933.63		0.03		0.42	0.27	0.02		
5.84		932.87		0.00	0.42	0.27	0.02		
5.91	933.66		0.03		0.45	0.30	0.02		
6.00		932.88		0.01	0.46	0.30	0.02		
6.05	933.68		0.02		0.48	0.32	0.02		
6.63		933.20		0.32	0.80	0.52	0.04		
6.72	933.72		0.04		0.84	0.55	0.04		
6.78		933.22		0.02	0.86	0.56	0.04		
6.86	933.75		0.03		0.89	0.58	0.04		
6.96		933.25		0.03	0.92	0.60	0.04		
7.03	933.78		0.03		0.95	0.62	0.04		
7.62		935.39		2.14	3.09	2.02	0.14	0.54	0.09
7.74	933.80		0.02		3.11	2.03	0.14	0.66	0.09
7.83		935.44		0.05	3.16	2.06	0.14	0.75	0.09
7.96	933.91		0.11		3.27	2.13	0.15	0.88	0.10
8.59		935.80		0.37	3.63	2.37	0.16	1.51	0.11
8.72	933.95		0.04		3.68	2.40	0.16	1.64	0.11
8.82		935.88		0.08	3.76	2.45	0.17	1.74	0.12
8.91	934.02		0.07		3.82	2.49	0.17	1.83	0.12
9.59		936.10		0.22	4.04	2.63	0.18	2.52	0.13
9.68	934.10		0.08		4.12	2.68	0.18	2.60	0.13
9.76		936.15		0.05	4.17	2.72	0.19	2.68	0.14
9.84	934.17		0.07		4.24	2.77	0.19	2.76	0.14
9.92		936.43		0.28	4.52	2.95	0.20	2.85	0.15
10.00	934.22		0.05		4.57	2.98	0.20	2.92	0.15
10.61		938.36		1.93	6.50	4.24	0.29	3.54	0.24
10.70	934.56		0.33		6.84	4.46	0.30	3.62	0.25
10.79		938.68		0.32	7.15	4.66	0.32	3.71	0.27
10.87	934.98		0.43		7.58	4.94	0.34	3.79	0.29
10.96		939.22		0.54	8.12	5.30	0.36	3.88	0.31

Table B.3. Continued

11.57	937.25		2.26		10.39	6.77	0.46	4.49	0.41
11.66		939.50		0.28	10.67	6.96	0.47	4.58	0.42
11.74	937.59		0.34		11.01	7.18	0.49	4.66	0.44
11.82		939.72		0.22	11.23	7.32	0.50	4.74	0.45
11.91	937.81		0.22		11.45	7.47	0.51	4.83	0.46
11.99		939.95		0.23	11.68	7.62	0.52	4.91	0.47
12.58	939.69		1.88		13.56	8.84	0.60	5.50	0.55
12.67		940.28		0.33	13.89	9.06	0.62	5.59	0.57
12.75	940.01		0.32		14.20	9.26	0.63	5.67	0.58
12.83		940.55		0.28	14.48	9.44	0.64	5.75	0.59
12.92	940.30		0.29		14.77	9.63	0.66	5.84	0.61
12.99		940.77		0.21	14.98	9.77	0.67	5.91	0.62
13.58	942.25		1.95		16.93	11.04	0.75	6.50	0.70
13.66		941.12		0.36	17.29	11.27	0.77	6.58	0.72
13.75	942.63		0.38		17.67	11.52	0.79	6.67	0.74
13.84		941.48		0.36	18.03	11.76	0.80	6.76	0.75
13.94	943.05		0.42		18.45	12.03	0.82	6.86	0.77
14.62		943.66		2.18	20.63	13.45	0.92	7.54	0.87
14.70	943.44		0.39		21.02	13.71	0.93	7.62	0.88
14.78		943.93		0.27	21.29	13.88	0.95	7.70	0.90
14.86	943.77		0.33		21.62	14.10	0.96	7.79	0.91
14.96		944.32		0.39	22.01	14.35	0.98	7.88	0.93
15.57	946.13		2.36		24.37	15.89	1.08	8.49	1.03
15.67		944.76		0.44	24.81	16.18	1.10	8.59	1.05
15.75	946.41		0.28		25.09	16.36	1.12	8.67	1.07
15.84		945.10		0.34	25.43	16.58	1.13	8.76	1.08
15.91	946.73		0.32		25.75	16.79	1.14	8.83	1.09
16.00		945.45		0.35	26.10	17.02	1.16	8.92	1.11
16.57	949.14		2.41		28.51	18.59	1.27	9.49	1.22
16.66		945.88		0.43	28.94	18.87	1.29	9.58	1.24
16.74	949.56		0.42		29.36	19.15	1.31	9.66	1.26
16.92		946.24		0.36	29.72	19.38	1.32	9.84	1.27
16.99	950.08		0.52		30.24	19.72	1.34	9.92	1.29
17.59		948.87		2.63	32.87	21.43	1.46	10.51	1.41
17.67	950.43		0.35		33.22	21.66	1.48	10.59	1.43
17.75		949.26		0.39	33.60	21.91	1.49	10.67	1.44
17.84	950.80		0.37		33.97	22.15	1.51	10.70	1.45

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.4. Soybeans (14%) – Replication No. 4

Start date and time:	09/05/2017 21:05	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	09/20/2017 09:15	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	15	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	14.13 ± 0.0577	m_{soy} (g) =	1798.39
$\overline{w_{soy,1}}$ (%) =	14.02 ± 0.0007	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	252.06
$\overline{w_{soy,2}}$ (%) =	13.79 ± 0.0008	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1546.33

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	981.77	982.12			0.00	0.00	0.00		
0.53	981.80		0.03		0.03	0.02	0.00		
0.61		982.13		0.01	0.04	0.03	0.00		
0.66	981.80		0.00		0.04	0.03	0.00		
0.80		982.16		0.03	0.07	0.05	0.00		
1.51	981.86		0.06		0.13	0.08	0.01		
1.60		982.19		0.04	0.16	0.11	0.01		
1.68	981.88		0.02		0.19	0.12	0.01		
1.81		982.22		0.03	0.21	0.14	0.01		
2.48	981.94		0.06		0.27	0.17	0.01		
2.58		982.28		0.06	0.33	0.21	0.01		
2.66	981.96		0.02		0.34	0.22	0.02		
2.76		982.30		0.02	0.37	0.24	0.02		
2.85	981.97		0.01		0.38	0.25	0.02		
3.47		982.37		0.07	0.45	0.29	0.02		
3.55	981.98		0.01		0.46	0.30	0.02		
3.63		982.39		0.02	0.49	0.31	0.02		
3.72	982.02		0.03		0.52	0.34	0.02		
3.80		982.41		0.02	0.54	0.35	0.02		
4.45	982.13		0.12		0.65	0.42	0.03		
4.58		982.43		0.02	0.67	0.44	0.03		
4.67	982.14		0.01		0.68	0.44	0.03		
4.76		982.45		0.02	0.71	0.46	0.03		
4.83	982.17		0.03		0.73	0.47	0.03		
5.45		982.58		0.13	0.86	0.56	0.04		
5.55	982.20		0.03		0.89	0.57	0.04		
5.62		982.64		0.06	0.94	0.61	0.04		
5.73	982.23		0.03		0.97	0.63	0.04		

Table B.4. Continued

5.81		982.67		0.03	1.01	0.65	0.04		
5.88	982.28		0.05		1.06	0.69	0.05		
6.47		984.37		1.70	2.76	1.78	0.12	0.56	0.07
6.55	982.42		0.14		2.90	1.88	0.13	0.64	0.08
6.63		984.56		0.19	3.09	2.00	0.14	0.72	0.09
6.70	982.61		0.19		3.28	2.12	0.14	0.80	0.09
6.81		984.78		0.22	3.50	2.27	0.15	0.90	0.10
7.47	985.70		3.09		6.59	4.26	0.29	1.56	0.24
7.56		985.04		0.26	6.85	4.43	0.30	1.65	0.25
7.64	986.09		0.39		7.24	4.68	0.32	1.73	0.27
7.72		985.42		0.37	7.61	4.92	0.34	1.81	0.29
7.81	986.31		0.22		7.84	5.07	0.35	1.90	0.30
7.91		985.62		0.20	8.04	5.20	0.35	2.01	0.30
8.50	988.57		2.26		10.30	6.66	0.45	2.60	0.40
8.59		985.91		0.29	10.59	6.85	0.47	2.68	0.42
8.67	988.90		0.33		10.92	7.06	0.48	2.76	0.43
8.75		986.16		0.25	11.17	7.22	0.49	2.85	0.44
8.83	989.17		0.27		11.44	7.40	0.50	2.92	0.45
9.47		988.46		2.30	13.74	8.89	0.61	3.56	0.56
9.56	989.38		0.21		13.95	9.02	0.62	3.65	0.57
9.64		988.72		0.25	14.21	9.19	0.63	3.73	0.58
9.72	989.69		0.31		14.51	9.39	0.64	3.81	0.59
9.81		989.02		0.31	14.82	9.58	0.65	3.91	0.60
9.91	990.00		0.32		15.14	9.79	0.67	4.00	0.62
10.45		991.61		2.59	17.72	11.46	0.78	4.55	0.73
10.54	990.37		0.37		18.09	11.70	0.80	4.63	0.75
10.63		991.99		0.38	18.47	11.94	0.81	4.72	0.76
10.90	990.76		0.39		18.86	12.20	0.83	5.00	0.78
11.54		994.78		2.79	21.65	14.00	0.95	5.63	0.90
11.63	991.17		0.41		22.06	14.27	0.97	5.72	0.92
11.70		995.20		0.42	22.48	14.54	0.99	5.80	0.94
11.79	991.57		0.40		22.88	14.80	1.01	5.88	0.96
11.88		995.63		0.43	23.31	15.08	1.03	5.97	0.98
12.47	994.42		2.85		26.16	16.92	1.15	6.56	1.10
12.55		996.09		0.46	26.62	17.22	1.17	6.64	1.12
12.63	994.86		0.44		27.06	17.50	1.19	6.73	1.14
12.75		996.55		0.45	27.52	17.79	1.21	6.84	1.16
12.83	995.31		0.45		27.97	18.09	1.23	6.92	1.18
12.93		997.02		0.47	28.44	18.39	1.25	7.02	1.20

Table B.4. Continued

13.48	998.60		3.29		31.73	20.52	1.40	7.57	1.35
13.56		997.50		0.48	32.21	20.83	1.42	7.65	1.37
13.65	999.07		0.47		32.68	21.14	1.44	7.74	1.39
13.75		998.00		0.50	33.19	21.46	1.46	7.84	1.41
13.85	999.56		0.49		33.67	21.78	1.48	7.94	1.43
14.51		1001.4		3.42	37.09	23.99	1.64	8.01	1.45

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.5. Soybeans (18%) – Replication No. 1

Start date and time:	02/16/2017 10:00	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	03/03/2017 13:30	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	15	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	18.27 ± 0.0573	m_{soy} (g) =	1820.00
$\overline{w_{soy,1}}$ (%) =	18.40 ± 0.0011	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	334.96
$\overline{w_{soy,2}}$ (%) =	17.78 ± 0.0015	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1485.04

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	933.44	972.81			0.00	0.00	0.00		
0.10	933.44		0.00		0.00	0.00	0.00		
0.21		972.81		0.00	0.00	0.00	0.00		
0.31	933.44		0.00		0.00	0.00	0.00		
0.90		972.82		0.01	0.01	0.01	0.00		
0.96	933.45		0.00		0.01	0.01	0.00		
1.21		972.83		0.01	0.03	0.02	0.00		
1.29	933.46		0.01		0.04	0.03	0.00		
2.00		972.85		0.02	0.06	0.04	0.00		
2.13	933.47		0.01		0.07	0.05	0.00		
2.25		972.86		0.01	0.08	0.05	0.00		
2.96	933.48		0.01		0.09	0.06	0.00		
3.08		972.86		0.00	0.09	0.06	0.00		
3.17	933.49		0.01		0.10	0.07	0.00		
3.29		972.87		0.01	0.10	0.07	0.00		
3.98	933.70		0.22		0.32	0.22	0.01		
4.06		972.89		0.02	0.34	0.23	0.02		

Table B.5. Continued

4.13	933.73		0.03		0.37	0.25	0.02		
4.21		972.91		0.02	0.39	0.26	0.02		
4.29	933.75		0.02		0.41	0.28	0.02		
4.38		972.93		0.02	0.43	0.29	0.02		
4.93	934.20		0.45		0.88	0.59	0.04		
5.02		972.97		0.04	0.92	0.62	0.04		
5.09	934.64		0.44		1.36	0.91	0.06		
5.17		973.00		0.03	1.39	0.94	0.06	0.08	0.06
5.25	935.13		0.49		1.88	1.27	0.09	0.16	0.09
5.42		973.52		0.52	2.40	1.62	0.11	0.33	0.11
5.94	938.62		3.49		5.89	3.97	0.27	0.85	0.27
6.02		974.05		0.53	6.42	4.32	0.29	0.93	0.29
6.17	940.14		1.51		7.93	5.34	0.36	1.08	0.36
6.28		974.16		0.11	8.04	5.42	0.37	1.19	0.37
6.94	946.23		6.10		14.14	9.52	0.65	1.85	0.65
7.07		975.28		1.12	15.26	10.28	0.70	1.98	0.70
7.19	947.34		1.11		16.37	11.02	0.75	2.10	0.75
7.25		975.33		0.05	16.42	11.06	0.75	2.16	0.75
7.96	953.48		6.14		22.56	15.19	1.04	2.87	1.04
8.07		976.27		0.94	23.50	15.82	1.08	2.98	1.08
8.37	954.38		0.90		24.40	16.43	1.12	3.28	1.12
9.00		976.50		0.23	24.63	16.59	1.13	3.91	1.13
9.17	955.28		0.90		25.53	17.19	1.17	4.08	1.17
10.17		989.70		13.20	38.73	26.08	1.78		
10.29	956.16		0.88		39.62	26.68	1.82		
10.93		992.91		3.21	42.82	28.84	1.97		
11.01	956.63		0.47		43.29	29.15	1.99		
11.13		993.75		0.84	44.13	29.72	2.03		

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.6. Soybeans (18%) – Replication No. 2

Start date and time:	03/07/2017 13:05	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	03/20/2017 18:00	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	13	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	18.63 ± 0.1527	m_{soy} (g) =	1800.00
$\overline{w_{soy,1}}$ (%) =	18.51 ± 0.0015	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	333.16
$\overline{w_{soy,2}}$ (%) =	17.88 ± 0.0051	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1466.84

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	959.09	944.41			0.00	0.00	0.00		
0.08	959.09		0.00		0.00	0.00	0.00		
0.17		944.41		0.00	0.00	0.00	0.00		
0.85	959.09		0.00		0.00	0.00	0.00		
0.94		944.41		0.00	0.00	0.00	0.00		
1.01	959.10		0.01		0.01	0.01	0.00		
1.10		944.42		0.01	0.02	0.01	0.00		
1.18	959.11		0.01		0.03	0.02	0.00		
1.27		944.43		0.01	0.03	0.02	0.00		
1.80	959.15		0.04		0.08	0.05	0.00		
1.88		944.44		0.01	0.09	0.06	0.00		
1.97	959.16		0.01		0.10	0.07	0.00		
2.06		944.44		0.00	0.10	0.07	0.00		
2.15	959.17		0.01		0.10	0.07	0.00		
2.81		944.49		0.05	0.16	0.11	0.01		
2.89	959.17		0.00		0.16	0.11	0.01		
2.98		944.50		0.01	0.17	0.12	0.01		
3.10	959.18		0.01		0.18	0.12	0.01		
3.79		944.59		0.09	0.27	0.18	0.01		
3.87	959.19		0.01		0.28	0.19	0.01		
3.98		944.62		0.03	0.31	0.21	0.01		
4.08	959.20		0.01		0.32	0.22	0.01		
4.73		947.01		2.39	2.71	1.85	0.13		
4.79	959.41		0.21		2.92	1.99	0.14	0.06	0.14
4.87		947.97		0.96	3.88	2.65	0.18	0.14	0.18
4.97	959.66		0.25		4.13	2.82	0.19	0.24	0.19
5.07		949.17		1.20	5.33	3.63	0.25	0.34	0.25
5.14	960.11		0.45		5.78	3.94	0.27	0.41	0.27

Table B.6. Continued

5.94		949.32		0.15	5.93	4.04	0.28	1.21	0.28
6.02	960.92		0.81		6.74	4.59	0.31	1.29	0.31
6.09		949.82		0.50	7.24	4.94	0.34	1.36	0.34
6.21	962.11		1.19		8.43	5.75	0.39	1.48	0.39
6.30		950.51		0.69	9.12	6.22	0.42	1.57	0.42
6.82	968.11		6.00		15.13	10.31	0.70	2.09	0.70
6.89		950.94		0.43	15.55	10.60	0.72	2.16	0.72
6.97	969.13		1.02		16.57	11.30	0.77	2.24	0.77
7.06		951.79		0.85	17.43	11.88	0.81	2.33	0.81
7.14	969.49		0.36		17.78	12.12	0.83	2.41	0.83
7.23		952.10		0.31	18.09	12.33	0.84	2.50	0.84
7.86	977.46		7.97		26.06	17.77	1.21	3.14	1.21
7.91		952.50		0.40	26.46	18.04	1.23	3.18	1.23
8.07	978.12		0.66		27.12	18.49	1.26	3.34	1.26
8.12		953.07		0.57	27.69	18.88	1.29	3.39	1.29
8.20	978.34		0.22		27.91	19.03	1.30	3.47	1.30
8.28		953.67		0.60	28.51	19.44	1.33	3.55	1.33
8.86	978.52		0.18		28.69	19.56	1.33	4.14	1.33
8.99		954.30		0.63	29.32	19.99	1.36	4.26	1.36
9.08	979.15		0.64		29.95	20.42	1.39	4.35	1.39
9.18		955.95		1.65	31.60	21.55	1.47	4.45	1.47
9.87	984.02		4.87		36.47	24.86	1.70		
9.95		956.16		0.21	36.68	25.00	1.70		

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.7. Soybeans (18%) – Replication No. 3

Start date and time:	03/23/2017 16:45	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	04/02/2017 18:00	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	10	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	18.13 ± 0.0577	m_{soy} (g) =	1815.00
$\overline{w_{soy,1}}$ (%) =	18.26 ± 0.0012	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	331.36
$\overline{w_{soy,2}}$ (%) =	18.82 ± 0.0015	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1483.64

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	939.77	937.34			0.00	0.00	0.00		

Table B.7. Continued

0.64	939.77		0.00		0.00	0.00	0.00		
0.72		937.34		0.00	0.00	0.00	0.00		
0.90	939.78		0.01		0.01	0.01	0.00		
0.98		937.35		0.01	0.02	0.01	0.00		
1.06	939.79		0.01		0.03	0.02	0.00		
1.65		937.35		0.00	0.03	0.02	0.00		
1.73	939.81		0.02		0.05	0.03	0.00		
1.81		937.36		0.01	0.06	0.04	0.00		
1.91	939.82		0.01		0.07	0.05	0.00		
1.97		937.37		0.01	0.08	0.06	0.00		
2.07	939.83		0.01		0.09	0.06	0.00		
2.64		937.58		0.21	0.30	0.20	0.01		
2.72	939.86		0.03		0.33	0.22	0.02		
2.80		937.62		0.04	0.37	0.25	0.02		
2.90	939.87		0.02		0.38	0.26	0.02		
3.05		937.68		0.06	0.44	0.30	0.02		
3.68	940.30		0.42		0.86	0.58	0.04		
3.84		937.72		0.05	0.91	0.61	0.04		
3.92	940.32		0.03		0.94	0.63	0.04		
4.02		937.77		0.05	0.98	0.66	0.045		
4.07	940.39		0.07		1.05	0.71	0.048	0.06	0.05
4.65		938.75		0.98	2.03	1.37	0.09	0.63	0.09
4.73	940.51		0.12		2.15	1.45	0.10	0.71	0.10
4.82		938.90		0.15	2.30	1.55	0.11	0.81	0.11
4.90	940.63		0.12		2.42	1.63	0.11	0.88	0.11
4.97		939.09		0.19	2.61	1.76	0.12	0.96	0.12
5.06	940.80		0.17		2.78	1.87	0.13	1.04	0.13
5.64		942.30		3.21	5.99	4.04	0.28	1.63	0.28
5.73	940.96		0.16		6.15	4.15	0.28	1.72	0.28
5.89		942.54		0.24	6.39	4.31	0.29	1.87	0.29
5.98	941.18		0.21		6.61	4.45	0.30	1.96	0.30
6.05		942.83		0.29	6.89	4.65	0.32	2.03	0.32
6.73	945.78		4.60		11.50	7.75	0.53	2.71	0.53
6.84		943.16		0.34	11.83	7.98	0.54	2.83	0.54
6.94	946.10		0.32		12.15	8.19	0.56	2.92	0.56
7.03		943.61		0.45	12.60	8.49	0.58	3.01	0.58
7.69	950.02		3.92		16.52	11.13	0.76	3.68	0.76
7.76		944.48		0.87	17.39	11.72	0.80	3.74	0.80
7.84	951.01		0.99		18.38	12.39	0.84	3.83	0.84

Table B.7. Continued

7.94		945.48		1.00	19.39	13.07	0.89	3.92	0.89
8.01	951.99		0.98		20.36	13.73	0.94	4.00	0.94
8.64		950.56		5.08	25.44	17.15	1.17	4.62	1.17
8.72	952.52		0.53		25.98	17.51	1.19	4.70	1.19
8.81		951.86		1.30	27.27	18.38	1.25	4.79	1.25
8.89	953.64		1.11		28.39	19.13	1.30	4.87	1.30
9.06		952.94		1.08	29.47	19.86	1.35	5.05	1.35
9.65	958.36		4.72		34.19	23.05	1.57		
9.72		954.23		1.29	35.48	23.91	1.63		

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.8. Soybeans (18%) – Replication No. 4

Start date and time:	04/11/2017 16:45	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	4/19/2017 12:25	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	9	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\widehat{w}_{soy,1}$ (%) =	18.60 ± 0.1000	m_{soy} (g) =	1898.88
$\overline{w_{soy,1}}$ (%) =	18.80 ± 0.0009	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	357.03
$\overline{w_{soy,2}}$ (%) =	18.50 ± 0.0008	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1541.85

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	933.68	940.22			0.00	0.00	0.000		
0.72	934.71		1.03		1.03	0.67	0.045		
0.80		940.34		0.12	1.15	0.74	0.051	0.08	0.05
0.88	936.34		1.63		2.78	1.80	0.123	0.16	0.12
0.97		940.34		0.00	2.78	1.80	0.123	0.25	0.12
1.07	937.23		0.89		3.66	2.38	0.162	0.35	0.16
1.67		940.48		0.14	3.80	2.47	0.168	0.95	0.17
1.77	938.10		0.87		4.67	3.03	0.207	1.04	0.21
1.85		940.51		0.04	4.71	3.05	0.208	1.13	0.21
1.93	938.33		0.24		4.95	3.21	0.219	1.21	0.22
2.76		944.00		3.49	8.43	5.47	0.373	2.03	0.37
2.83	938.90		0.56		9.00	5.83	0.398	2.11	0.40
2.91		944.52		0.52	9.52	6.17	0.421	2.19	0.42
2.97	939.12		0.22		9.74	6.31	0.431	2.25	0.43
3.06		944.74		0.22	9.96	6.46	0.440	2.34	0.44
3.65	943.13		4.02		13.97	9.06	0.618	2.93	0.62

Table B.8. Continued

3.73		944.92		0.18	14.15	9.18	0.626	3.01	0.63
3.85	943.85		0.72		14.87	9.64	0.657	3.13	0.66
3.93		945.25		0.33	15.20	9.86	0.672	3.21	0.67
4.00	944.18		0.33		15.53	10.07	0.687	3.28	0.69
4.74		950.47		5.22	20.75	13.46	0.918	4.02	0.92
4.86	944.81		0.63		21.38	13.87	0.945	4.14	0.95
4.94		950.94		0.47	21.85	14.17	0.966	4.22	0.97
5.01	945.33		0.52		22.37	14.51	0.989	4.29	0.99
5.68		954.46		3.52	25.89	16.79	1.145	4.96	1.14
5.79	945.80		0.47		26.36	17.10	1.166	5.07	1.17
5.87		954.72		0.26	26.62	17.27	1.177	5.15	1.18
6.64	951.77		5.97		32.59	21.14	1.441	5.91	1.44
6.72		955.32		0.60	33.19	21.53	1.468	6.00	1.47
6.82	952.48		0.71		33.90	21.99	1.499		
6.98		955.84		0.52	34.42	22.32	1.522		
7.07	953.73		1.25		35.67	23.13	1.577		

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.9. Soybeans (18%) – Replication No. 5

Start date and time:	04/24/2017 16:15	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	05/03/2017 15:45	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	9	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\hat{w}_{soy,1}}$ (%) =	18.17 ± 0.0577	m_{soy} (g) =	1780.00
$\overline{w_{soy,1}}$ (%) =	18.40 ± 0.0006	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	327.49
$\overline{w_{soy,2}}$ (%) =	18.11 ± 0.0013	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1452.51

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	934.21	936.37			0.00	0.00	0.00		
0.66	934.23		0.02		0.02	0.01	0.00		
0.74		936.37		0.00	0.02	0.01	0.00		
0.83	934.24		0.01		0.03	0.02	0.00		
0.91		936.37		0.00	0.03	0.02	0.00		
0.99	934.24		0.00		0.03	0.02	0.00		
1.10		936.38		0.01	0.04	0.03	0.00		
1.67	934.26		0.02		0.06	0.04	0.00		

Table B.9. Continued

1.74		936.39		0.01	0.07	0.05	0.00		
1.88	934.27		0.01		0.08	0.06	0.00		
1.95		936.40		0.01	0.09	0.06	0.00		
2.04	934.28		0.02		0.10	0.07	0.00		
2.66		936.43		0.03	0.13	0.09	0.01		
2.74	934.29		0.01		0.14	0.10	0.01		
2.83		936.44		0.01	0.15	0.10	0.01		
2.91	934.30		0.01		0.16	0.11	0.01		
2.99		936.46		0.02	0.18	0.12	0.01		
3.08	934.32		0.02		0.20	0.14	0.01		
3.66		936.87		0.41	0.61	0.42	0.03		
3.75	934.38		0.06		0.67	0.46	0.03		
3.82		936.95		0.08	0.75	0.52	0.04		
3.91	934.49		0.11		0.86	0.59	0.04		
3.99		937.05		0.10	0.96	0.66	0.045		
4.08	934.62		0.13		1.08	0.75	0.051		
4.65		938.00		0.96	2.04	1.40	0.096	0.57	0.10
4.70	934.76		0.15		2.19	1.51	0.103	0.62	0.10
4.78		938.20		0.20	2.38	1.64	0.112	0.70	0.11
4.88	934.87		0.11		2.49	1.72	0.117	0.80	0.12
5.05		938.36		0.16	2.65	1.83	0.125	0.97	0.12
5.67	937.93		3.06		5.71	3.93	0.268	1.59	0.27
5.75		938.58		0.22	5.93	4.08	0.278	1.67	0.28
5.84	938.16		0.23		6.16	4.24	0.289	1.75	0.29
5.93		938.98		0.40	6.56	4.51	0.308	1.85	0.31
6.09	938.50		0.34		6.90	4.75	0.324	2.01	0.32
6.66		944.19		5.21	12.11	8.34	0.568	2.57	0.57
6.74	938.90		0.40		12.51	8.61	0.587	2.66	0.59
6.82		944.58		0.39	12.90	8.88	0.606	2.74	0.61
6.93	939.17		0.27		13.18	9.07	0.619	2.85	0.62
7.01		945.04		0.46	13.64	9.39	0.640	2.93	0.64
7.66	946.89		7.72		21.35	14.70	1.002	3.58	1.00
7.74		946.14		1.10	22.45	15.46	1.054	3.66	1.05
7.82	948.71		1.82		24.27	16.71	1.139	3.74	1.14
7.92		947.14		1.00	25.27	17.40	1.186	3.84	1.19
8.00	950.00		1.29		26.56	18.29	1.247	3.91	1.25
8.09		948.48		1.34	27.90	19.21	1.310	4.01	1.31
8.69	959.10		9.10		37.00	25.47	1.74		
8.80		949.89		1.41	38.41	26.44	1.80		

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.10. Soybeans (22%) – Replication No. 1

Start date and time:	06/30/2017 17:45	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	07/06/2017 07:00	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	6	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	21.57 ± 0.0577	m_{soy} (g) =	1834.89
$\overline{w_{soy,1}}$ (%) =	22.08 ± 0.0007	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	405.22
$\overline{w_{soy,2}}$ (%) =	21.80 ± 0.0006	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1429.67

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	934.27	933.55			0.00	0.00	0.00		
0.61	934.69		0.42		0.42	0.29	0.02		
0.68		933.64		0.09	0.51	0.36	0.02		
0.78	934.81		0.12		0.63	0.44	0.03		
0.87		933.79		0.14	0.78	0.54	0.04		
0.98	934.90		0.09		0.87	0.61	0.04		
1.64		935.69		1.90	2.77	1.94	0.13	0.60	0.08
1.72	935.29		0.38		3.16	2.21	0.15	0.68	0.10
1.82		935.98		0.29	3.44	2.41	0.16	0.78	0.11
1.91	935.55		0.26		3.71	2.59	0.18	0.87	0.13
2.66		939.03		3.06	6.76	4.73	0.32	1.62	0.27
2.74	935.78		0.23		7.00	4.89	0.33	1.70	0.28
2.78		939.42		0.39	7.38	5.16	0.35	1.74	0.30
2.86	936.18		0.40		7.78	5.44	0.37	1.82	0.32
2.95		939.86		0.44	8.23	5.75	0.39	1.91	0.34
3.65	940.45		4.27		12.49	8.74	0.60	2.61	0.55
3.73		944.90		5.04	17.53	12.26	0.84	2.68	0.79
3.85	940.96		0.51		18.04	12.62	0.86	2.81	0.81
3.93		945.54		0.64	18.68	13.07	0.89	2.89	0.84
4.02	941.40		0.44		19.12	13.38	0.91	2.98	0.86
4.70		952.84		7.30	26.42	18.48	1.26	3.66	1.21
4.79	941.88		0.48		26.90	18.82	1.28	3.75	1.23
4.86		953.36		0.52	27.42	19.18	1.31	3.82	1.26
4.95	942.22		0.34		27.76	19.42	1.32	3.91	1.27
5.02		953.91		0.55	28.31	19.80	1.35	3.98	1.30
5.55	549.61		7.39		35.70	24.97	1.70	4.20	1.45

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.11. Soybeans (22%) – Replication No. 2

Start date and time:	07/06/2017 21:30	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	07/12/2017 23:10	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	6	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	21.67 ± 0.1155	m_{soy} (g) =	1772.72
$\overline{w_{soy,1}}$ (%) =	21.88 ± 0.0026	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	387.82
$\overline{w_{soy,2}}$ (%) =	21.47 ± 0.0012	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1384.90

t [d]	$(\overline{m_{RC}})_*$ [g] $*=A,t$	$*=B,t$	$\left(\sum m_{CO_2}\right)_*$ [g] $*=A$	$*=B$	$*=A+B$	$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
0.00	955.77	960.92			0.00	0.00	0.000		
0.42	956.30		0.53		0.53	0.38	0.026		
0.61		961.07		0.15	0.68	0.49	0.033		
0.77	956.44		0.14		0.82	0.59	0.040		
0.88		961.21		0.14	0.96	0.69	0.047		
1.48	957.89		1.45		2.41	1.74	0.119	0.58	0.07
1.56		961.56		0.35	2.76	1.99	0.14	0.66	0.09
1.73	958.31		0.42		3.18	2.29	0.16	0.83	0.11
1.81		961.80		0.24	3.42	2.47	0.17	0.91	0.12
1.90	958.62		0.31		3.73	2.69	0.18	1.00	0.13
2.44		965.41		3.61	7.34	5.30	0.36	1.55	0.31
2.54	958.92		0.30		7.64	5.51	0.38	1.64	0.33
2.60		965.73		0.32	7.96	5.75	0.39	1.71	0.34
2.70	959.29		0.37		8.33	6.01	0.41	1.81	0.36
2.81		966.36		0.63	8.96	6.47	0.44	1.91	0.39
3.46	965.05		5.76		14.72	10.63	0.72	2.56	0.67
3.54		966.86		0.50	15.22	10.99	0.75	2.64	0.70
3.69	965.67		0.62		15.84	11.44	0.78	2.80	0.73
3.81		967.52		0.66	16.50	11.91	0.81	2.91	0.76
3.90	966.13		0.46		16.96	12.24	0.83	3.00	0.78
4.48		972.68		5.16	22.12	15.97	1.09	3.58	1.04
4.57	966.39		0.26		22.38	16.16	1.10	3.67	1.05
4.65		972.95		0.27	22.65	16.36	1.12	3.75	1.07
4.74	966.91		0.52		23.17	16.73	1.14	3.85	1.09
4.81		973.52		0.57	23.74	17.14	1.17	3.91	1.12
5.45	970.99		4.08		27.82	20.09	1.37	4.55	1.32
5.52		973.96		0.44	28.26	20.40	1.39	4.62	1.34
5.60	971.40		0.41		28.67	**20.70**	1.41	4.71	1.36

Table B.11. Continued

5.70		974.27		0.31	28.98	20.92	1.43	4.80	1.38
5.80	971.68		0.28		29.25	21.12	1.44	4.90	1.39
5.88		974.72		0.45	29.71	21.45	1.46	4.98	1.41
5.97	972.25		0.57		30.28	21.86	1.49	5.07	1.44
6.07		975.23		0.51	30.79	22.23	1.52	5.11	1.45

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.12. Soybeans (22%) – Replication No. 3

Start date and time:	09/20/2017 19:30	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	09/26/2017 14:20	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	6	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	21.67 ± 0.0471	m_{soy} (g) =	1796.68
$\overline{w_{soy,1}}$ (%) =	21.78 ± 0.0024	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	391.35
$\overline{w_{soy,2}}$ (%) =	21.67 ± 0.0009	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1405.33

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*= A,t$	$*= B,t$	$*= A$	$*= B$	$*= A+B$				
0.00	932.42	931.48			0.00	0.00	0.00		
0.56	932.87		0.45		0.45	0.32	0.02		
0.65		931.60		0.12	0.58	0.41	0.03		
0.73	933.01		0.14		0.72	0.51	0.03		
0.81		931.73		0.13	0.85	0.60	0.04		
0.90	933.14		0.13		0.98	0.69	0.05		
1.54		934.98		3.25	4.23	3.01	0.21	0.64	0.16
1.60	933.55		0.41		4.64	3.30	0.22	0.70	0.17
1.68		935.37		0.39	5.02	3.57	0.24	0.77	0.19
1.77	933.89		0.34		5.36	3.81	0.26	0.86	0.21
1.89		935.74		0.37	5.73	4.08	0.28	0.99	0.23
2.53	938.30		4.41		10.14	7.22	0.49	1.62	0.44
2.61		936.23		0.49	10.63	7.57	0.52	1.71	0.47
2.67	938.80		0.50		11.13	7.92	0.54	1.77	0.49
2.78		936.68		0.45	11.58	8.24	0.56	1.87	0.51
2.86	939.27		0.48		12.06	8.58	0.58	1.95	0.53
2.94		937.19		0.51	12.57	8.94	0.61	2.03	0.56
3.55	943.06		3.79		16.35	11.64	0.79	2.64	0.74
3.60		937.67		0.48	16.83	11.98	0.82	2.70	0.77

Table B.12. Continued

3.69	943.55		0.49		17.32	12.33	0.84	2.78	0.79
3.77		938.13		0.46	17.78	12.65	0.86	2.86	0.81
3.90	944.12		0.57		18.35	13.06	0.89	2.99	0.84
3.98		938.68		0.55	18.90	13.45	0.92	3.07	0.87
4.55	948.01		3.89		22.79	16.22	1.11	3.65	1.06
4.63		939.25		0.57	23.36	16.62	1.13	3.72	1.08
4.70	948.57		0.56		23.92	17.02	1.16	3.80	1.11
4.79		939.80		0.55	24.47	17.41	1.19	3.89	1.14
4.91	949.16		0.59		25.06	17.83	1.22	4.00	1.17
5.52		944.47		4.67	29.73	21.16	1.44	4.61	1.39
5.62	949.71		0.55		30.28	21.55	1.47	4.71	1.42
5.71		944.99		0.52	30.80	21.92	1.49	4.80	1.44
5.78	950.18		0.47		31.28	22.26	1.52	4.82	1.45

[1]Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

Table B.13. Soybeans (22%) – Replication No. 4

Start date and time:	09/26/2017 20:30	M_{CO_2} (g mol^{-1}):	44.0
End date and time:	10/04/2017 09:00	$M_{C_6H_{12}O_6}$ (g mol^{-1}):	180.0
No. of days:	8	$mol\ C_6H_{12}O_6/mol\ CO_2$:	0.167
$\overline{\widehat{w}_{soy,1}}$ (%) =	21.53 ± 0.0577	m_{soy} (g) =	1795.32
$\overline{w_{soy,1}}$ (%) =	21.86 ± 0.0005	$m_{H_2O}\ (g) = \overline{w_{soy,1}} m_{soy} =$	392.46
$\overline{w_{soy,2}}$ (%) =	21.66 ± 0.0003	$m_{dm}(g) = m_{soy} - m_{H_2O} =$	1402.86

t [d]	$(\overline{m_{RC}})_*$ [g]		$\left(\sum m_{CO_2}\right)_*$ [g]			$\sum m_{CO_2,s}$ [g kg^{-1}]	DML [%]	t' [d]	DML' [%]
	$*=A,t$	$*=B,t$	$*=A$	$*=B$	$*=A+B$				
0.00	947.86	955.98			0.00	0.00	0.00		
0.53	948.39		0.53		0.53	0.38	0.03		
0.61		956.08		0.10	0.63	0.45	0.03		
0.69	948.49		0.10		0.73	0.52	0.04		
0.80		956.20		0.12	0.85	0.61	0.04		
0.85	948.60		0.11		0.96	0.68	0.047		
1.56		957.37		1.17	2.13	1.52	0.10	0.66	0.05
1.61	948.80		0.20		2.33	1.66	0.11	0.72	0.06
1.72		957.55		0.18	2.51	1.79	0.12	0.82	0.07
1.79	949.01		0.21		2.72	1.94	0.13	0.90	0.08
1.87		957.81		0.26	2.98	2.13	0.14	0.97	0.09

Table B.13. Continued

2.54	950.96		1.95		4.93	3.52	0.24	1.65	0.19
2.63		958.22		0.41	5.34	3.81	0.26	1.74	0.21
2.72	951.41		0.45		5.79	4.13	0.28	1.82	0.23
2.85		958.62		0.40	6.19	4.41	0.30	1.95	0.25
2.91	951.81		0.40		6.59	4.70	0.32	2.02	0.27
3.54		961.22		2.60	9.19	6.55	0.45	2.64	0.40
3.65	952.17		0.36		9.55	6.81	0.46	2.75	0.41
3.75		961.59		0.37	9.92	7.07	0.48	2.86	0.43
3.84	952.59		0.42		10.34	7.37	0.50	2.94	0.45
3.92		962.00		0.41	10.75	7.67	0.52	3.02	0.47
4.52	956.30		3.71		14.46	10.31	0.70	3.63	0.65
4.57		962.52		0.52	14.98	10.68	0.73	3.68	0.68
4.66	956.72		0.41		15.40	10.98	0.75	3.76	0.70
4.75		963.02		0.50	15.90	11.33	0.77	3.86	0.72
4.83	957.32		0.60		16.50	11.76	0.80	3.94	0.75
4.90		963.49		0.47	16.97	12.10	0.82	4.01	0.77
5.50	960.75		3.43		20.40	14.54	0.99	4.60	0.94
5.58		964.09		0.60	21.00	14.97	1.02	4.69	0.97
5.67	961.45		0.70		21.70	15.47	1.05	4.77	1.00
5.75		964.69		0.60	22.30	15.90	1.08	4.86	1.03
5.85	962.05		0.60		22.90	16.32	1.11	4.96	1.06
6.52		968.85		4.16	27.06	19.29	1.32	5.63	1.27
6.58	962.66		0.61		27.67	19.72	1.34	5.69	1.29
6.67		969.46		0.61	28.28	20.16	1.37	5.78	1.32
6.75	963.21		0.55		28.83	20.55	1.40	5.86	1.35
6.83		970.06		0.60	29.43	20.98	1.43	5.94	1.38
6.92	963.80		0.59		30.02	21.40	1.46	6.02	1.41
7.00		970.64		0.58	30.60	21.81	1.49	6.10	1.44
7.52	969.66		5.86		36.46	25.99	1.77	6.13	1.45

[1] Time and dry matter loss values were adjusted to $t' = t - t_{0.05}$ and $DML' = DML - 0.05$.

APPENDIX C. RESULTS OF STATISTICAL ANALYSES

C.1. Comparison of moisture contents

Table C.1. Student's t-test results for Δw_1: Two-sample assuming equal variances

Test parameter	$w = 14\%$		18%		22%	
	$\hat{w}_{soy,1}$[1]	$w_{soy,1}$	$\hat{w}_{soy,1}$	$w_{soy,1}$	$\hat{w}_{soy,1}$	$w_{soy,1}$
Mean	14.37	14.33	18.36	18.47	21.61	21.90
Variance	0.06	0.09	0.06	0.04	0.01	0.02
Observations	4	4	5	5	4	4
Pooled variance	0.07		0.05		0.01	
Hypothesized mean difference	0		0		0	
Degrees of freedom	6		8		6	
t-Stat	0.18		-0.81		-3.97	
$p(T \leq t)$ one-tail	0.430		0.220		0.004	
t-critical	1.943		1.860		1.943	
$p(T \leq t)$ two-tail	0.860		0.439		0.007	
t-critical	2.447		2.306		2.447	

Table C.2. Student's t-test results for $\Delta w_{1\to2}$: Two-sample assuming equal variances

Test parameter	$w = 14\%$		18%		22%	
	$w_{soy,1}$	$w_{soy,2}$	$w_{soy,1}$	$w_{soy,2}$	$w_{soy,1}$	$w_{soy,2}$
Mean	14.33	14.36	18.47	18.22	21.90	21.65
Variance	0.09	0.15	0.04	0.19	0.02	0.02
Observations	4	4	5	5	4	4
Pooled variance	0.12		0.12		0.02	
Hypothesized mean difference	0		0		0	
Degrees of freedom	6		8		6	
t-Stat	-0.12		1.19		2.68	
$p(T \leq t)$ one-tail	0.453		0.134		0.019	
t-critical	1.943		1.860		1.943	
$p(T \leq t)$ two-tail	0.905		0.268		0.036	
t-critical	2.447		2.306		2.447	

C.2. Comparison of time parameters

Table C.3. ANOVA: Comparison of lag times

Comparing among $\boldsymbol{w}$ with $\boldsymbol{t_{start} = t_{0.05}}$					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	26.2731	13.1366	4.26	0.0458
Error	10	30.8089	3.0809		
Corrected total	12	57.0820			
R-square	CV	RMSE	Mean	Critical value of t	
0.4603	57.9731	1.7552	3.0277	2.2281	
Comparisons significant at the 0.05 level are indicated by ***.					
Comparison	Difference	95% Confidence Limits			
14 vs. 18	0.681	-1.943	3.305		
14 vs. 22	3.398	0.632	6.163	***	
18 vs. 22	2.717	0.093	5.340	***	
Comparing among $\boldsymbol{w}$ with $\boldsymbol{t_{start} = t_{0.10}}$					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	25.8759	12.9379	4.01	0.0527
Error	10	32.2898	3.2290		
Corrected total	12	58.1659			
R-square	CV	RMSE	Mean	Critical value of t	
0.4449	52.6725	1.7969	3.41	2.2281	
Comparisons significant at the 0.05 level are indicated by ***.					
Comparison	Difference	95% Confidence Limits			
14 vs. 18	0.658	-2.028	3.344		
14 vs. 22	3.365	0.534	6.196	***	
18 vs. 22	2.707	0.021	5.393	***	
Comparing within 14% and $\boldsymbol{t_{0.05}}$ vs. $\boldsymbol{t_{0.10}}$					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	1	0.2665	0.2665	0.04	0.8465
Error	6	39.1129	6.5188		
Corrected total	7	39.3794			
R-square	CV	RMSE	Mean	Critical value of t	
0.0068	56.5179	2.5532	4.5175	3.4604	
Means with the same letter are not significantly different.					
Tukey grouping	Mean	N	J		
A	4.700	4	2		
A	4.335	4	1		

Table C.3. Continued

Comparing within 18% and $t_{0.05}$ vs. $t_{0.10}$

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	1	0.3763	0.3764	0.13	0.7317
Error	8	23.8862	2.9858		
Corrected total	9	24.2626			

R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.0155	44.9049	1.7279	3.8480	3.2612	2.5201

Means with the same letter are not significantly different.

Tukey grouping	Mean	N	J
A	4.042	5	2
A	3.654	5	1

Comparing within 22% and $t_{0.05}$ vs. $t_{0.10}$

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	1	0.3160	0.3160	19.00	0.0048
Error	6	0.0998	0.0166		
Corrected total	7	0.4158			

R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.7600	11.3491	0.1289	1.1362	3.4604	0.2231

Means with the same letter are not significantly different.

Tukey grouping	Mean	N	J
A	1.3350	4	2
B	0.9375	4	1

Table C.4. ANOVA: Comparison of time to reach 0.5% dry matter loss

Comparing among w, includes lag time

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	51.1722	25.5861	7.41	0.0106
Error	10	34.5493	3.4549		
Corrected total	12	85.7215			

R-square	CV	RMSE	Mean	Critical value of t
0.5970	32.2354	1.8587	5.7661	3.8768

Comparisons significant at the 0.05 level are indicated by ***.

Comparison	Difference	95% Confidence Limits		
14 vs. 18	2.332	-1.087	5.750	
14 vs. 22	5.053	1.450	8.655	***

Table C.4. Continued

18 vs. 22	2.721	-0.697	6.139		
Comparing among $\boldsymbol{w}$, excludes lag time, with $\boldsymbol{t_{start}} = \boldsymbol{t_{0.05}}$					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	10.3838	5.1919	10.72	0.0033
Error	10	4.8423	0.4842		
Corrected total	12	15.2261			
R-square	CV	RMSE	Mean	Critical value of t	
0.6819	23.3753	0.6958	2.9769	3.8768	
Comparisons significant at the 0.05 level are indicated by ***.					
Comparison	Difference	95% Confidence Limits			
14 vs. 18	1.9415	0.6619	3.2211	***	
14 vs. 22	1.9300	0.5812	3.2788	***	
18 vs. 22	0.0115	-1.2681	1.2911		
Comparing among $\boldsymbol{w}$, excludes lag time, with $\boldsymbol{t_{start}} = \boldsymbol{t_{0.10}}$					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	10.0145	5.0073	13.63	0.0014
Error	10	3.6734	0.3673		
Corrected total	12	13.6879			
R-square	CV	RMSE	Mean	Critical value of t	
0.7316	22.2135	0.6061	2.7285	3.8768	
Comparisons significant at the 0.05 level are indicated by ***.					
Comparison	Difference	95% Confidence Limits			
14 vs. 18	1.8990	0.7845	3.0135	***	
14 vs. 22	1.9050	0.7302	3.0798	***	
18 vs. 22	0.060	-1.1085	1.1205		
Comparing within 14%					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	43.5920	21.7960	6.44	0.0184
Error	9	30.4784	3.3865		
Corrected total	11	74.0705			
R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.5885	33.2975	1.8402	5.5267	3.9484	3.633
Means with the same letter are not significantly different.					
Tukey grouping	Mean	N	J		
A	8.218	4	1		
B	4.320	4	2		
B	4.045	4	3		

Table C.4. Continued

Comparing within 18%

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	43.9343	21.9671	25.61	< 0.001
Error	12	10.2942	0.8578		
Corrected total	14	54.2285			

R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.8102	26.6968	0.9262	3.4693	3.4693	1.5628

Means with the same letter are not significantly different.

Tukey grouping	Mean	N	J
A	5.8860	5	1
B	2.3760	5	2
B	2.1460	5	3

Comparing within 22%

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	2.2885	1.1442	4.49	0.0444
Error	9	2.2924	0.2547		
Corrected total	11	4.5809			

R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.4996	19.6823	0.5047	2.5642	3.9484	0.9964

Means with the same letter are not significantly different.

Tukey grouping	Mean	N	J
A	3.1650	4	1
A	2.3875	4	2
A	2.1400	4	3

C.3. Dry matter loss rates calculation

Table C.5. Linear regression results: Dry matter loss rates of soybeans at 14% moisture using $t_{start} = t_{0.05}$

Replication No.	1	2	3	4	Pooled
Regression Statistics					
Multiple R	0.9986	0.9922	0.9897	0.9984	0.9858
R square	0.9972	0.9844	0.9796	0.9969	0.9717
Adjusted R square	0.9791	0.9683	0.9617	0.9736	0.9672
Standard error	0.0429	0.1008	0.1107	0.0467	0.1344
Observations	56	63	57	44	220
ANOVA					
Degrees of freedom					
Regression	1	1	1	1	1
Residual	55	62	56	43	219
Total	56	63	57	44	220
Sum of squares					
Regression	35.9023	39.7693	32.8831	29.8807	135.8886
Residual	0.1011	0.6294	0.6858	0.0944	3.9530
Total	36.0034	40.3988	33.5689	29.9751	139.8417
Mean square					
Regression	35.9023	39.7693	32.8439	29.8807	135.8886
Residual	0.0018	0.0102	0.0126	0.0022	0.0181
F-statistic	19524	3917	2610	13606	7528
p-value	8.53 x 10^{-71}	4.71 x 10^{-57}	2.3 x 10^{-48}	2.19 x 10^{-54}	5.24 x 10^{-171}
Regression estimates[a]					
Slope (v_{DML} ± *S.E.*)	0.1342 ± 0.0010	0.1170 ± 0.0019	0.1188 ± 0.0023	0.1692 ± 0.0014	0.1280 ± 0.015
t-Stat	139.73	62.59	51.82	116.65	86.77
p-value	7.35 x 10^{-72}	9.50 x 10^{-58}	5.23 x 10^{-49}	1.98 x 10^{-55}	1.43 x 10^{-171}

[a] Intercept was forced through zero; *S.E.* = standard error.

Table C.6. Linear regression results: Dry matter loss rates of soybeans at 18% moisture using $t_{start} = t_{0.05}$

Replication No.	1	2	3	4	5	Pooled
Regression Statistics						
Multiple R	0.9961	0.9910	0.9843	0.9956	0.9689	0.9809
R square	0.9921	0.9821	0.9688	0.9911	0.9389	0.9622
Adjusted R square	0.9366	0.9464	0.9304	0.9566	0.8954	0.9543
Standard error	0.0654	0.1187	0.1292	0.0737	0.1739	0.1486
Observations	19	29	27	30	24	119
ANOVA						
Degrees of freedom						
Regression	1	1	1	1	1	1
Residual	18	28	26	29	23	128
Total	19	29	27	30	24	129
Sum of squares						
Regression	9.7132	21.6137	13.4747	17.5694	10.6740	71.9729
Residual	0.0770	0.3945	0.4337	0.1573	0.6952	2.8298
Total	9.7903	22.0083	13.9084	17.7267	11.3691	74.8027
Mean square						
Regression	9.7132	21.6137	13.4747	17.5694	10.6740	71.9729
Residual	0.0043	0.0141	0.0167	0.0054	0.0302	0.0221
F-statistic	2269	1534	808	3238	353	3255
p-value	1.54 x 10^{-19}	2.51 x 10^{-25}	1.49 x 10^{-20}	1.74 x 10^{-30}	4.88 x 10^{-15}	2.19 x 10^{-92}
Regression estimates[a]						
Slope	0.3041	0.2853	0.2266	0.2218	0.2563	0.2499
(v_{DML} ± *S.E.*)	± 0.0064	± 0.0073	± 0.0080	± 0.0039	± 0.0136	± 0.0044
t-Stat	47.64	39.16	28.42	56.90	18.79	57.05
p-value	2.15 x 10^{-20}	5.34 x 10^{-26}	4.15 x 10^{-21}	2.62 x 10^{-31}	1.87 x 10^{-15}	6.88 x 10^{-93}

[a] Intercept was forced through zero; *S.E.* = standard error.

Table C.7. Linear regression results: Dry matter loss rates of soybeans at 22% moisture using $\boldsymbol{t_{start} = t_{0.05}}$

Replication No.	1	2	3	4	Pooled
Regression Statistics					
Multiple R	0.9831	0.9961	0.9993	0.9899	0.9816
R square	0.9665	0.9922	0.9987	0.9800	0.9634
Adjusted R square	0.9165	0.9565	0.9602	0.9506	0.9544
Standard error	0.1506	0.0824	0.0317	0.1192	0.1643
Observations	21	29	27	35	112
ANOVA					
Degrees of freedom					
Regression	1	1	1	1	1
Residual	20	28	26	34	111
Total	21	29	27	35	112
Sum of squares					
Regression	16.0651	24.2972	19.8759	23.7184	79.1101
Residual	0.4533	0.1902	0.001	0.4827	2.9984
Total	13.5184	28.4874	19.9026	24.2011	82.1086
Mean square					
Regression	16.0650	24.2972	19.8756	23.7184	79.1101
Residual	0.0226	0.0068	0.0010	0.0143	0.0270
F-statistic	576	3577	19787	1670	2934
p-value	1.12×10^{-15}	3.10×10^{-30}	9.27×10^{-38}	7.61×10^{-30}	1.15×10^{-81}
Regression estimates[a]					
Slope (v_{DML} ± *S.E.*)	0.2975 ± 0.0124	0.2775 ± 0.0046	0.2913 ± 0.0021	0.2095 ± 0.0051	0.2528 ± 0.0047
t-Stat	24.008	59.81	140.66	40.87	54.12
p-value	3.22×10^{-16}	4.37×10^{-31}	5.31×10^{-39}	1.72×10^{-30}	1.27×10^{-81}

[a] Intercept was forced through zero; *S.E.* = standard error.

Table C.8. Linear regression results: Dry matter loss rates of soybeans at 14% moisture using $\boldsymbol{t_{start} = t_{0.10}}$

Replication No.	1	2	3	4	Pooled
Regression Statistics					
Multiple R	0.9984	0.9977	0.9871	0.9986	0.9875
R square	0.9967	0.9954	0.9744	0.9972	0.9753
Adjusted R square	0.9772	0.9769	0.9566	0.9740	0.9704
Standard error	0.0452	0.0556	0.1172	0.0414	0.1225
Observations	52	55	57	44	208
ANOVA					
Degrees of freedom					
Regression	1	1	1	1	1
Residual	51	54	56	43	207
Total	52	55	57	44	208
Sum of squares					
Regression	32.2224	36.3432	29.3578	29.9288	122.8579
Residual	0.1040	0.1667	0.7702	0.0738	3.1090
Total	32.3264	36.5099	30.1279	27.0026	125.9669
Mean square					
Regression	32.2224	36.3432	29.3578	26.9288	122.8579
Residual	0.0020	0.0031	0.0137	0.0017	0.01502
F-statistic	15799	11774	2135	15680	8180
p-value	3.35×10^{-64}	6.31×10^{-64}	1.08×10^{-45}	1.12×10^{-55}	8.95×10^{-168}
Regression estimates[a]					
Slope (v_{DML} ± *S.E.*)	0.1358 ± 0.0011	0.1339 ± 0.0012	0.1166 ± 0.0025	0.1724 ± 0.0014	0.1340 ± 0.015
t-Stat	125.69	108.51	46.20	125.22	90.44
p-value	3.08×10^{-65}	6.93×10^{-65}	2.78×10^{-46}	9.49×10^{-57}	2.28×10^{-168}

[a] Intercept was forced through zero; *S.E.* = standard error.

Table C.9. Linear regression results: Dry matter loss rates of soybeans at 18% moisture using $t_{start} = t_{0.10}$

Replication No.	1	2	3	4	5	Pooled
Regression Statistics						
Multiple R	0.9956	0.9914	0.9919	0.9933	0.9797	0.9795
R square	0.9912	0.9829	0.9839	0.9867	0.9599	0.9595
Adjusted R square	0.9245	0.9472	0.9422	0.9509	0.9123	0.9512
Standard error	0.07189	0.1109	0.0914	0.08718	0.1394	0.1526
Observations	16	29	25	29	22	122
ANOVA						
Degrees of freedom						
Regression	1	1	1	1	1	1
Residual	15	28	24	28	21	121
Total	16	29	25	29	22	122
Sum of squares						
Regression	8.6942	19.7948	12.2672	15.7503	9.7777	66.7295
Residual	0.0775	0.3445	0.2007	0.2128	0.4083	2.8286
Total	8.7717	20.1394	12.4679	15.9631	10.1860	69.5481
Mean square						
Regression	8.6942	19.7948	12.2673	15.7503	9.7777	66.7295
Residual	0.005	0.0123	0.0054	0.0076	0.0194	0.0233
F-statistic	1682	1609	2467	2072	503	2865
p-value	< 1 x 10^{-14}	< 1 x 10^{-14}	< 1 x 10^{-14}	< 1 x 10^{-14}	< 1 x 10^{-14}	< 1 x 10^{-14}
Regression estimates[a]						
Slope	0.3191	0.12977	0.2623	0.2135	0.3049	0.2633
(v_{DML} ± *S.E.*)	± 0.0078	± 0.0074	± 0.0068	± 0.0047	± 0.0136	± 0.004
t-Stat	41.01	40.11	38.30	45.52	22.42	53.52
p-value	8.05 x 10^{-17}	2.77 x 10^{-26}	4.91 x 10^{-23}	8.43 x 10^{-28}	3.7610^{-16}	4.34 x 10^{-86}

[a] Intercept was forced through zero; *S.E.* = standard error.

Table C.10. Linear regression results: Dry matter loss rates of soybeans at 22% moisture using $\boldsymbol{t_{start} = t_{0.10}}$

Replication No.	1	2	3	4	Pooled
Regression Statistics					
Multiple R	0.9864	0.9983	0.9994	0.9942	0.9871
R square	0.9731	0.9966	0.9989	0.9885	0.9744
Adjusted R square	0.9231	0.9609	0.9604	0.9591	0.9654
Standard error	0.1282	0.0519	0.0276	0.0859	0.1309
Observations	21	29	27	35	112
ANOVA					
Degrees of freedom					
Regression	1	1	1	1	1
Residual	20	28	26	34	111
Total	21	29	27	35	112
Sum of squares					
Regression	11.8882	22.2467	17.9422	21.6487	72.5002
Residual	0.3285	0.0755	0.0199	0.2512	1.9001
Total	12.2167	22.3222	17.9621	21.8999	74.4010
Mean square					
Regression	11.8882	22.2467	17.9422	21.6487	72.5002
Residual	0.0164	0.0027	0.0008	0.0074	0.0171
F-statistic	724	8249	23431	2930	4234
p-value	1.37×10^{-16}	4.13×10^{-35}	1.11×10^{-38}	8.21×10^{-34}	1.20×10^{-89}
Regression estimates[a]					
Slope (v_{DML} ± *S.E.*)	0.3220 ± 0.0119	0.2987 ± 0.0033	0.2946 ± 0.0019	0.2317 ± 0.004	0.2728 ± 0.0042
t-Stat	26.90	90.82	153.07	54.13	65.07
p-value	3.53×10^{-17}	3.85×10^{-36}	5.91×10^{-40}	1.41×10^{-34}	3.11×10^{-90}

[a] Intercept was forced through zero; *S.E.* = standard error.

Table C.11. ANOVA: Comparison of dry matter loss rates

Comparing among $\boldsymbol{w}$ with $\boldsymbol{t_{start}} = \boldsymbol{t_{0.05}}$

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	0.0459	0.0230	19.71	0.0003
Error	10	0.0117	0.0012		
Corrected total	12	0.0576			

R-square	CV	RMSE	Mean	Critical value of t
0.7976	15.2600	0.0341	0.2237	3.8768

Comparisons significant at the 0.05 level are indicated by ***.

Comparison	Difference	95% Confidence Limits		
14 vs. 18	-0.1241	-0.1868	-0.0613	***
14 vs. 22	-0.1340	-0.2002	-0.0678	***
18 vs. 22	-0.0100	-0.0727	0.0528	

Comparing among $\boldsymbol{w}$ with $\boldsymbol{t_{start}} = \boldsymbol{t_{0.10}}$

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	2	0.0572	0.02861	23.81	0.0002
Error	10	0.0131	0.0013		
Corrected total	12	0.0703			

R-square	CV	RMSE	Mean	Critical value of t
0.8221	15.1597	0.03622	0.2389	3.8768

Comparisons significant at the 0.05 level are indicated by ***.

Comparison	Difference	95% Confidence Limits		
14 vs. 18	-0.1405	-0.2071	-0.0739	***
14 vs. 22	-0.1475	-0.2177	-0.0773	***
18 vs. 22	-0.0070	-0.0736	0.0596	

Comparing within 14%

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	1	0.00005	0.00005	0.08	0.7835
Error	6	0.00328	0.00055		
Corrected total	7	0.00332			

R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.0136	17.0450	0.0233	0.1371	3.4604	0.0404

Means with the same letter are not significantly different.

Tukey grouping	Mean	N	J
A	0.1395	4	2
A	0.1348	4	1

Comparing within 18%

Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	1	0.0011	0.0011	0.74	0.4161
Error	8	0.0122	0.0015		

Table C.11. Continued

Corrected total	9	0.0133			
R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.0842	14.5092	0.0391	0.2694	3.2612	0.0570
Means with the same letter are not significantly different.					
Tukey grouping	Mean	N	J		
A	0.2800	5	2		
A	0.2588	5	1		
Comparing within 22%					
Source	DF	Sum of Squares	Mean Square	F-value	Pr > F
Model	1	0.0007	0.0007	0.43	0.5358
Error	6	0.0093	0.0015		
Corrected total	7	0.0099			
R-square	CV	RMSE	Mean	Critical value of t	Minimum significant difference
0.0670	14.1460	0.0393	0.2778		
Means with the same letter are not significantly different.					
Tukey grouping	Mean	N	J		
A	0.2870	4	2		
A	0.2688	4	1		

C.4. Minimum number of replications calculations

Table C.12. Iterative calculations of minimum number of replications

$w = 14\%$ $\left(\sigma_{v_{DML}}\right)_p = 0.0017$ % d^{-1} $\alpha = 0.05$ $j = 3w \times 1T = 3$

$\delta = 0.0070$ % d^{-1} $\beta = 0.80$

k	$\hat{r}$	df	t_1	t_2	r (calculated)	r (rounded)
1	6	10	2.2281	0.8791	1.2068	2
2	2	2	4.3027	1.0607	3.6000	4
3	4	6	2.4469	0.9057	1.4050	2
4	2	2	4.3027	1.0607	3.5956	4

$w = 18\%$ $\left(\sigma_{v_{DML}}\right)_p = 0.0083$ % d^{-1} $\alpha = 0.05$ $j = 3w \times 1T = 3$

$\delta = 0.0333$ % d^{-1} $\beta = 0.80$

k	$\hat{r}$	df	t_1	t_2	r (calculated)	r (rounded)
1	6	10	2.2281	0.8791	1.2068	2
2	2	2	4.3027	1.0607	3.6000	4
3	4	6	2.4469	0.9057	1.4050	2
4	2	2	4.3027	1.0607	3.5956	4

$w = 22\%$ $\left(\sigma_{v_{DML}}\right)_p = 0.0025$ % d^{-1} $\alpha = 0.05$ $j = 3w \times 1T = 3$

$\delta = 0.0098$ % d^{-1} $\beta = 0.80$

k	$\hat{r}$	df	t_1	t_2	r (calculated)	r (rounded)
1	6	10	2.2281	0.8791	1.2068	2
2	2	2	4.3027	1.0607	3.6000	4
3	4	6	2.4469	0.9057	1.4050	2
4	2	2	4.3027	1.0607	3.5956	4