

FEASIBILITY OF STORING CANOLA IN SILO BAGS (HARVEST BAGS)

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Abstract

Silo bags are a recently-developed temporary grain storage system that is becoming more widely used in Western Canada. Silo bag storage systems have been used on the Canadian Prairies to store cereal grains and oilseeds without any scientific information about the effect of changing conditions over time on seed quality. The main goal of this study was to examine the conditions that would allow safe storage of canola in these bags in the Canadian Prairie provinces. To achieve this objective, the effect of storage moisture content (by storing canola at 8.9, 10.5, and 14.4% moisture content for 40 weeks) and the effect of storage time (by storing canola at approximately 12% moisture content for 40 weeks and unloading three different times) on quality of canola during storage silo bags in were studied. A regression model was developed to predict CO₂ concentration inside silo bags containing canola, which can be used as an indicator of spoilage and the permeability of silo bag material to carbon dioxide and oxygen was also measured.

Canola at three different moisture contents (m.c.) 8.9, 10.5 and 14.4% (wet basis), which represent dry, straight and damp classifications, were stored in silo bags for 40 wk (from autumn 2010 to summer 2011) at Winnipeg, Manitoba to determine the quality changes of the stored canola. For each moisture content three bags were tested, and each bag was loaded with approximately 20 t canola with > 90% initial germination. Seed germination, free fatty acid value (FAV), and moisture content of canola at seven

locations in each silo bag were analysed every 2 wk along with carbon dioxide concentrations of intergranular air and temperature of canola. For dry grade canola (8.9 % m.c.), the germination was maintained above 90%, and FAV also stayed within 1.5 times the initial value during the 40-week storage. The germination of straight grade (10.5% m.c.) canola maintained its initial value in most parts of the silo bags, except at the top layer. However, the germination of damp (14.4% m.c.) canola dropped to below 80%, and FAV doubled its initial value within 8 wk of storage. High levels of CO₂ and localized hotspots in damp canola indicated intense biological activity. Canola, which was graded as Canada Grade 1 at the beginning of the storage, became Grade 1, Grade 2, and Feed Grade for the dry, straight and damp grade canola, respectively at the end of the storage.

Another study was conducted for two storage years (2011-12 and 2013-14) to determine the changes in grain quality while storing at around 12.0% moisture content (m.c., wet basis) canola in silo bags. Canola was stored in three silo bags (67 tonnes /bag) and unloaded at three different times (one bag at a time) which represent 20 wk of storage (unloaded in the middle of winter), 28 wk of storage (unloaded at the end of winter) and 40 wk of storage (unloaded after summer storage). Canola seed quality parameters (germination, FAV, and moisture content), and intergranular composition (CO₂ and O₂ levels) at different locations in silo bags were analysed every two weeks. Temperature of canola at various locations in the silo bags were recorded every 30 min for the duration of storage. The germination of canola at most parts of the silo bags stayed above a safe level up to the end of the winter season (20 wk of storage). At the top layer of the silo bags, germination of canola decreased to below 30% during summer storage (after 40 wk of

storage). Moisture content of canola increased at the top layer in both storage years. The FAV values remained within 1.5% of initial value until 20 wk of storage, and increased more than 2-fold of initial values after summer storage. The commercial grades after first, second and third unloading (after 20, 28 and 40 wk of storage) were Grade 1, Grade 2 and Feed Grade, respectively in year 1, whereas for year 2, these were Grade 1, Grade 1 and Grade 2, respectively. The grain quality analysis and commercial grading results indicated that ambient temperature had a major role in quality of canola during storage.

Carbon dioxide (CO₂) can be used as an indicator of incipient grain spoilage during storage because respiration of seeds and fungi produce CO₂. A polynomial regression model was developed using the data collected from the field study to predict CO₂ concentration inside a silo bag with canola. The initial moisture content of canola, monthly average temperature, and storage time were used as inputs for this model. The coefficient of determination (R² value) of this regression model was 0.76 and had a root mean square error (RMSE) value of 0.196. The standardized coefficients (β coefficient values) of independent variables of the model indicated that initial moisture content was 3.9 times more important than storage temperature for CO₂ concentration prediction. Validation of this model with the field study had an RMSE of 0.160. Predictability of this model was compared with previously developed CO₂ prediction models and our current model performed better than the other prediction models. The current model can be used to predict CO₂ concentration inside a silo bag with canola up to 200 days of storage.

Permeability to CO₂ and O₂ of the silo bag material was determined using a specially designed testing unit and the effects of storage environment conditions and stretching of silo bag while loading the grain on permeability of the material were also

analysed. The permeability of silo bag material to CO₂ was $21.61 \pm 1.50 \times 10^{-6} \text{ m}^3 \text{ m d}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$, and for O₂ was $1.95 \pm 0.36 \times 10^{-6} \text{ m}^3 \text{ m d}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$, at room temperature. The permeability for CO₂ and O₂ was affected by the storage environment and stretching.

The results from this study indicated that, dry (8% m.c.), straight (10% m.c.), and damp (14% m.c.) grade canola can be stored up to 40, 24, and 4 weeks, respectively after harvest without any quality loss under Canadian Prairie conditions. Canola with 12% moisture content could be stored up to 20 weeks (or up to the late winter) without any quality deterioration under the western Canadian conditions. Storing wet canola in the summer season will cause an increase in biological activity inside the silo bag, which increased the deterioration of the canola. Improvement of sealing techniques to maintain the airtightness of silo bag, development of a coupled three dimensional model to predict moisture content, temperature and CO₂, and testing the silo bags for storing cereal grains and pulses under Canadian Prairie conditions will help farmers to understand the adaptability of these silo bags on western Canadian farms.

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Chapter 1

Introduction

Canola is one of the major oilseed crops widely cultivated around the world, and Canada is the world's largest producer. The annual average production of canola in Canada is 9.4 million tonnes (StatsCanada, 2013). The prairie region (Manitoba, Saskatchewan and Alberta) accounts for nearly 99% of total area of canola production in Canada (StatsCanada, 2013). The recommended moisture content (m.c.) for storage of canola is 10%, wet basis (Mills, 1996). Numerous challenges occur during harvest and post-harvest storage of canola due to the small size of the seeds (Ochandio et al., 2010a). Respiration rate of freshly harvested canola is high, resulting in large amounts of carbon dioxide (CO₂) and heat (Mills, 1996). It requires 6 weeks to stop respiration, and then seeds enter a quasi-dormant stage (Thomas, 1984). If canola is harvested at more than 10% m.c. it will spoil rapidly, therefore it is recommended to dry the seeds as early as possible after harvest. Usually, farmers in the same region harvest grain at around the same time, and there is a lack of equipment to transport their product from the field to the storage site. Therefore, farmers are looking for new techniques to avoid the harvest delay

and reduce the transportation cost. At present, large harvest bags (silo bags) are becoming popular for on-field temporary storage of cereals and oilseeds (together referred to as grains) in several countries including Canada when harvest exceeds grain bin capacity.

The silo bag is a grain storage system, in which grain is stored in specialized plastic membrane bags with the help of dedicated grain loading and unloading equipment. This system has been adopted successfully to store grains in Latin American countries, especially in Argentina. Usually, these bags are 2.7 to 3.0 m in diameter, and 60.9 to 76.2 m long, and hold approximately 200 to 300 tonnes of grain depending on the grain type. The harvest bags are made of three-layer polyethylene membrane (235 μm thick), and provide a nearly air tight environment for the grains. The first two white layers act as UV filters and the third black layer keeps out the sunlight. These bags stretch up to 10% during the grain filling process. A silo bag can be filled at a rate up to 200 tonnes per hour using special equipment called a 'grain bag loader', and unloaded at 180 tonnes per hour using another specialized equipment called an 'extractor' or 'unloader'. Both ends of the silo bags are sealed, rain proofed, and might provide high level of air tightness. Because of this, the respirations of biotic components of the grain bulk (grain, insect and fungi) increase CO_2 and decrease O_2 concentration. This creates an unfavourable modified atmosphere for insects and moulds (Darby and Caddick, 2007).

Silo bag storage is probably a cost-effective method compared to other temporary storage systems, and the main advantage is the reduction of transportation cost during the harvest season to storage sites. Silo bags can also fill storage capacity gap. Silo bags can be placed in any part of the farm, but preparation of the storage site plays a major role in

the success of the silo bag storage systems. However, there are some concerns over seed spoilage, insect and mould damage, moisture migration, and quality losses. When compared to other storage systems, a high proportion of the stored bulk is held at the surface (peripheral) layer of the bag storage system. This peripheral layer undergoes large temperature and moisture changes during storage. Large temperature gradients will result in moisture migration and condensation. Moisture migration and accumulation of condensation inside the harvest bag can create localized seed spoilage. More than 18% of the stored bulk in silo bags in Australia had some quality changes (Darby and Caddick, 2007). They recommended a site with good drainage, free of sharp objects and accessibility throughout the year for a silo bag storage system. Australia does not have issues with accessibility but in western Canada deep snow and freezing and thawing of soil can cause serious accessibility issues.

Harvest bags have been tested for storing wheat, barley, corn, canola, soybean and many other crops under Argentinean weather conditions (Bartosik et al., 2002; Ochandio et al., 2010a; 2010b; Rodriguez et al., 2004; 2008). Bartosik et al. (2002) tested silo bags for the storage of wheat (12.5% and 16% m.c., wet basis). In their study, the germination of wet wheat dropped from 94 to 51% after 45 days of storage and was further reduced to 41% after 150 days of storage. Also, there were reductions in baking quality and increases in CO₂ concentration (22%) in wet wheat. Barley stored at dry moisture contents (11% and 11.5%, wet basis) in harvest bags for 5 months in Argentina had no significant effect in protein value and germination (Ochandio et al., 2010a).

Storage moisture and temperature play a major role in assessing safe storage condition of grains (Sathya et al., 2009; Nithya et al., 2011) in any storage structure.

Grain germination and free fatty acid value are the main quality parameters used to determine the quality of the most of the cereal grains and oilseeds during storage (Sathya et al., 2009). Grain temperature and moisture content changes during storage affect spoilage. Carbon dioxide concentration of intergranular air can be used as an indicator of grain quality (White et al., 1982). Ochandio et al. (2010) stored dry canola (6% moisture, wet basis) without any quality deterioration for 12 months in Argentina, but in another study, Rodriguez et al. (2004) reported significant change in free fatty acid value of high moisture sunflower seeds (16.4%, wet basis) in 160 days of storage in silo bags. Safe storage guidelines for wheat, rye and canola developed by various researchers (Nithya et al., 2011; Sathya et al., 2008; Sathya et al., 2009) also recommended short storage times for high moisture products.

In Canada, most canola is harvested from the field in late August or early September, and often harvest moisture content of canola is higher than safe storage moisture (>10% m.c.). Farmers dry the canola to safe storage moisture contents using a near ambient drying or heated air drying system and then store it in steel bins on the farm for 1 to 12 months (a maximum period until the next August). Farmers sell their canola whenever they can get a high market value, so the accessibility of silo bags throughout the year is important to unload the bags at any time of the year. The sweating effect (higher respiration up to 6 weeks after storage) of freshly harvested canola can cause significant damage in canola quality during storage. Since the silo bag storage is an on-field storage technique, farmers do not have the access to dryer to reduce the moisture content of the canola on the field. So, it is necessary to determine the maximum storage time in silo bags without any losses while loading the harvested canola directly

(approximately at 12% m.c.) into the silo bag without any prior drying. Unloading bags whenever abnormal conditions are found during storage (reduction in germination below safe level, sudden increase in CO₂ level or heating of seeds) may also help farmers to reduce the loss due to spoilage of canola and get maximum benefit from their product.

These silo bags are manufactured and tested with various grains only in Argentinean weather conditions, which are totally different from Canadian weather conditions, and farmers in Canada are using these bags to store their products without any scientific study. Therefore, there is a need for testing these bags under Canadian conditions to determine the changes in quality parameters during storage of canola at different moisture contents and maximum storage time for storing canola in these silo bags in order to reduce the quality and quantity losses.

Carbon dioxide is one of the reliable parameters that can be used as an indicator of incipient grain spoilage during storage. All living organisms in the grain storage system (grain, insects and fungi) release CO₂ and consume O₂ during the respiration process (White et al., 1989). Spoilage of grain caused by insects and mould increases the biological activity in a grain bulk, increases the CO₂ level, and this elevated level of CO₂ can be correlated with the spoilage of grain inside the grain storage structure. White et al. (1982) and Proynk et al. (2004) developed polynomial regression models to predict CO₂ production rates of wheat and canola using the storage temperature, moisture content of grain and storage time. Jian et al. (2014) estimated interstitial CO₂ and O₂ concentrations of wheat, soybean and canola stored at 10, 15, 20, 23, 25, 30, 35 and 40°C. They found at 10°C storage, CO₂ concentration was less than 5% in all moisture contents of canola, but at 40°C storage CO₂ concentration reached around 21% for 14% m.c. canola after 5 days

of storage. They correlated microfloral activity in the high moisture canola at high temperature storage to the increased level of CO₂.

Permeability of the packaging material is one of the major parameters that determine the success of modified and controlled atmospheric storage of grain and food materials. A silo bag is made of both, high density polyethylene (HDPE) and low density polyethylene (LDPE). Permeability of HDPE to CO₂ and O₂ at 25°C are $19.0 \times 10^{-8} \text{ m}^3 \text{md}^{-1} \text{m}^{-2} \text{atm}^{-1}$, and $6.5 \times 10^{-8} \text{ m}^3 \text{md}^{-1} \text{m}^{-2} \text{atm}^{-1}$, respectively. For LDPE, permeability to CO₂ and O₂ at 25°C are $105.0 \times 10^{-8} \text{ m}^3 \text{md}^{-1} \text{m}^{-2} \text{atm}^{-1}$, and $19.5 \times 10^{-8} \text{ m}^3 \text{md}^{-1} \text{m}^{-2} \text{atm}^{-1}$, respectively (Osborn and Jenkins, 1992). Abalone et al. (2011) assumed the silo bag material had 50% of HDPE and 50% of LDPE and calculated equivalent permeability for silo bag plastic material ($3.22 \times 10^{-7} \text{ m}^3 \text{md}^{-1} \text{m}^{-2} \text{atm}^{-1}$ to CO₂, and $9.75 \times 10^{-8} \text{ m}^3 \text{md}^{-1} \text{m}^{-2} \text{atm}^{-1}$ to O₂). Thus, the permeability of silo bag material has never been measured and the stretching of silo bag during loading of grain may affect the permeability of silo bag material. The energy of the gas molecules has strong correlation with the permeability rate of packaging materials (Delassus, 1997) and the temperature gradient between inner (grain) and outer (ambient air) parts of a silo bag can change the energy of gas molecules, which may alter the permeability rate. Measurement of permeability of silo bag material for CO₂ and O₂ as well as the effect of stretching and storage time on permeability rate can help to develop better mathematical models to predict the interstitial gas concentration in silo bags which can then be used to predict spoilage of grain during storage.

1.1. Objectives

Based on the current literature, it is clear that there is lack of knowledge about the suitability of a silo bag storage system for Canadian Prairie conditions, quality changes of grain during bag storage, permeability of the bag material and the effect of stretching and duration of storage on permeability; and temperature, moisture and intergranular gas composition changes in bag storage system. An extensive study to determine the feasibility of using silo bags was proposed with the following objectives to avoid the potential quality and quantity losses of grain while storing canola under Canadian Prairie weather conditions:

1. To determine the feasibility of using silo bags to store canola at three moisture contents under Canadian Prairie conditions by monitoring changes in seed quality parameters (germination and FAV), intergranular gas concentration, and seed temperature;
2. To determine the effect of storage time of 12% m.c. canola on grade and quality by monitoring changes in seed quality parameters (germination and FAV), intergranular gas concentration, and seed temperature while storing, and unloading at three different times: (i) middle of winter (about 20-24 weeks storage), (ii) early spring (about 28-32 weeks storage) and (iii) end of summer storage (40 weeks storage);
3. To develop a prediction model to predict CO₂ levels inside silo bag and validate it using field data collected from objectives 1 and 2; and

4. To determine the permeability of the new silo bag material for CO₂ and O₂ and material exposed to environment for 7 and 10 months.

Chapter 2

Review of literature

2.1. Storage of canola

Canola is the major oilseed crop grown in Canada and the canola industry contributes nearly 15.4 billion dollars/year to the Canadian economy (CCC, 2013). Due to the higher market value than for cereals, the canola production area increased almost 150% in 10 years (3.6 million ha in 2002 to 8.5 million ha in 2012). Canola production also increased almost 3 times in these 10 years (4.5 million tonnes in 2002 to 13.3 million tonnes in 2012) (StatsCanada, 2013). Canola contain around 42-45% oil, 22-24% protein and 32-34% carbohydrates (Ratnayake and Daun, 2009). The sudden boom in canola production also resulted in an increase in canola crushing facilities across Canada. Canola oil accounts for nearly 75% of the vegetable oil market in Canada (CCC, 2013). Consumers prefer canola oil over the other vegetable oils because of its very low saturated fatty acid level (7%). Canola oil also contains high level (61% of fatty acid content) of monounsaturated fatty acid (MUFA) such as oleic acid, which reduces low-

density lipoprotein (LDL) cholesterol (unhealthy cholesterol) levels in the human body. Low level saturated fatty acid also helps to increase the long-chain omega3 fatty acid (eicosapentaenoic acid (EPA)) content. After the extraction of oil from the canola, the high protein canola meal is used as animal feed (Ratnayake and Daun, 2009).

Most of the farmers in the Canadian Prairie region start their canola seeding by early May and harvesting starts in the middle of August (late summer). The moisture level and temperature of the grain influences events that occur during storage and may lead to spoilage and self-heating. Canola and other high oil seeds are more prone to deterioration in storage than cereal grains and must be stored at a lower moisture level than cereals to prevent moulding. Straight grade moisture content for canola is 10% (wet basis) (CGC, 2014). Therefore, farmers store canola at around 10% moisture to avoid a weight penalty while selling the seeds; thus 10% m.c. is often considered as a safe storage moisture for canola (Moysey and Norum, 1975). Below 10% m.c. mould activity is low at Canadian storage conditions but some mould species can grow at higher grain temperatures (30 to 40°C), even when the moisture content of canola is less than 8% (Sathya et al., 2009). The oil fraction in the canola does not absorb moisture so moisture is held in the fiber, protein and starch fractions of the seed, therefore, the equilibrium moisture level for canola is lower than that of cereal grains at the same relative humidity (Thomas, 1984). Thus, the safe storage moisture for canola is lower than wheat and other cereal grains to avoid deterioration of seeds during storage due to mould growth. The temperature and moisture gradients inside the bins play a major role in the rate of deterioration of canola. To avoid these temperature and moisture gradients, the bins should be aerated once loaded with freshly harvested canola. The temperature of canola

after swathing and combining is 8°C warmer than the ambient air temperature (Prasad et al., 1978). This temperature difference results in moisture migration between warmer and cold regions of the bin, and the rewetting of canola happens even in low moisture canola (8% m.c.) when the seeds are loaded into the bins at high temperatures and not cooled immediately with the aeration systems (Frisen and Huminicki, 1986). This creates adverse effects rapidly. Post-harvest ripening and respiration of freshly harvested canola (sweating effect) make the farmer to use different storage management practices for canola. Generally, this sweating effect continues up to 6 to 8 weeks after harvesting, and then seeds go into a semi-dormant stage if the storage moisture and temperature are low (Mills, 1989). Mould growth and heating of canola produce a distinct odour, and reduce the market value of the seed because the oil and meal produced from these heated seeds still has this odour and discolouration, and it is hard to remove it by further processing (Thomas, 1984). Mites and moulds are the major reasons for deterioration of canola during storage. *Aspergillus glaucus* group, *Aspergillus candidus* Link, and *Penicillium* spp. are the major fungal species and *Acarus* and *Glycyphagus* spp. are the predominant mites that attack canola during storage (Armitage, 1984). Mites prefer moist and mouldy seeds, and reduce the seed weight and quality; and may produce a distinct odour (Mills, 1989). Mite infestation in canola is negligible before any fungal infection (Pronyk et al., 2004). So identification and controlling fungal infection is the most important management practice during canola storage. Burrell et al. (1980) reported that, rapeseeds with 10.6% m.c. stored at 25°C clumped together after 11 days of storage, and visible moulds were noticed after 21 days of storage. They recommended that, clumping or caking of canola is the best indicator for deterioration, so the freshly harvested canola

should be dried below 10% m.c. within 2 weeks. The conditioning of canola can be divided into three categories: aeration, near-ambient and heated-air drying. Aeration is used to cool the canola, by which the moisture migration can be eliminated. Near-ambient and heated-air drying systems are used to reduce the moisture content of the canola to recommended storage moisture. Canola can be dried at up to 82°C, if the seeds are used for oil extraction and the maximum drying temperature for seeding purposes is 45°C (Thomas, 1984). The Canola Council of Canada recommends western Canadian farmers to start aeration systems right after loading canola into bins to reduce the effect of sweating and post-maturation (CCC, 2013). The drying practices also have to be readjusted for canola because of the higher airflow resistance of canola than cereals, which decreases airflow because the fan is working against higher static pressure and increases drying time.

The quality of canola can be determined by moisture content, appearance, smell of crushed seeds, free fatty acid value (FAV), colour of the extracted oil, seed germination, visible mould and mite infestation. In Canada, canola is graded using numerical grade by visual and laboratory assessment (Daun and Burch, 1984). Immaturity of the seeds (green seeds), heated seeds, weathering, and admixture are the main grading factors used for canola grading. Table 1 explains the Canadian canola grading standards (CGC, 2014).

Ergosterol, the main sterol found in fungal bodies was also used to determine the deterioration level of canola. Total ergosterol level increased with storage time, temperature, and storage moisture (Pronyk et al., 2006). Ergosterol level increased rapidly in 14% m.c. canola due to the higher water activity and the higher amount of

fungal growth in high moisture seeds. The CO₂ production and ergosterol production had a positive correlation.

2.2. Factors affecting seed quality during storage

All types of grain storage structures (grain bins, silo bags and bunkers) can be considered as ecosystems, and the interactions of the biotic (grain, insects, and mould) and abiotic (temperature, relative humidity, solar radiation, gas concentration) factors of these ecosystems play a major role in quality changes of grain during storage (Sinha, 1973). Mould infection is the most common reason for spoilage of canola seed during storage. Mould growth increases dramatically and rapid deterioration of canola happens over certain limits of moisture content and temperature (Wallace, 1983). Jayas and White (2003) ranked canola moisture content and temperature as the first and second most important factors affecting the seed quality during storage. Germination and the free fatty acid value are the two most common factors used to determine the spoilage of grain during storage (Nithya et al., 2012; Pomeranz, 1992; Sathya et al., 2009; Sun et al., 2014; White and Jayas, 1991). Mould infection and insect infestation in grain bulks affect the germ portion of the seed, which reduces the viability of the seeds (Muir, 2001). Decrease of germination below 90% of the seed's initial germination is considered as indication of grain spoilage during storage (Karunakaran et al., 2001; Schroth et al., 1998). Breakdown of lipids in the seed by the hydrolysis process from the enzymatic secretions from the mould activity in the grain bulk produces the free fatty acids (Sathya et al., 2009). Deterioration of grain due to mould activity increases the FAV and increase of FAV by

1.5-fold of initial value was used as an indicator of grain quality deterioration (White and Jayas, 1991).

Deterioration of wheat at five moisture contents (15, 16, 17, 18 and 19%, wet basis) stored at different temperature regimes was studied by Karunakaran et al. (2001), and they found that there were no changes in germination of 15 and 16% moisture content wheat stored at 25°C for 70 days. But in high moisture (17, 18 and 19%) wheat, germination decreased with the increase in storage time. They also noticed visible mould in the high moisture wheat stored at 25°C. Sathya et al. (2009) stored canola with four moisture contents (7.5, 10.0, 12.5, and 15.0%, wet basis at 10, 20, 30 and 40°C) for 16 weeks to test the quality changes (in environmental chambers with 50±5% r.h.). They found germination of dry canola (7.5 and 10% moisture content) remained above 80% after 16 weeks of storage when stored at 10°C, and only 7.5% moisture canola had germination above 80% after 16 weeks of storage when stored at 20°C. For high moisture canola (12.5 and 15.0%), germination dropped below 80% after 3 weeks of storage at 10°C. At high temperature storage (30 and 40°C), germination dropped below 80% in all the samples after 4 weeks of storage. They also found increase in FAV value with increase in moisture content of the canola and storage temperature. The rate of increase of FAV was at an acceptable level (less than 1.5-fold increase) for dry canola (7.5% m.c.) stored at low temperature but for high moisture seeds FAV increased more than 3-fold after 3 weeks of storage at high temperature (30 and 40°C). In this study, however, the moisture content of canola stored at 20, 30 and 40°C gradually reduced from initial moisture contents over time, and the final moisture content of 7.5 and 10.0% initial moisture canola were 3.2 and 2.6%, respectively after 16 weeks of storage. At 40°C

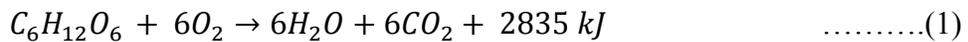
storage, all the samples reached below 6% moisture content after 4 weeks of storage, so it is difficult to correlate the results with the storage moisture content of the canola.

Sun et al. (2014) tested the quality changes of high and low oil content canola varieties stored at different moisture content and temperature regimes. They stored high and low oil content canola varieties with 8, 10, 12 and 14% moisture contents (wet basis), at 10, 20, 30 and 40°C for 20 weeks and analysed the quality changes of canola during storage. They found the safe storage moisture for high oil content canola was lower than low oil content varieties. The germination of canola decreased with increase in storage temperature, and FAV of canola stored at 10, 20, and 30°C increased with increase in storage time, but for canola stored at 40°C, FAV increased up to 6 weeks of storage and dropped dramatically, and they speculated that the oxidation of free fatty acids at higher temperature might be the reason for this drop in FAV after 6 weeks at 40°C storage. Similar trend of increase in FAV with increase in storage time, and decrease of FAV with increase in storage temperature and storage time were found by Nithya et al. (2011) and Rajaramanna et al. (2010) when storing durum wheat and rye at 10, 20, 30 and 40°C. Rajaramanna et al., (2010) stored rye with 10.0, 12.5, 15.0, and 17.5% moisture contents (maintained moisture contents for 16 weeks of storage) and compared their results with the results of Sathya et al. (2009) (moisture content of rye was slowly reduced with increase in storage time). In both the studies, rye stored at low temperature (10°C) maintained germination throughout the storage period (16 weeks) irrespective of storage moisture content. But, germination of high moisture rye decreased significantly with increase in storage time when stored at 20, 30 and 40°C and FAV increased with increase in storage moisture content and temperature (Rajaramanna et al., 2010). Mills and Sinha

(1980) compared the safe storage time of different moisture content rapeseeds stored in the laboratory at different temperatures with rapeseed stored in non-aerated farm bins for 5 months and they concluded that rapeseed with 8.5% or less moisture could be stored for 5 months in non-aerated bins without any spoilage.

2.3. Respiration of grains during storage and controlling insect infestation by using modified atmosphere

Respiration of canola during storage is negligible compared to the respiration of insects and moulds growing on the seeds. Moulds consume the carbohydrates of the seeds and convert the dry matter of seeds into CO₂, water, and heat.



Cereal grains and oilseeds absorb some CO₂ during storage. This sorption rate is faster during initial hours and reaches an equilibrium level after 24 h. Canola at 10% m.c. were stored at two levels of CO₂ concentrations (48%, 69%) and the sorption rates were 400, 620 mg/kg of canola, respectively (Cofie-Agblor et al., 1998). The absorption rate was higher for canola when compared with cereal grains, and the high oil level in the canola may influence the rate and amount of CO₂ diffusion into the seeds. The respiration rate of rapeseed increased with storage temperature and water activity (Magan, 1990). Respiration of rapeseed with 0.95 a_w stored at 25 and 30°C increased linearly with the duration of storage. But the respiration of 0.70 a_w rapeseed was low irrespective of storage temperature. At 25°C, oxygen utilization rates of 0.70 and 0.95 a_w rapeseed were

2.18 and 70.58 ml/ kg of rapeseed/h, respectively. Oxygen utilization rate of 0.85 a_w rapeseed stored at 20 and 30°C were 5.68 and 13.43 ml/ kg of rapeseed/h, respectively. Mould growth in 0.70 a_w rapeseed was negligible when compared with the higher water activity rapeseed, and the respiration rate of this 0.70 a_w rapeseed was also lower than high moisture seeds. At the same storage temperature and moisture content, the carbon dioxide production rate of canola was lower than that of wheat. The higher oil content of canola may cause the lower respiration of seeds.

The CO₂ production rates of 12 and 14% m.c. canola stored at 25-30°C temperature regime were 172 and 290 mg/(d/kg dry matter) (Proynk et al., 2004). But there was no significant difference in CO₂ production rate of freshly harvested and stored canola. White et al. (1982) used the intergranular CO₂ level as an indicator for predicting deterioration of canola. At a storage temperature of 30°C, the canola with 9.7 and 11.3% m.c. produced 12 and 40 mg CO₂/kg of dry mass of seed in 24 h. They predicted that, maximum allowable time for storing 10% m.c. canola at 20°C was 88 days, at which time the cumulative CO₂ in the bins was 1104 mg/kg of dry mass of seed. Canola also absorbs some CO₂ it produces during storage. The CO₂ sorption rate of canola stored at 20°C was measured along with cereal grains by Cofie-Agblor et al. (1998). They noticed that, the CO₂ uptake rate of canola was quicker than that of cereal grains. Canola reached sorption equilibrium level after 24 h of exposure, but oats took 144 h to attain the sorption equilibrium. This study was conducted in a modified atmosphere with high CO₂ concentrations (49 and 71% by volume), and the sorption rate of canola at normal storage conditions were not examined.

Carbon dioxide has been used for disinfestation of stored grain bulks in some grain handling facilities. Increasing CO₂ concentration of the storage atmosphere changes the physiological and metabolic characteristics of the insects (Navarro et al., 2002). The increase in CO₂ creates the stimulus to the opening of spiracles by the insects, which induces the water loss from the insect body and desiccation. The loss of water from the insect body increases with the increase in CO₂ level. Higher CO₂ level decreases the heart and wing muscles activity. Higher CO₂ concentration in the atmosphere also causes the acidification of internal body fluids of insects, which modifies the metabolic processes of insects. This alters the permeability of neuronal cell membranes, which is the reason for anesthesia produced in insects while exposed to elevated levels of CO₂.

Table 2.1 Grading standard for canola

Grade name	Degree of soundness	Standard of cleanliness	Damage (%)			Foreign material included in dockage (%)						Inconspicuous Admixture (%)
			Green	Heated	Total	Ergot	Excreta	Insect excreta	Sclerotinia	Stones	Total	
No. 1 Canada	Reasonably well matured, sweet, good natural colour	Not more than 1.0% of other seeds that are conspicuous and that are not readily separable from canola, to be assessed as dockage	2.00	0.10	5.00	0.05	0.02	0.10	0.05	0.05	1.00	5.00
No. 2 Canada	Fairly well matured, sweet, reasonably good natural colour	Not more than 1.5% of other seeds that are conspicuous and that are not readily separable from	6.00	0.50	12.00	0.05	0.02	0.20	0.10	0.05	1.50	5.00

		canola, to be assessed as dockage										
No. 3 Canada	May have the natural odour associated with low-quality seed, not distinctly sour, musty, rancid, or any odour that would indicate serious deterioration	Not more than 2% of other seeds that are conspicuous and that are not readily separable from canola, to be assessed as dockage	20.00	2.00	25.00	0.05	0.02	0.30	0.15	0.05	2.00	5.00

(Source: CGC, 2014)

Various elevated levels of CO₂ concentrations were tested along with reduced O₂ concentrations for controlling storage insects (Riudavets et al., 2002). At low temperature and high humidity conditions, the grain bulk needed to be exposed for a long time with 35% CO₂ concentration to control the major storage insects, and 4 to 20 days of exposure is needed for control of insects with higher CO₂ levels (50-90%) and reduced O₂ (< 5%). Nearly 99% mortality was achieved for adults of *Lasioderma serricornis* (Fabricius), *Cryptolestes ferrugineus* (Stephens), *Oryzaephilus surinamensis* (Linnaeus), and *Tribolium castaneum* (Herbst) in 4 days when exposed to 50% CO₂ and 3% O₂ condition. But the mortality rate for *Rhyzopertha dominica* (Fabricius) was lower with this gas concentration. When the grain was exposed to 90% CO₂, 3% O₂ and 7% N₂, all adults including *R. dominica* were killed in 4 days. Navarro et al. (2002) tested the effect of CO₂ concentration (60 to 90%) and temperature (30 to 45°C) for disinfestations of storage insects. At 40°C, atmosphere with 90% CO₂ needed to be kept for 10 h to kill *Trogoderma granarium* (Everts) larvae; but this disinfestation period increased significantly when the storage temperature decreased even when the CO₂ concentration was the same (29 h for 35°C). They also found that *Ephestia cautella* (Walker) was the least susceptible insect to higher CO₂ concentrations (6 h needed to achieve LT₉₉ at 40°C). They conducted this study at a lab scale level with a 15 m³ plastic container and the respiration and absorption of CO₂ by the grains were not taken into account.

Mortality of *T. castaneum*, *Plodia interpunctella* (Hübner) increased with decrease in O₂ and increase in CO₂ concentrations. Respiration and metabolic rate of insects increase with increase in storage temperatures; so low oxygen concentration at high temperature causes a lethal effect on insects. High CO₂ concentrations (>32%) also

have the same lethal effect on insects even with high O₂ levels (>15%). But if the CO₂ level is less than 30%, the O₂ level should be less than 1% to cause a lethal atmosphere (Harein and Press, 1968).

Controlling insect infestations using higher CO₂ concentrations were tried at large bin scale levels. Jay et al. (1970) stored wheat in concrete silos with 35% CO₂ and 14% O₂ and they found that the mortality of *T. castaneum* was 93.3% after 96 h of exposure. But the mortality of *T. castaneum* was 9.2% at normal atmospheric conditions. An atmosphere with 61% CO₂, 8% O₂ and 31% N₂ was tested for controlling *T. castaneum* after 96 h of exposure. Jay and Pearman (1973) found 0.1 live and 1.1 dead insects in treated bins, and 144.4 live and 59.8 dead insects in control bins. Alagusundaram et al. (1995) used dry ice for CO₂ production inside the grain bins for controlling insects in wheat. The mortality of most of the storage insects were more than 90% at the grain layer 0.55 m above the floor when they placed dry ice at the floor, but the mortality was 30% at the top layer of the bin.

2.4. Silo bag storage system for cereals and oilseeds

A silo bag storage system creates a nearly hermetic storage condition if it is sealed and maintained properly. The hermetic storage system is also a modified atmosphere storage system, in which the airtight storage structure allows the modification of internal gas composition through the respiration of biological organisms (grain, insects and fungi)

contained within the system. The respiration of these biological organisms increases the CO₂ level, heat, and water vapour; and depletes O₂ concentration of the internal atmospheric composition. The level of airtightness plays a major role in the success of a hermetic storage unit. This process also depends on moisture content and temperature of the grain. High moisture grains have more biological activity than the dry grains, so conversion rate of O₂ to CO₂ is more rapid and intensive in wet grains. The stored grain also absorbs some CO₂, and this absorption rate depends on grain type, moisture content, and temperature.

The grains in the silo bags have a higher heat exchange rate with ambient air and soil than grains in bins, because of the higher surface/volume at the periphery of the bags. Wheat with an initial temperature of 40°C loaded into the silo bags during the summer season in Argentina reached a temperature of 17°C within a couple of months of storage (Bartosik et al., 2008). National Institute of Agricultural Technologies (INTA), Argentina conducted several experiments to test the suitability of silo bags for temporary storage of grains, and Bartosik (2008) recommended that dry (below 76% ERH) grains (wheat, soybean, barley, corn, canola, sunflower) can be stored more than 6 months without any quality deterioration. He also mentioned that storing dry grains in silo bags will not produce any lethal effect for insect development inside silo bags, but the cool temperature in winter may slow down the development of insects. Storing wet grains in silo bags may create a favorable environment for mould growth and lead to increase in CO₂ and decrease in O₂ concentrations inside silo bag which create lethal environment for insects. But the storage time is limited because of mould development in grain which will reduce

the grain quality. Gaston et al. (2009) noticed a rise in wheat moisture content at the top layer of the silo bag, and they suggested that temperature gradients between the top and other parts of the silo bag may have caused the migration of moisture to the top layer of the bag.

Bartosik et al. (2002) tested silo bags to store wheat under Argentinian weather condition for 150 days. One 67 m long bag was filled with 170 tonnes of 12.5% moisture (wet basis) wheat; another bag of the same length was filled with 16.4% moisture wheat and kept in the field for 150 days. They collected samples at three different locations (0.1 m, 0.75 m, 1.6 m from the surface of the bag) at the beginning of the test, and at 45, 80 and 150 days of storage for analysing the quality (test weight, seed germination, and baking test) of the grain. They monitored the temperature of the grain at 10 min intervals using data loggers, and also CO₂ and O₂ concentrations were analysed on the 5th and 100th day of storage using a fast gas analyser. They found no significant change in the moisture content of dry grain (12.5% moisture, but in wet grain bag moisture content of grains near the surface reduced slightly to 15.7%. The average moisture of dry grain bag increased from 12.5 to 13.2%, and in wet grain bag the average moisture content dropped from 16.4% to 16.0%. They reported that the deviation in the moisture content was due to error during sampling and during moisture measurement. There were no replicates in the experiment, as well as no details were provided about statistical analysis of data, which makes it difficult to understand the cause of error. The test weight of dry and wet wheat samples were reduced by 3 and 14 kg/m³, respectively after 150 days of storage, and they reported that this loss of test weight did not affect the grade of the wheat according to

wheat standards in Argentina. There was no significant change in germination for dry wheat, but average germination of wet wheat dropped from 94 to 51% after 45 days of storage and was further reduced to 41% after 150 days of storage. The germination of bottom and middle (1.6 m, 0.75 m from surface of the bag) layers were 26.75 and 22.0%, respectively after 150 days of storage. The drop in germination clearly indicates the significant amount of deterioration in the wet wheat as well as some mould growth during storage. The reduction in germination and drop in test weight did not affect the grade of the wheat based on local grain grading standards. There were significant changes in baking quality of wet wheat after 150 days of storage; gluten content was reduced by 7.2 percentage points and loaf volume was reduced from 675 to 605 cm³. The CO₂ concentrations inside wet and dry wheat grain bags were 22% and 13%, respectively after 100 days of storage. There was no change in O₂ concentration in wet wheat, but in dry wheat O₂ level decreased to 10.4% after 100 days of storage. The authors could not explain the reason for CO₂ development in dry wheat, because there was no fungal and insect activity found in the bags. In both the treatments (wet and dry wheat), the temperature of the grains close to the surface of the bags followed the daily temperature fluctuations.

Rodríguez et al. (2004) stored sunflower seeds with two different moisture contents (8.4%, 16.4%, wet basis) in silo bags for 160 days and analysed the quality parameters. The experiment was conducted in a similar manner to the wheat experiment (Bartosik et al., 2002), and seed samples were collected at 47, 106 and 160 days after storage began. In the 8.4% m.c. seed bag, the moisture contents at the upper layer (0.1 m

from surface of the bag), and bottom layer (1.6 m from surface) were 9.5 and 8.3%, respectively after 160 days of storage. The same trend was noticed in the high moisture sunflower (16.4% m.c.) bag with moisture of 20.5% at the upper layer, and 15.5% at the bottom layer. These trends clearly indicate the moisture migration inside the silo bags during storage. The authors could not explain the reason for difference in moisture contents of top and bottom layers in dry seeds (8.4% m.c.), but they stated that, the incipient condensation on high moisture seeds caused the moisture migration in the wet seed bag. In both the bags, the effect of ambient temperature was minimal at the middle and bottom layer of seeds, and the top layer seeds were affected by the daily temperature fluctuations. At the end of the test, the final temperatures of the bags were not sufficiently high to cause storage problems. They found that there was no significant change in oil content in both treatments, but fat acidity value of the wet seeds increased to significantly high level after 160 days of storage. The CO₂ and O₂ levels of 8.4 m.c. sunflower seeds were 18.9, and 4.5%, respectively, and in 16.4% m.c. bag were 70.0, and 4.7%, respectively after 125 days of storage. The higher level of CO₂ concentration in high moisture bags implies the higher amount of biological activity due to fungal growth. But it is highly unusual to get CO₂ concentration in a regular grain storage ecosystem above 21.0% without any modified atmosphere or external CO₂ supply.

Ridley et al. (2011) conducted an experiment to fumigate the harvest bags with phosphine to kill storage insects. Three harvest bags were filled with 240 tonnes each of freshly harvested wheat with the moisture contents of 9.2, 9.9, and 10.2% (wet basis). The ends of bags were sealed tightly and kept for three months before the experiment.

Two bags were fumigated with aluminium phosphide tablets (500 per bag), and third bag was kept as a control bag. Tablets were kept in 20 mm diameter PVC pipes and inserted horizontally into the silo bag at 10 phosphine release points per bag. In each bag 14 bioassay cages each containing 20 g of mixed culture (all life stages of insect and wheat) were placed. Fumigation was carried out for 17 days and phosphine venting was carried out for 1 week at the end of the fumigation. A rapid increase in phosphine concentrations at the release points was noticed and it remained high throughout the fumigation period. The concentration of phosphine reached 1500 ppm at the release point after seven days and the concentration decrease began after 11 days. The lowest concentration of phosphine was recorded at the start of bags (head part) and phosphine level was more than 200 ppm throughout the bag for 13 days. There were very few live adults found in the fumigated bags, and the number of dead adults in the control bag was negligible. They proved that, phosphine fumigation can be successfully carried out in harvest bags, but it will remove the advantage of modified atmospheric storage with no chemical usage for insect control and also increase the storage cost. During phosphine venting, the tail end of the bag was kept open which allowed the grain to get in contact with atmospheric air, but it created a localised damp spot when the atmospheric humidity was high. Phosphine fumigation may control the insects, but keeping the end open, and inserting a pipe will lead to mould growth and other issues like rain water penetration and increase of rodent activity.

Cardoso et al. (2009) tested the feasibility of phosphine fumigation for controlling insect infestation in wheat inside silo bags and they also tested the change in phosphine

concentration to find suitable application techniques for phosphine fumigation. They filled 3 silo bags with wheat at 13.4% moisture and used 2 bags for 2 dosage levels of phosphine (1 g/m³ phosphine for 1st bag, and 2 g/m³ phosphine for 2nd bag) and kept the 3rd bag as a control. The aluminum phosphide tablets were applied through 40 mm diameter plastic tubes inserted every 5 m along the bag length, and concentration of phosphine was measured for 10 days at four different locations in each treatment silo bag: close to the tail (closing) end of the bag, in between the first two phosphine application points, on a phosphine application point, and on the head end (filling start end) of the bag. For measuring mortality of the insects, they placed insect cages (plastic tubes with 10 live rice weevils (*Sitophilus oryzae*)) at the phosphine measurement locations and measured mortality of the insects after 10 days. They found that, phosphine level reached 200 ppm in 2 and 3 days and in higher (2 g/m³) and lower (1 g/m³) dosage level bags, respectively. The 100% mortality of insects was achieved at both the dosage levels. They also found that dosage of 1 g/m³ phosphine itself created the required concentration of phosphine (200 ppm) for fumigation for 5 days. Higher phosphine concentrations were found at the points close to application points and lower concentration at tail end of the bag due to the lack of air tightness.

Pasin et al. (2009) tested 163 silo bags filled with maize in Argentina for fungal contaminations. The average moisture content of maize was 12.0 to 13.2% while filling into the bags. The bags were filled between April and June (autumn) and opened between September and November (spring-summer). They found that, the bags filled later had a higher level of the mycotoxins, fumonisins B1 and B2 than the bags filled earlier, and

bags opened late contained more fumonisins B1 and B2 contamination than the bags opened earlier. Castellari et al. (2010) found mycotoxin-producing fungal species in the corn samples stored in silo bags. They collected 176 samples from 23 silo bags located in different parts of Argentina with the moisture content above 13% (wet basis), and found two aflatoxin-producing fungal species (*Aspergillus flavus* Link and *A. parasiticus* Speare) and one fumonisins producing fungus (*Fusarium verticillioides* (Sacc.) Nirenberg) in all bags. They tested the damage and air tightness of the bags and found out there were no physical damages and the bags had good air tightness to the end of storage. These results prove that storing high moisture grains in the harvest bags even with the airtight conditions (which provide hermetic conditions) does not help to prevent fungal growth as well as mycotoxin development in stored grain.

2.5. Advantages of harvest bags

If silo bags could be used properly, there are several advantages (Bartosik et al., 2002; Darby and Caddick, 2007; Ochandio et al., 2010a; Ridley et al., 2011; Rodríguez et al., 2004):

- The initial cost of storage is minimal when compared to bin storage.
- Silo bags or harvest bags are very effective storage units for short term storage of grains, when used as a harvest buffer.
- Grain bags can be laid in the field itself, which reduces the waiting as well as transporting time during harvesting.

- Freight cost of grain may be reduced at harvest time when freight charges are high, and farmers can move the grain outside the peak harvest times.
- There will be minimal wastage and shrinkage loss if used correctly.
- Grain bags will allow farmers an additional storage space during bumper crop year.
- Grain segregation based on variety or grade is possible.
- Based on the quantity of the grain, bags can be cut of different lengths.
- There will be minimal quality loss if dry grain is stored correctly for a short duration.
- Harvest bags can also be used and are more suitable for storing organic grains.
- Total expenditure for storage units is minimised and farmers have the choice of choosing length and number of bags based on the yield, rather than having a large capital cost tied up in permanent storage structures.
- The grain cools quickly in the autumn and winter.

2.6. Disadvantages

The major disadvantages of using silo bags are (Bartosik et al., 2002; Darby and Caddick, 2007; Ochandio et al., 2010a; Ridley et al., 2011; Rodríguez et al., 2004):

- The life span of a silo bag is 12-18 months, and they cannot be reused. So, long-term costs can exceed that of permanent storage units, if bags are used every year as a routine storage practice.

- Selection and preparation of storage sites plays a major role in the success of a silo bag storage system.
- The silo bags are prone to damage by birds, rodents, and other animals. So, regular inspection and maintenance of air tightness are necessary to keep the grain safe.
- The moisture content of grain should be at the recommended safe storage level or less.
- Isolation of localised spoilage of grain and removal of that grain is impossible, if a grain extractor is used for unloading.
- Grain quality cannot be monitored during storage without damaging bags.

2.7 Importance of carbon dioxide levels in grain storage structures on grain quality

Carbon dioxide levels of a corrugated-steel bin filled with 254 tonnes of maize was measured using a CO₂ sensor (BinTech Company, Denver, CO, USA) for 8 months of storage and these CO₂ levels were used to detect spoilage of maize during storage (Maier et al., 2010). Results showed that, CO₂ concentration increased above 1000 ppm in June (ambient CO₂ concentration is near 400 ppm), and further increased to 5000 ppm in July, which correlated with spoilage of maize due to mould infection. Removal of spoiled maize from the top surface of bin reduced the CO₂ levels inside the grain bin. Maier et al. (2010) noticed a strong correlation between mould growth and increase in

CO₂ concentration, and the headspace CO₂ concentration was above 9000 ppm when 90% of the maize kernels were infected by fungi. They also noticed, concentration of CO₂ inside a grain bin was between 500 and 1200 ppm during onset of mould growth, and during severe mould infestation CO₂ levels went above 4000 ppm.

Canola with 6% moisture content (wet basis) were stored in silo bags under Argentinian weather conditions for 12 months (Ochandio et al., 2010a) and were analysed for quality and CO₂ concentration changes during storage. It was found that, the CO₂ levels ranged from 1 to 8% (10,000 to 80,000 ppm), which indicated the moderate biological activity inside silo bags, and there was no significant change in quality parameters (free fatty acid values, foreign material), and no loss in grain grade after 12 months of storage. They found some perforations in the bag, and they suggested the rain water leak through these perforations might have caused the moderate level of biological activity inside the silo bag. The recommended storage moisture content for canola is 9-10% (wet basis), and in this experiment very dry seed (only 6% m.c.) was tested to find the feasibility of storing canola in silo bags. At this dry condition, there should not be any moisture migration inside the bags, as well as not much biological activity to produce enough CO₂ to create hermetic conditions.

Soybeans were stored in silo bags in Argentina for 5 months, and the CO₂ level was less than 3% in the winter time for all three moisture contents seeds (11.5, 12.9, and 14.9% m.c.), and CO₂ level went up to 9.0 and 10.0% in spring in 12.9 and 11.5% moisture content seeds, respectively (Cardoso et al., 2008). In October, in these bags CO₂ levels rose to 16.0, 18.0% and then decreased to 10.0, 13.0% and remained the same for

the rest of the storage period. But in high moisture seeds the CO₂ level was less than 2.0% for these 5 months of storage. There were perforations at the bottom of 11.5 and 12.9% moisture soybean silo bags, which may be the reason for water and O₂ penetration into the bags and mould growth in the spring period. But the researchers noticed no holes in the high moisture bags and seeds were in good shape after 5 months of storage. High moisture wheat (18.0%) stored in silo bags in Argentina rapidly spoiled completely due to the high mould activity and researchers reported that the CO₂ level above 10% inside silo bags resulting from the higher biological activity inside the grain bulk due to mould and insect activity, and this elevated CO₂ level was an indicator of unsafe storage conditions.

Experiments conducted by INTA, Argentina to check the quality changes of maize (Casini et al. 2009; Rodriguez et al., 2002a) and soybean (Rodriguez et al., 2002d) indicated that CO₂ concentration inside the silo bags reached only up to 10% and O₂ level stayed above 10% (ambient O₂ concentration in air is about 21%), which created a non-lethal atmosphere for insect growth, and Cardoso et al. (2009) indicated this atmosphere would not control *S. oryzae* growth for 60 to 80 days inside a grain bulk. Cardoso et al. (2009) also stated that, creating a lethal environment for insect control (>15% CO₂) was linked to biological activity due to mould growth inside the grain bulk, which decreases the quality parameters of the grain. Rodriguez et al. (2002b) noticed a drop in germination and test weight of wheat, and Rodriguez et al. (2002c) noticed oil acidity increased in sunflower seeds stored in silo bags with elevated CO₂ levels due to fungal growth. Rodriguez et al. (2004) tested the storage of soybean, maize and sunflower in

harvest bags in the Buenos Aires Province, Argentina. Maize, soybean, and sunflower seeds were stored at two moisture contents (14.8 and 19.5% for maize, 12.5 and 15.6% for soybean, 8.4 and 16.4% for sunflower) for 160 days, and they concluded that there was no significant change in quality of all three seeds. These trials were conducted at safe storage temperature limits, and the quality of the seeds used in these experiments was poor. The initial germination of sunflower seeds was too low, so authors omitted the germination for quality assessment. There was a significant increase in free fatty acid values in high moisture sunflower seeds, and they reported loss of germination for maize and soybean for both the moisture contents. They found a high level (70%) of CO₂ concentration in wet sunflower seeds, but there was no explanation from the authors.

Rodriguez et al. (2008) tested the factors affecting CO₂ concentration of silo bags filled with wheat, and they found CO₂ concentration increased with increase in moisture content of wheat. Carbon dioxide level was around 5% for silo bags with 14% m.c. wheat, but in silo bags with wet wheat (19% m.c.) CO₂ concentration was 30%. They also noticed the CO₂ concentration of good quality wheat was 5 to 7.5% less than in the silo bags with poor quality wheat. The grain temperature had significant effect on CO₂ concentration inside silo bags when the moisture content of the wheat was above 14%. The CO₂ concentration during the warm season was 7% higher than that during the cold season when moisture content of the wheat was higher than 16%, but the CO₂ concentration remained at a constant level inside the silo bags filled with wheat below 14% moisture content.

Bartosik et al. (2008) used the CO₂ concentration inside the silo bags for early detection of spoilage of grain. They stored soybean at 11.5, 12.9 and 14.9% moisture content (wet basis), and CO₂ levels were below 3% at all three m.c.s during the winter season. Carbon dioxide concentrations increased to 9 and 10% during early spring for 11.5 and 12.9% m.c. bags and further increased to 16 and 18% during late spring. The CO₂ concentration of the silo bag with 14.9% m.c. soybean was around 2% throughout the storage period and they noticed perforations at the bottom of bag during unloading, which would have affected the integrity of air tightness of the silo bag. Carbon dioxide concentrations inside silo bags filled with 13 and 16% m.c. wheat were <5% and 17%, respectively.

Cardoso et al. (2008) researched the factors affecting the CO₂ concentration inside silo bags with soybeans, and they found CO₂ concentrations increased with increase in moisture content of the soybean. They also reported CO₂ concentration increased about 1.5% for each 10°C increase. Barley with average moisture contents of 11.0% and 11.5% (wet basis) were stored in two silo bags for 5 months in Argentina (Ochandio et al., 2010b). The initial germination of barley in both the bags was 100.0% and after 5 months of storage germination was above 98.0% in the 11.0% barley bag, and it was slightly lower (97.6%) than the malting industry standard (at least 98.0% germination) in the 11.5% bag. There was no significant change in protein value throughout the storage period, and CO₂ level inside the bags were at 11 and 13% for 11.0 and 11.5% m.c. barley, respectively. This result also proved that, barley with relatively dry moisture (11.0%) can

be stored for short duration (up to 5 months) without any significant change in malting qualities in the harvest bags.

Ochandio et al. (2012) measured the CO₂ concentration inside hermitically sealed glass jars with soybean at three different moisture contents (11, 13 and 17%) for 1 year stored at different temperatures (5 and 35°C). There was no change in CO₂ concentration in 11% moisture content jars stored at 5°C for the whole storage period but in 17% moisture content soybean jar CO₂ concentration increased to 5 to 7%. In soybeans stored at 35°C, CO₂ concentration was increased above 20% for 17% moisture content after 100 days of storage, and in 11% moisture soybean jars CO₂ concentration was around 5% after 230 days of storage. In glass jars with 13% moisture soybeans, CO₂ concentrations were less than 1% and 12% after 1 year of storage at 5 and 35°C, respectively. The results proved that, storing soybean at high moisture content at hot temperature increased the biological activity due to mould growth, which caused the increase in CO₂ concentration, and dry soybean can be stored for longer times at 5°C without any spoilage.

Darby and Caddick (2007) assessed the use of harvest bags under Australian conditions. They conducted field trials as well as a survey of farms and concluded that air tightness was not achieved to a safe level to create hermetic storage conditions. They recommended that harvest bags can be used only for short duration for storing dry grains. They found that, storing wet grains in silo bags for more than 8 weeks led to mould damage. They also found that the grain in the peripheral layer was affected more compared to the grain in the middle or bottom of the bags because of the accumulation of moisture due to moisture migration and condensation and rapid temperature fluctuations.

The major advantage of silo or harvest bag is: these can provide a hermetic condition with elevated level of CO₂, which provide an undesirable condition for insect or mould survival in the grain bulk. But achieving 100% air tightness, which creates a hermetic condition, is practically difficult, and also storing grains with low moisture content did not create a suitable modified atmosphere to kill insects inside the silo bags.

2.8 Prediction of CO₂ levels and mathematical models for predicting temperature, moisture and intergranular gas composition profiles

2.8.1. CO₂ prediction models

Carbon dioxide level inside a grain bulk has been used as an indicator of grain spoilage for a long time in the grain handling industry (White et al., 1989). All the living organisms in the grain storage ecosystem, i.e., grain, insects, fungi and other microorganisms (bacteria, actinomycetes) consume O₂ and produce CO₂, heat, and moisture during respiration. Production of CO₂ is negligible in healthy grain, but if the grain bulk has insect or fungal infection CO₂ production will be increased. The higher the biological activity, the higher the CO₂ level inside the grain bulk. Muir et al. (1980) noticed elevated levels of CO₂ close to the spoilage locations in polythene, wooden, and steel bins, and open piles of wheat. Maier et al. (2006) monitored CO₂ levels inside three steel tanks and two grain piles with maize (15.5 to 16.3% moisture content) using CO₂

sensors (in each steel tank: four sensors in the headspace and four sensors in the bottom fan exhaust air streams; in ground piles: one sensor in each exhaust fan air stream). For steel tanks CO₂ levels were monitored between April and September, 2005, and in ground piles they were measured from January to September, 2005. In steel tanks CO₂ levels increased from 500 ppm (0.05%) to 5000 ppm (0.5%) at the middle of June and CO₂ concentration decreased after aeration fans were turned on. They noticed a localized hot spot close to the top grain surface, which might be the reason for this increase in CO₂ level. They also noticed increased level of CO₂ in one ground pile and they noticed spoiled grain close to the sensor location, which measured high CO₂ concentrations. These results proved that CO₂ levels inside grain storage structures can be used an indicator for grain spoilage.

White et al. (1982) developed a parabolic regression equation (Eqn. 2) to predict CO₂ production rate of wheat using a laboratory study. They stored hard red spring wheat with eight moisture contents (14 to 25%, wet basis) in 300 mL flasks at 10, 20, 30, and 40°C. Gas samples were collected using 4 mL syringes three times a week (Monday, Wednesday and Friday) from the flasks stored at 10 and 20°C, and five times a week (Monday to Friday) from the flasks stored at 30 and 40°C and analyzed using a gas chromatograph (Perkin-Elmer Sigma 3B) equipped with a thermal conductivity detector. The R² value for the equation was 0.794 and standard error was 0.437, and they also found moisture content was 1.5 times more important than storage temperature for predicting cumulative CO₂ production using standardized regression coefficients of the variables. Though this regression model was developed using a wide range of storage

conditions (storage moisture and temperature), this equation was developed based on CO₂ production of wheat. Each type of grain respire differently; notably respiration of canola is very different than that of wheat. So there is a need for a separate model of CO₂ production of canola.

$$\log_{10}(R (CO_2)) = -4.054 + 0.0406(T) - 0.0165 (\theta) + 0.0001 (\theta^2) + 0.2389 (M) \dots\dots\dots(2)$$

Where,

R (CO₂) = rate of CO₂ production (mg / kg of grain in a day)

T = temperature (°C)

θ = time (days in storage)

M= moisture content of the grain (% , wet basis)

Pronyk et al. (2004) stored freshly harvested canola at 10, 12 and 14% moisture content at two temperature regimes (25-30°C and 30-35°C) in 1 L flasks and measured CO₂ production over 3 h periods using a respirometer. They developed a prediction model (Eqn. 3) using a backward regression method to predict CO₂ production rate and R² value of this model was 0.765. This model was developed using the CO₂ production of canola at higher storage temperature (>25°C), which creates a favourable environment for mould growth which might increase the CO₂ production due to the higher biological activity.

$$\log_{10}(R (CO_2)) = -1.521 + 0.0462(T) + 0.0159 (\theta) - 0.00006990 (\theta^2) + 187 (M) \dots\dots\dots(3)$$

Where,

R (CO_2) = rate of CO_2 production (mg/ kg of dry matter seed in a day)

T = temperature ($^{\circ}\text{C}$)

θ = time (days in storage)

M = moisture content of the grain (% , wet basis)

2.8.2. Mathematical modelling

Physical experiments cannot be conducted for each and every case to test the airflow rate, temperature distribution, moisture distribution and inter granular gas composition of a grain bulk. Mathematical simulation models were developed based on several physical and biological factors involved in grain storage ecosystems for helping farmers and grain storage facility managers to assess the effect of various parameters of a grain bulk to determine the best storage management practices (Alagusundaram et al., 1994). These models were developed using different solution methods and validated against the measured experimental data. The importance of each variable affecting different parameters like airflow distribution, temperature and moisture profile, and intergranular gas composition can be tested using sensitivity analysis (Sinicio et al., 1997). Analytical solutions can be developed under simplifying assumptions to simulate these parameters. Smith et al. (2001) developed an analytical solution model to predict the CO_2 through grain bulks. But tedious solving procedure of Laplace's and partial differential equations made this analytical method difficult and also the model had limitations based on end-use. Thus numerical models (finite difference models and finite element models), are commonly used to explain the behavior of several physical systems.

Several scientists developed different numerical models to predict temperature profiles in stored grain bulks (Alagusundaram et al., 1988, 1990; Jayas et al., 1994; Mani et al., 2000; Jian et al., 2005). Three-dimensional finite element models developed to predict temperature distributions in rapeseed and barley-filled bins had average standard error of means of 3.3 K and 3.1 K, respectively (Alagusundaram et al., 1990). These models also predicted higher temperature at the south side of bins. A two-dimensional finite difference model developed to predict temperature profiles in a maize bin predicted that the headspace temperatures were 1 to 8°C warmer than ambient temperature, and the measured and predicted temperatures of the silo were not significantly different at 1% level of significance using the F-test (Alabandan, 2005). A combined heat and moisture transfer model using control-volume formulation was developed to predict the migration of moisture in stored grain due to temperature gradients. The measured and predicted values were not significantly different for temperature and moisture distribution values at winter and summer storage conditions (Khankari et al., 1994). Several three dimensional numerical models also had been developed to predict the CO₂ diffusion in grain bins (Alagusundaram et al., 1991; 1994; 1996). Grains absorb some CO₂ while in storage, and these sorption values of grains at different initial CO₂ concentrations had significant effect on sensitivity of a model (Cofie-Agblor et al., 1998).

Numerous simulation models were developed using finite difference and finite element methods to predict temperature, moisture and intergranular gas composition of grain bulks in bins (Alagusundaram et al., 1988, 1990; Jayas et al., 1994; Mani et al., 2000). But for predicting these parameters of grain bulk in silo bags, only very few

studies have been conducted. A mathematical model was developed to predict the moisture migration and temperature distribution inside silo bags with wheat (Gaston et al., 2009). The developed model predicted an increase in moisture from 1.0 to 1.5 percentage points (wet basis) near the top layer of the grains (close to surface of the bags). They stated that the temperature difference between the top layer and rest of the grain in the bag caused the moisture migration from the grain mass to the top of the bag, which creates condensation near the top of the bag. This experiment was conducted in Argentina, where the seasonal temperature fluctuation is moderate; even then the moisture migration inside harvest bags caused 1-1.5 percentage point moisture increase in the top layer. So it is expected that there will be greater increase in moisture content at the top layer of grains in harvest bags under Canadian conditions (especially the Prairie provinces), where seasonal temperature fluctuations can be up to 70°C (30°C to -40°C) in a given year. The increase in moisture combined with higher temperature at the top of the bag will provide a favourable condition for mould growth in the grain bulk.

A computational model was developed based on O₂ mass diffusion in a porous media to predict the O₂ infiltration rate due to the surface damage in silo bag containing maize (Bispo dos Santos et al., 2007). The O₂ diffusion rate increased with an increase in the size of silo bag damage (size of perforation or cut in the silo bag) and decrease in temperature. They also found that, the deterioration of grain started close by the damage due to the favorable condition created by the elevated O₂ concentration for the growth of insects and mould. A finite volume model was also developed to predict the effect of O₂ depletors to accelerate the anaerobiosis of maize in the silo bags (Lobo Peas et al., 2007).

The model predicted that, use of O₂ depletors reduced the O₂ mass fraction values to 0.06 within 6 h after sealing, but the silo bags without depletors had O₂ mass fraction value of 0.1 after 12 h of sealing. But, there were no details about the O₂ depletors used in this experiment and statistical analysis, which made it difficult to interpolate the results with field conditions.

A lumped-capacity differential model was developed to predict the gas concentration in the interstitial atmosphere of silo bags with wheat (Abalone et al., 2011). The model was validated against the field experiment data of silo bags containing 12 to 15% m.c. wheat, and the standard error (SE) of prediction for CO₂ were 2.7 percentage points for dry wheat (12 to 13.5% m.c.), and 2.5 percentage points for damper (14.0 to 15.0% m.c.) seeds, respectively. For O₂ prediction, the SEs were 2.5 and 1.9 percentage points for dry and damp seeds, respectively. The sensitivity of this lumped-capacity model was tested against the respiration and permeability of the bags (Abalone et al., 2011), and the results showed that the interstitial atmospheric gas concentration was more sensitive to the respiration of seeds than to the permeability of the silo bags. But for developing this model, the average permeability of low density polyethylene (LDPE) and high density polyethylene (HDPE) was used and the effects of weather and stretching on permeability of the bag material were not considered. The assumption made in this study was that half of bag thickness was LDPE and other half was HDPE. The permeability of this bag material may be different than the average permeability value of LDPE and HDPE, and the stretching and temperature gradient between grain and ambient air may

also alter the permeability rate because the energy of gas molecules has a strong relationship with permeability rate (Delassus, 1997).

Jian et al. (2015a) developed a model to predict the soil temperatures at different depths under silo bags using heat conduction equations (HCE) and the coupled three-dimensional transient heat, momentum and mass transfer model (Fourier series model) inside the silo bags containing canola. The developed model was validated using the data reported by Chelladurai et al. (2011), and magnitude of relative error for predicting soil temperature with the developed HCE and Fourier series models were 0.4 ± 0.0 , and 2.2 ± 0.1 , respectively. They also found the maximum absolute difference between measured and predicted soil temperature with HCE and Fourier series models were 4.9°C , and 16.8°C , respectively. Linear regression between measured and predicted temperatures indicated that the prediction accuracy of the HCE model was higher than the Fourier series model for predicting soil and canola temperatures. The HCE model had 93% and 88% accuracies for predicting soil and canola temperatures, respectively.

Jian et al. (2015b) also developed a three-dimensional model to predict moisture content and temperature of canola inside silo bags stored under Canadian Prairie conditions and coupled it with the soil temperature developed by Jian et al. (2015a). Data collected from the field experiment (Chelladurai et al. (2011)) were used for validation of the developed model. The maximum absolute difference between measured and predicted temperatures of 10.5% m.c. canola was 4.4°C , and the prediction accuracy was more than 90%. But the accuracy for predicting temperatures of 14.4% m.c. canola was low (23% to 79%), and they speculated that the hot spots developed in the damp canola silo bags

might have been the reason for low prediction accuracies. Similar to the temperature prediction models, the developed moisture content prediction models also had higher accuracy for predicting moisture content of canola in 10.5% m.c. canola silo bags than 14.4% m.c. canola.

2.9 Permeability

Permeability is defined as transfer of gas molecules from the product to the external environment, through package material or from the external environment to the product. Permeation rate is the rate at which the gas or vapour passes through the polymer or packaging material. Permeability coefficient is the volume of gas or vapour passing through a unit area of polymer per unit time, with a unit pressure difference across the sample (Delassus, 1997).

Permeability of a material differs from gas to gas and it depends on dimension (dynamic diameter) and shape of gas molecules. For example, for some packaging materials like polyethylene (PE) N_2 has the smallest permeability rate, O_2 has a higher rate, and CO_2 has the highest permeability rate (Cooksey, 2004). The basic theory is, when the gas molecule has a smaller dynamic diameter, it can easily diffuse through the barrier material and it has the highest diffusion rate. Commonly, the permeability ratios of a material for N_2 , O_2 , and CO_2 gases are 1:4:14 (Delassus, 1997). But the size and shape of gas molecules are not the only parameters that affect the permeability. Temperature and humidity also play major roles for permeability of a material for different gases (Cooksey, 2004). For hygroscopic materials like nylon, polyvinyl alcohol,

and polyvinyl acetate, increase in relative humidity (r.h.) increases the permeability for gases. But for polyethylene materials (low and high density polyethylene), permeability for N₂, O₂, and CO₂ are unaffected by the change in r.h., because of their excellent water barring capacities. Increase in temperature increases the permeability of a material for different gases, and this linear relationship between temperature and permeability is controlled by the gas transition temperature (T_g) of the material (Delassus, 1997). Permeability of a material increases about 9% per °C increase in temperature above T_g and permeability increases at about 5% per °C increase in temperature below T_g. When the temperature increases, the gas molecules attain more energy and they easily pass through the packaging materials due to the high energy (Cooksey, 2004). In polythene based films, mechanical stress on the films has less effect on permeability when compared to temperature and humidity (Mrkić et al., 2007). The tensile drawing orientation has significant effect on permeability coefficient of co-extruded low density polyethylene (LLDPE) films for O₂ and CO₂ gases (Compan et al., 1996). Villaluenga and Seoane (2000) reported that, the number of layers of LLDPE films did not affect the permeability coefficient of LLDPE films for carbon dioxide. The O₂ and CO₂ permeability values of a material were measured by “Dow cell” method prior to the 1970s. In this method, permeability rate was measured either by a manometric method or a volumetric method. In the manometric method, two compartments of a cell, separated by the packaging film or barrier material, are filled with carrier gas in one compartment (receiving compartment) and O₂ or CO₂ is added the other compartment (permanent gas compartment). The inlet pressures of both gases are controlled and pressures in the

compartments and temperature of the inlet gas are measured. Then the permeability constant, P is calculated as (Villaluenga and Seoane, 2000):

$$P = \frac{Ql}{At\Delta p} \dots\dots\dots(4)$$

Where,

Q is the change in O_2 or CO_2 in the receiving compartment (m^3)

l is the thickness of film or barrier material (m)

A is the area of film or barrier material (m^2)

t is the time of steady state (d)

Δp is the pressure difference between compartments (atm).

Volumetric measurements use change in volume of material on both sides of the films or barrier materials for measuring permeability, and this technique is mostly used to measure permeability of films for liquids (especially water). A new coulometric sensor method replaced these traditional methods in 1970, in which a sensor was used to measure the O_2 transmission rate through the films (Trodel, 1999). But the major drawback in this method is, it can be only used for measuring permeability for O_2 . Permeability of a packaging film or barrier material for CO_2 and other gases can be measured only through the traditional manometric methods.

Chapter 3

Materials and methods

3.1. Effect of storage moisture content on canola seed quality

For testing the effect of initial storage moisture content on the canola seed quality during storage in silo bags under Canadian Prairie conditions (Objective 1), canola with three initial moisture contents were loaded into silo bags and stored for 10 months.

3.1.1. Canola

Canola were obtained from a commercial elevator (Richardson International Limited, Dauphin, Manitoba) at three different moisture contents (8, 10, and 14%) and loaded into grain bags on October 7-8, 2010 using a bag loader. Grain bags to store canola were obtained from Grain Bags Canada, Humboldt, SK. The bags were manufactured in Argentina (Manufacturer: IpesaSilo, Ciudadela, Argentina) and these

bags are 2.74 m diameter (9 feet) in diameter and 76.2 m (250 feet) in length. The storage capacity of these bags was approximately 250 tonnes of canola. The harvest bags were made of three-layer polyethylene membrane (235 μm thick). The first two white layers acted as a UV filter and the third black layer kept out the sunlight. During loading with the canola, this bag stretched up to 10% of its diameter. The moisture content level was considered as an experimental treatment, and each treatment had three replicates. The canola were loaded in nine silo bags (3 treatments \times 3 replications) with approximately 20 tonnes of canola per bag, and stored until August 10, 2011.

3.1.2. Preparation of storage site

Selecting an appropriate storage site and site preparation plays a major role in success of a silo bag storage system. A site was selected close to the Environmental Health and Safety building at University of Manitoba's Fort Garry campus, Winnipeg, which is easily accessible throughout the year and had a hard surface. All the sharp objects, stones, and plant stubs were removed and ground was thoroughly graded using a grader (Figure 3.1). Then padding was prepared with a height of 0.23 m, and width of 3.66 m using gravel and on top of this padding sand was spread with 0.10 m height to create a smooth surface to avoid any damage to the bags due to small stones or sharp objects in the gravel. At one corner of this elevated surface, a drainage pipe was installed to avoid water pooling around the silo bags due to rain or snow melting. Two insulated storage sheds (1.83 m \times 1.10 m \times 1.83 m) with electrical power supply were placed on

the storage site for placing data collection systems (data loggers and computers) for temperature measurement.

3.1.3. Loading of Canola into grain bags

Canola was loaded into grain bags using specialized equipment called a ‘grain bag loader’ (Model: Mainero 2230, Carlos Mainero & CO SAICFI, Bell Ville, Córdoba, Argentina). The grain bag loader consisted of a hopper, a grain auger, grain filling tunnel and platform with two wheels. The grain filling speed and stretching of bags were controlled by the disc brake system equipped on both the wheels. The brake lever with a pressure gauge mounted on the frame controlled the hydraulic pump by which braking pressure was controlled. The filling tunnel was embedded with a retractile support for mounting bags on the grain bag loader. The canola was hauled by 44 tonnes capacity Super Bee trucks from the elevator to the experiment site, and then loaded into a grain cart using a grain auger. Canola from the grain cart was transferred to the hopper of the grain bag loader, and then thrown into the bags through the filling tunnel by the auger.

Before filling with the canola, the entire grain bag (76.2 m length) was mounted on the bag loader, and about 3 m of bag was pulled out for sealing the head portion of the bag. Two 0.61 m × 1.22 m (2" × 4") treated lumber (length 3.66 m (12')) were placed at the bottom and top of the bag at 30 cm from the end of bag and screwed together at every 15 cm (Figure 3.2). This sealed end was then tucked under the bag about 50 cm from the line of the start, which gave good airtightness because of the weight of grain inside the bag. After the head end preparation, canola were filled inside

the bag for the required bag length, and the loader was moved forward about 3 m without grain flow at the end and bag was cut from the loader. Similar to the head portion, at the tail end also two 0.61 m × 1.22 m (2" ×4") treated lumbers (length 12') were placed at the bottom and top of the bag at 30 cm from the end of bag and screwed together at every 15 cm. Then the bag was rolled 5 to 6 times with the wood to create sealing at the tail end.



Figure 3.1 Preparation of storage site for silo bag storage system

3.1.4. Unloading of canola

Canola was unloaded from the grain bags after storage using a specialized equipment called a “grain bag unloader” or “bag extractor” (Model: EXG 300, Akron, San Francisco, Córdoba, Argentina). This unit consisted of two augers (one horizontal and one vertical auger), a bag cutter and bag collection roll. Augers of this extractor worked through the 540 rpm power take-off (PTO) shaft of a tractor, and the hydraulics portion helped to move the vertical auger up and down. The whole extractor unit hitched with a 120 HP tractor and augers and hydraulics were operated by the tractor's PTO shaft and hydraulics. After storage the 0.61 m × 1.22 m (2" × 4") lumber at the tail end was opened and a vertical cut was made for about a 1.83 m (6 feet) length at the top of the bag. Then the bag material was hooked into the teeth of bag collection roll, and the cut at the top helped to slide the bag material around the vertical auger. The extractor moved backwards and the seeds were moved to the centre part by the horizontal auger and then moved up by the vertical auger. The seeds from the grain bag can directly load into semi-truck or a Suber B truck using this extractor (Figure 3.3).

3.1.5. Sample collection

Samples were collected through seed sampling ports at the centre along the length of the bag (Figure 3.4 A). Seed sampling ports were created by placing PVC nipples with bulkhead fittings into the bags (Figure 3.5)). These nipples were closed at the end with caps and the cuts made for inserting these fittings were closed using tape

(specially designed for silo bag by IpesaSilo, Ciudadela, Argentina) and expansion Styrofoam (Great Stuff Gaps & Cracks, The Dow Chemical Company, Midland, MI). Once every 2 wk, seed samples were collected using a grain trier (1.83 m length brass probe with 12 openings, Model: 16-OH, Seedburo Equipment Company, Des Plaines, IL). The probe had 12 compartments and seed samples were collected from the compartments at 0.15, 0.8 and 1.35 m from the top of the bag, which represent the top, middle and bottom layers of the silo bag (Figure 3.4 B). Seven samples were collected from each bag. Four samples of the total seven samples represented middle and top layer of grain from each side and three samples represented the bottom, middle, top layers of the grain from the centre of the bag. From the side ports, seed samples were collected using a probe by standing on the ground, and for collecting seed samples from the top ports, a scaffolding was fabricated (height – 2.13 m (7'), length – 4.57 m (15') , and width – 3.66 m) using two aluminum beams and a wooden floor was placed on top of it. A ladder was used to climb up the scaffolding and samples were collected from the top ports using this scaffolding without damaging the bags (Figure 3.6).



Figure 3.2 Loading of canola into silo bags



Figure 3.3 Unloading of canola from silo bags

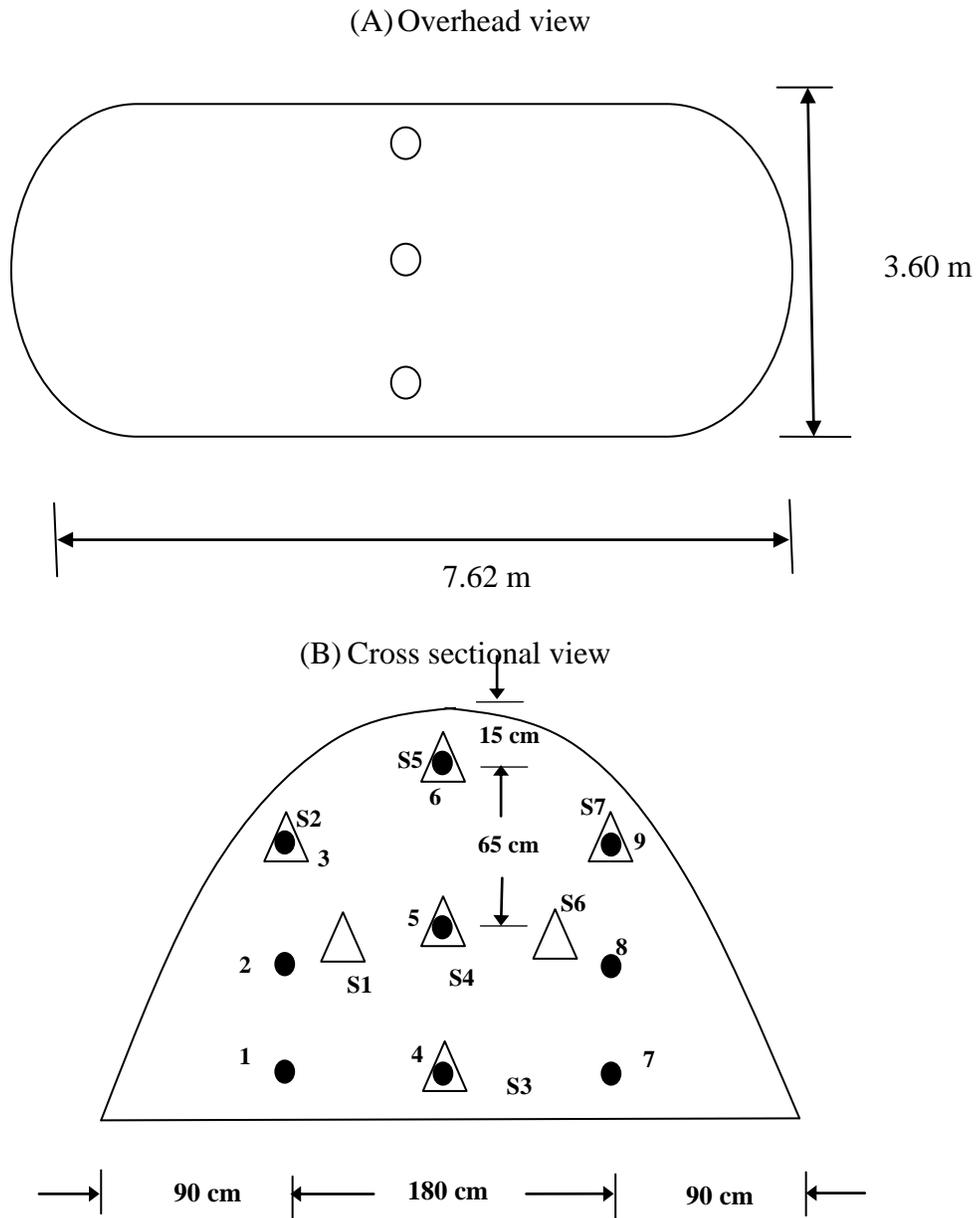


Figure 3.4 Overhead (A) and cross sectional (B) view of sampling locations. In the graph, Δ - seed sampling locations; \bullet -temperature measurement locations and CO_2 collection locations



Figure 3.5 Silo bags with sampling arrangement (i) seed sampling ports, (ii) CO₂ sampling tubes, and (iii) thermocouples

3.1.6. Grain temperature

The position for the temperature monitoring and gas sampling were located at the same location as the seed sampling locations at 0.15, 0.80 and 1.35 m from the top of the bag, which represented the top, middle and bottom layers of seeds in the bag (Figure 3.4 B). Thermocouples were connected to a data acquisition system (Model: 4970A Agilent Technologies Inc., Santa Clara, CA) for continuous temperature monitoring at 30 min intervals.



Figure 3.6 Seed sampling from the top port using scaffolding

3.1.7. Intergranular CO₂ concentration

Gas sampling locations were established at the same locations along the length of the bag, where seed samples were collected for quality analysis. Three PVC tubes with lengths of 0.15, 0.80 and 1.35 m (4 mm internal diameter, 6 mm outer diameter) were inserted closer to each seed sampling ports through the PVC coupling and this coupling was sealed using extending Styrofoam. End of these tubes were covered by mesh to avoid entry of canola during gas sample collection and the thermocouples for temperature measurement were attached to these gas sampling tubes using electrical tape. From each bag, gas samples were collected at nine locations (Figure 3.4B), similar to temperature

measurement. For measuring CO₂ concentration inside the silo bags, gas samples were collected through the tubes inserted at the gas sampling locations by using 60 mL syringes at 2 wk intervals. These gas samples were analyzed using a gas chromatograph (Model: Clarus 420, Perkin Elmer, Woodbridge, ON) with a thermal conductivity detector (Shunmugam et al., 2005). The complete process of silo bag storage experiment (from loading to unloading of canola) is explained in a flow chart (Figure 3.7).

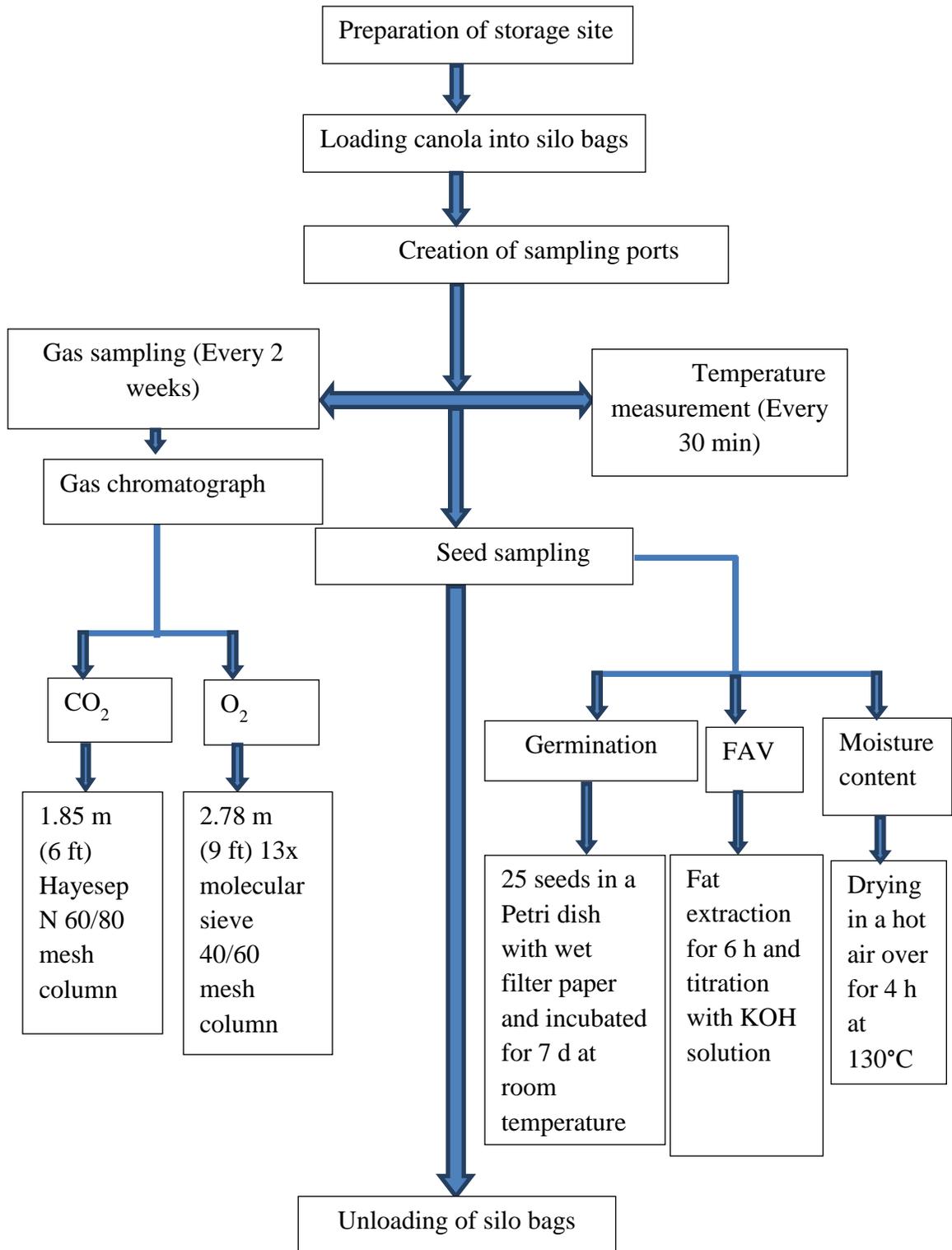


Figure 3.7 Flowchart to explain the sample collection and quality assessment methods in the silo bag experiment

3.1.8. Quality measurement

3.1.8.1. Measurement of seed moisture content

Moisture content of canola was measured using ASABE S352.2 standard method (ASABE, 2012). About 10 g of canola was placed in a moisture measurement dish, and dried in a hot air oven for 4 h at 130°C. After the drying dish was covered and kept in a desiccator for 15 min to cool, and final weight of sample was measured using a weighing balance. Moisture content of all the canola samples was measured in triplicate.

Moisture content of the sample was calculated using the following equation.

$$\text{Moisture content (m.c.) of canola (\%)} = \frac{m_i - m_f}{m_i - m_d} \times 100$$

Where,

m_i = Initial weight of sample + dish (g)

m_f = Final weight of sample + dish (g)

m_d = Empty weight of dish (g)

3.1.8.2. Measurement of seed germination

Germination of canola seeds was measured every 2 wk by the method developed by Wallace and Sinha (1962). A Whatman No.3 filter paper (90 mm diameter) was placed inside a petridish and wetted with 5.5 mL of distilled water. Randomly selected 25 canola from the collected samples were placed on the saturated filter paper and then the petridishes were placed in a stand. Then the whole stack was covered using a polythene bag to avoid loss of moisture from seeds and filter paper and incubated at room temperature (23±1°C) for 4 days. After 4 days, polythene cover was opened and

incubated at room temperature for another 3 days. The number of seeds germinated was counted after 7 days and germination was expressed in percentage.

3.1.8.3. Measurement of free fatty acid values (FAV)

After moisture content measurement, the dried samples were ground using a laboratory mill (Model: M2, Stein laboratory mill, Seedburo Equipment Co., Des Plaines, IL) and about 5 g of ground sample was placed on a Whatman No. 5 filter paper (110 mm diameter). Then the filter paper was folded and placed inside a sample holder of the fat extractor (Model: Goldfish fat extractor, Laboratory Construction Co., Kansas City, MO). About 30 mL of extraction solvent (petroleum ether) was placed in glass beakers of the extractor and boiled at 60°C for 6 h when the solvent was evaporated and condensed, and passed through the ground sample multiple times. After 6 h, the sample holders were replaced with solvent collection glass tubes, and the mixture of oil and solvent collected in the beakers were boiled until the entire solvent was recovered in the collection tubes. Then the separated oil was mixed with 25 mL of TAP solution (50% toluene, 50% ethyl alcohol with 0.4% phenolphthalein as an indicator) and titrated with known normality potassium hydroxide (KOH) solution. The FAV of canola samples were determined in triplicate using the following equation and expressed in mg of KOH / 100 g of dried canola (AOAC, 1962).

$$FAV (mg\ KOH/100\ g\ of\ dried\ canola) = \frac{(KOH_S - KOH_B) \times N}{W_s} \times 100$$

Where,

KOH_S = KOH used in titration of oil + KOH mixture (mL)

KOH_B = KOH used in titration of blank KOH solution (mL)

W_s = Weight of sample (g)

N = Normality of KOH solution (N)

3.1.9. Commercial grading

Commercial grade of the canola were determined in the Quality Lab at Richardson International, Winnipeg, MB based on Canadian Grain Commission standard test protocol (CGC, 2012) before loading into silo bags and two days prior to unloading from the bags. Moisture content, oil content, amount of green and heated seeds, and dockage level were analysed in the quality lab for determining the commercial grade.

3.1.10. Statistical analysis

The effect of storage moisture content, storage time, and sample location in the bags on quality parameters (seed germination and FAV content), changes in moisture inside the bag, and intergranular CO₂ concentration were analysed using analysis of variance (ANOVA) technique. Tukey's test with 95% confidence interval (at $\alpha = 0.05$ level) was used for means comparisons. The preliminary analysis with Tukey's method showed that there were no significant differences in quality parameters among samples collected from the side and centre ports. So samples were clustered into three groups: Top, Middle, and Bottom. These three groups represented three layers of seeds inside the silo bags. Temperature and CO₂ locations were also clustered into these three groups. A three-factorial design model (moisture content, storage time and sample location) was

used for ANOVA test and SAS 9.3 software was used for statistical analysis (SAS, 2012). The moisture contents were the three initial moisture contents of the canola seed. The sample locations were the top, middle, and bottom layers.

3.2. Effect of storage time

For testing the effect of storage time (length of storage) on the canola seed quality during storage in silo bags under Canadian Prairie conditions (Objective 2), canola with 12% moisture content(wet basis) were stored in silo bags for 10 months in each of the 2 storage years (2011-12 and 2013-14).

3.2.1. Canola

For the first year study (2011-12), canola (Grade: Canada No.1) with 12% moisture content were received from Richardson Pioneer grain elevator, Dauphin, Manitoba on September 29, 2011, and loaded into three bags. Each bag was loaded with approximately 67 tonnes of canola using the grain bag loader, and lengths of all three bags were 21.33 m (70 ft). Bags used for this study were similar to the bags used in our previous objective (2.74 m diameter and 238 μ m thickness bags, with 10% stretching capacity while loading, Manufacturer: IpesaSilo, Ciudadela, Argentina). For the second year experiment (2013-14), 12% m.c canola was received from the same grain elevator on October 11, 2013. The bag dimension, canola grade and loading method were the same as that conducted in the first year study. To prevent the damage to silo bags from mice and other rodents, agricultural lime (calcium carbonate) was spread on the ground before silo bags were loaded with canola (Figure 3.8).

For testing the effect of storage time on canola seed quality, three unloading times were chosen to represent: (i) after 20 weeks of storage (unload in late winter when ground was frozen); (ii) after 28 weeks of storage (unload at the start of summer when

ground was thawed and accessible); and (iii) after 40 weeks of storage (unload at the end of summer). At each time, one bag was unloaded using the grain bag extractor. In first year, bags were unloaded on March 1, May 3, and September 8, 2012, respectively. Due to the long and cold winter in 2013-14, bags were unloaded on April 3, June 5 and July 29, respectively in the second year experiment.



Figure 3.8 Application of agricultural lime on the ground during loading of silo bags

3.2.2. Grain quality assessment, temperature and intergranular CO₂ and O₂ measurement

Seed samples were collected similar to the method used in section 4.1 with modification. The modification was to cover the expansion Styrofoam with another layer

of grain bag tapes. This covering eliminated the risk of accumulation of snow and water at the top of Styrofoam at the side ports. To check the effect of location along the length of bag (head, centre and tail portions) on canola quality parameters, seed samples were collected at three different locations along the length of each bag: 4.6 m from the tail (referred to as T), middle (referred to as C), and 4.6 m from the head of the bag (referred to as H) (Figure 3.9). At each location (T, C and H), three sample ports were made (one on each side, and one at the top), and seven seed samples were collected using the Boxcar probe (Figure 3.9). The sampling code was the same as that conducted in effect of storage moisture content experiment (explained in section 3.1). The same protocols mentioned in section 4.1 were used to determined quality parameters.

From each bag, temperature measurement and gas sample collection were carried out at 27 locations. Temperature monitoring was done using data acquisition systems (Model: 4970A Agilent Technologies Inc., Santa Clara, CA). Gas samples collected from the gas sampling locations were analysed using the gas chromatograph (Model: Clarus 420, Perkin Elmer, Woodbridge, ON) with a 2.78 m (9 ft) 13x molecular sieve 40/60 mesh column and a 1.85 m (6 ft) Hayesep N 60/80 mesh column (2 mm internal diameter) for determination of O₂ and CO₂ concentrations, respectively. The detector was a thermal conductivity sensor (Shunmugam et al., 2005).

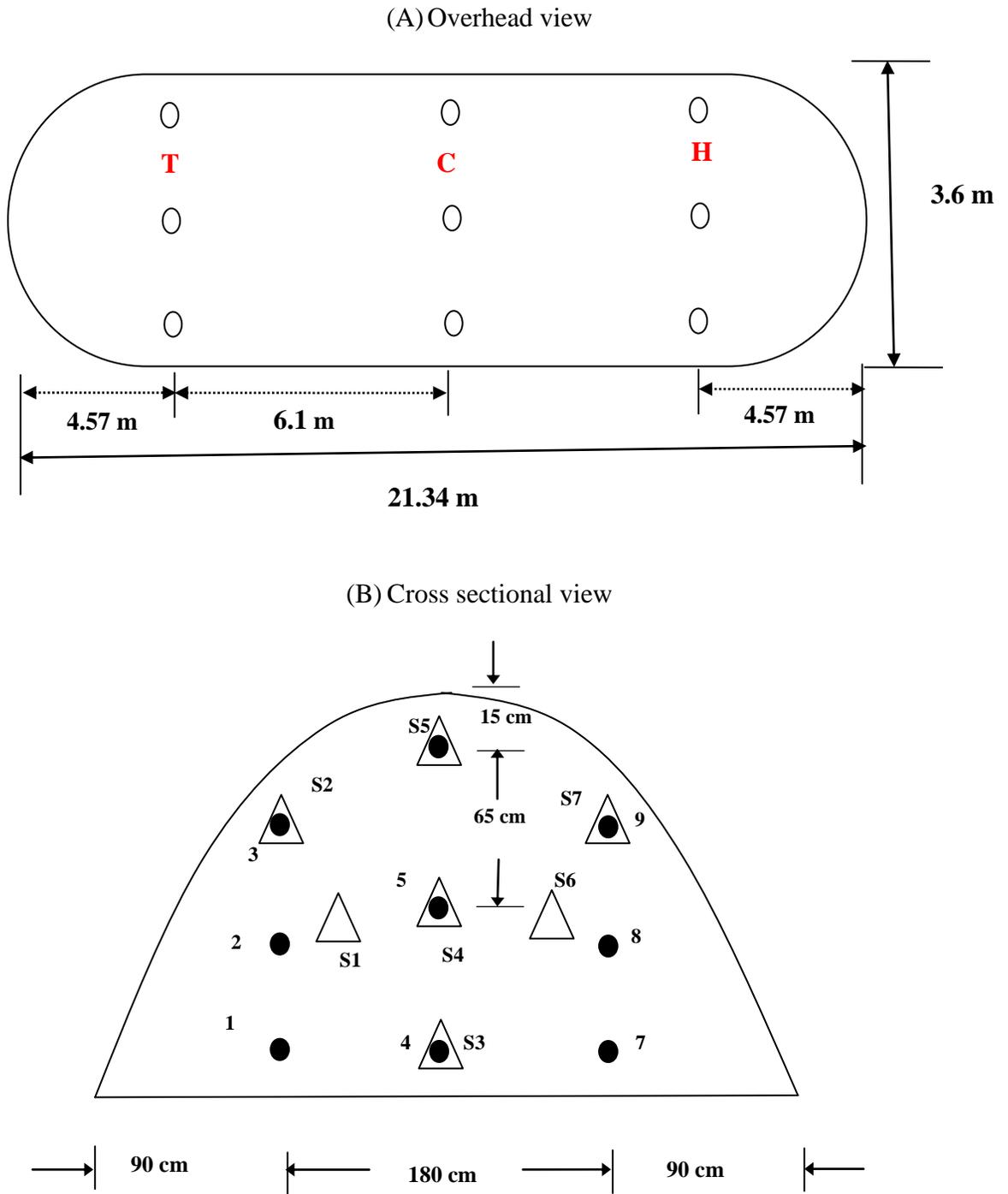


Figure 3.9 Plan and cross sectional view of bags with sampling locations (Δ S - seed sampling locations; \bullet - temperature measurement locations and CO_2 collection locations)

3.2.3. Statistical analysis

Analysis of variance (ANOVA) test was used to determine the effect of storage time on canola seed germination, FAV, CO₂ and O₂ levels inside silo bags, and changes in moisture content of canola during storage. The comparison of means was carried out using Tukey's method ($\alpha=0.05$). At each location along the length (T, C and H), seed sample were grouped into top (S2, S5, S7), middle (S1, S4, S6), and bottom (S3), and a total of nine groups per bag (T-Top, T-Middle, T-Bottom, C-Top, C-Middle, C-Bottom, H-Top, H-Middle and H-Bottom) were made for the analysis. A three factorial model (location along the length of the bag (3 levels (H, C and T), layer of grain (three levels (Top, Middle, and Bottom), and storage period (20 levels)) were used for ANOVA tests using SAS 9.3 software (SAS, 2012).

Temperature and moisture profiles of canola at the head, centre, and tail parts at different seasons (fall, winter, spring, and summer) were created using SURFER software (Version 12, Golden Software Inc, Golden, Colorado) (Surfer, 2012). Temperatures at noon on October 10, 2011; January 15, 2012; May 1, 2012 and July 31, 2012 at top, middle and bottom layer canola in the head, centre, and tail parts were used to create temperature profiles of fall, winter, autumn and summer seasons, respectively. Moisture content profile maps at fall, winter, autumn and summer seasons were created using moisture content values measured on week 2, week 14, week 28, and week 36, respectively.

3.3. Prediction of CO₂ level inside silo bag

3.3.1. Model development

Carbon dioxide concentrations measured inside the silo bags containing 8.9, 10.5 and 12.4% initial moisture content canola during 40 weeks of storage were used to develop the CO₂ prediction model. Similar to previous studies (White et al., 1982; Proynk et al., 1994), initial moisture content of the canola, storage time (day of storage), grain temperature during loading, and monthly average temperature were used as independent variables to develop the regression model to predict the CO₂ level inside the silo bag. SigmaPlot 13 software was used for the analysis of data and regression model development. The following assumptions were made while developing the prediction model:

- CO₂ concentration was uniform throughout the bag.
- Mean moisture content of canola remained same as initial moisture content.
- The bag material was impermeable.
- CO₂ produced by mould and insects were not taken into account.
- CO₂ concentration $\geq 21\%$ was considered as 21%; and concentration $\leq 0\%$ was considered as 0%.

3.3.2. Model validation

Carbon dioxide concentration data collected from the effect of the storage time experiment (2013-14) was used to validate the developed regression model. The

CO₂ levels also predicted by previously developed CO₂ production models by White et al. (1982), and Proynk et al. (2004) (Eqn.2 and 3) were compared with these validation results. The results of effect of moisture content and effect of storage time experiments proved that there was moisture migration inside silo bags due to temperature gradient. So the measured moisture content values of the 2013-14 study were given as input for the developed model for testing the sensitivity of the regression model.

3.4. Measurement of permeability of silo bag material

A permeability testing setup was designed and fabricated based on the setup used by Villaluenga and Seoane (2000) for testing permeability of low-density polyethylene films. Measurement of permeability was carried out in a static phase, which represents the real time environment. The testing cell has two identical cylindrical compartments made up of stainless steel (Figures 3.10 & 3.11). The internal diameter of the cylindrical compartment was 80 mm and the height of each compartment was 40 mm. The silo bag material was placed in between these two compartments and tightened with the screws at the top to ensure air tightness.

Testing gas (O₂ or CO₂ (99.9% purity) was purged into one compartment (testing chamber) and carrier gas (N₂ (99.9% purity) was purged into another compartment (receiving chamber). A leak test was performed using soap water at all the joints and connections. The inlet gas flow was controlled by a flow meter and a gas flow of 0.3 L/s was maintained during purging. The gases were held in the testing and receiving cells for 2 days (48 h); and about 1.5 mL of gas samples were collected using 2 mL syringe at 0, 4, 8, 12, 24, 48 h after purging from the receiving cell. The collected gas sample was then analyzed using the gas chromatograph (Model: Clarus 420, Perkin Elmer, Woodbridge, ON). The pressures of testing and receiving cells were measured using a micro manometer (Model: AXD 540, ALNOR products, Shoreview, MN) and the permeability was calculated using the following equation (ASTM, 2012; Singh and Heldman, 2009):

$$P = \frac{Ql}{At\Delta P}$$

Where,

P is the permeability of the material $\text{m}^3 \text{m m}^{-2} \text{d}^{-1} \text{atm}^{-1}$

Q is the concentration of O_2 or CO_2 in receiving compartment (m^3)

l is the thickness of film or barrier material (m)

A is the area of film or barrier material (m^2)

t is the time of steady state (d)

Δp is the pressure difference between compartments (atm).

The quantity of the O_2 or CO_2 in the receiving chamber (m^3) was calculated from the concentration of O_2 or CO_2 measured in ppm using the gas chromatograph. Five types of bag materials were tested in five replicates:

- New material - before loading any grain into silo bag;
- From the side of a silo bag (which has maximum stretch (10%)) after 7 months of canola storage;
- From the folded tail portion of a silo bag (no stretch) after 7 months of canola storage;
- From the side of a silo bag (which has maximum stretch (10%)) after 10 months of canola storage;
- From the folded tail portion of a silo bag (no stretch) after 10 months of canola storage.

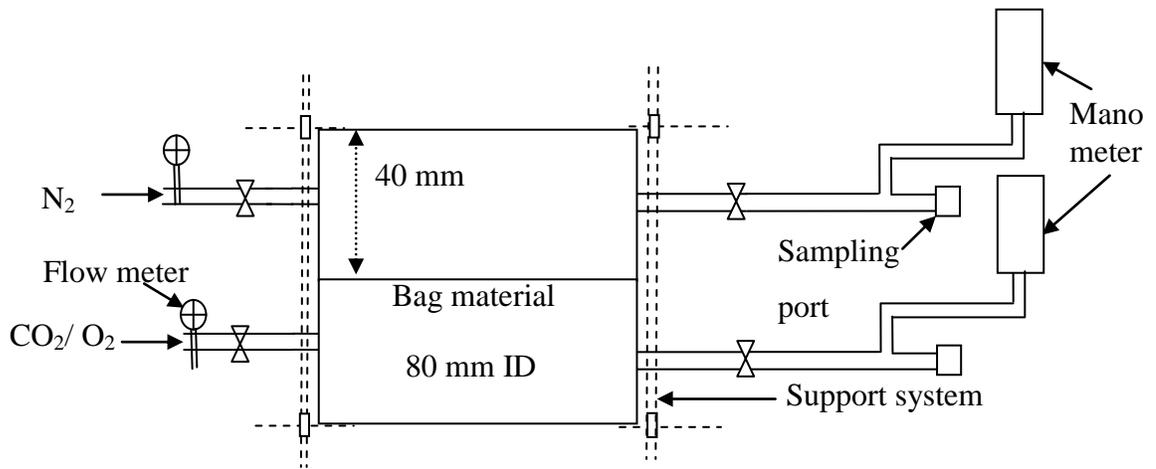


Figure 3.10 Schematic view of permeability measurement system

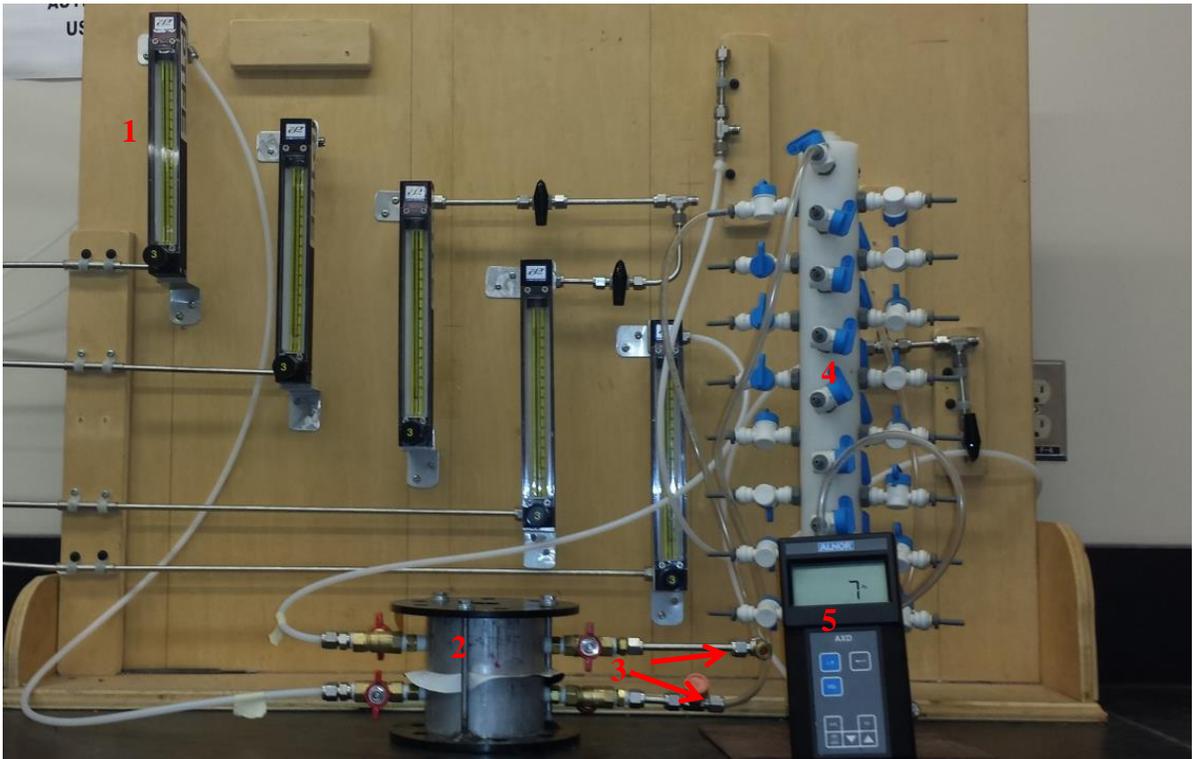


Figure 3.11 Permeability testing system (1) flow meter; (2) test cell; (3) gas sampling ports; (4) multiport chamber; (5) micro manometer

Chapter 4

Results and discussion

4.1. Effect of storage moisture on quality of canola

4.1.1. Results

4.1.1.1. **Moisture content**

The initial moisture contents of canola delivered from the elevator were $8.9\pm 0.2\%$ (referred to as dry moisture bag), $10.5\pm 0.3\%$ (referred to as straight moisture bag), and $14.4\%\pm 0.1\%$ (referred to as damp moisture bag). In dry moisture bags, the moisture content of the canola at the top layer was higher than that at middle and bottom layers of bags for up to 28 wk of storage (Fig. 4.1). In dry moisture bags, the moisture gradient at different parts of the bags stayed within one percentage point throughout the storage periods, but damp moisture bags experienced larger moisture gradients, especially after 28 wk of storage. The storage initial moisture and storage time and sampling location had

a significant effect ($p \leq 0.008$) on changes in moisture content inside the silo bag during storage. There were no significant differences in moisture content between the top and middle layers of the bag ($\alpha = 0.05$), but there were significant differences between the top and bottom, as well as the middle and bottom layer of the bag due to hotspot development. The moisture migration inside the bags due to temperature gradients between the top and bottom layers of the grain profile was noticed in all three moisture content canola bags from the moisture profile graphs (Figs. 4.2- 4.7). In winter, the moisture contents of top layer of silo bags were 9.5, 12.2, and 15.6% in dry, straight and damp moisture content silo bags, respectively, but in summer, the top layer moisture contents were 7.9, 9.8, and 13.5%, respectively.

4.1.1.2. Germination

In dry moisture bags, germination of the seeds at all sampling locations was more than 90% even at the end (40 wk) of the storage (Fig. 4.8). The seed germination was more than 80% at all sampling locations, except three locations near the top of the straight moisture bag. A localized hotspot was found at the middle portion of a bag in damp moisture bags. Germination of seed decreased with increased storage time in damp moisture bags. Germination of canola in damp moisture bags decreased below 50% at top and bottom parts of the bags after 16 wk of storage. Germination of canola in dry and straight grade canola bags was always higher than that of damp canola bags, except the top portion of the straight grade canola bags. All the individual factors and their interactions had significant effects ($p < 0.001$) in change in germination of canola. Means

comparisons test showed that there were no significant changes in germination of dry canola up to 32 wk of storage, but the germination of canola was significantly affected after 12 wk of storage in damp canola bags ($p < 0.001$). Germination at the top layer of canola in silo bags was significantly lower than the middle and bottom layers of canola in straight and damp canola bags.

4.1.1.3. Free fatty acid values

Free fatty acid values increased from 23.2 to 25.9, 23.5 to 35.0, and 25.2 to 41.2 mg KOH/100 g of dry seed inside bags with dry, straight and damp grade canola, respectively, after 28 wk of storage (Fig. 4.9). In straight grade bags, FAV values were higher near the top of bags and correlated with the high moisture seeds near the top of bags. The storage time, storage moisture and their interactions had significant effects ($p \leq 0.005$) on FAV values, but the sampling location had no significant effect ($p = 0.171$) on changes in FAV. There was no significant change in FAV of dry canola throughout the storage time, but in 14% m.c. canola bags, it increased significantly after 8 wk of storage. In straight grade canola, there was significant difference between FAV value at the start and 8 wk of storage, but there was no significant difference between 8 and 40 wk of storage.

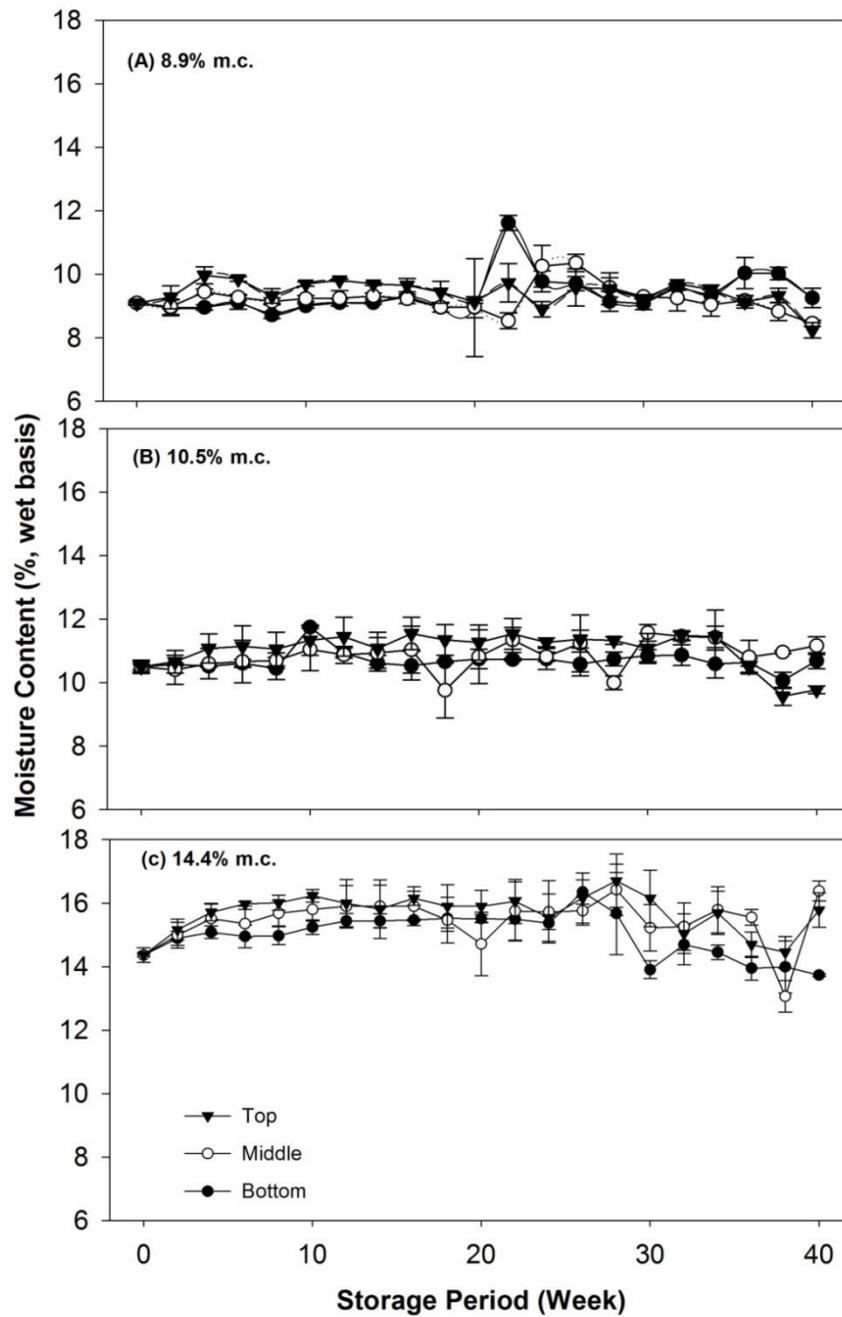


Figure 4.1 Moisture content of canola seed at different layers of silo bags containing 3 moisture levels of canola during storage (Loading: Oct 7&8, 2010; Unloading: Aug 10, 2011)

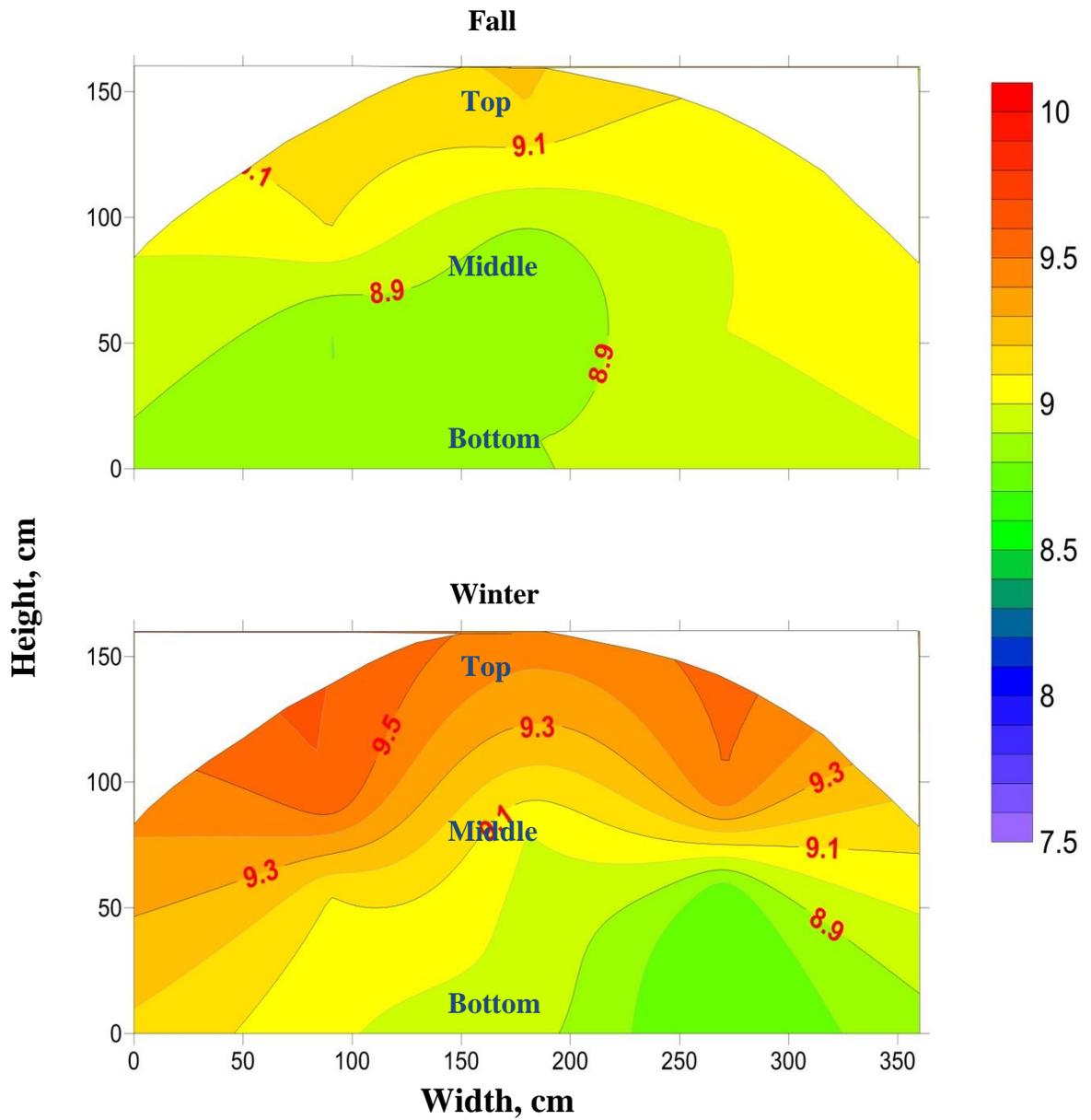


Figure 4.2 Moisture profile of silo bag with dry moisture content canola in fall and winter

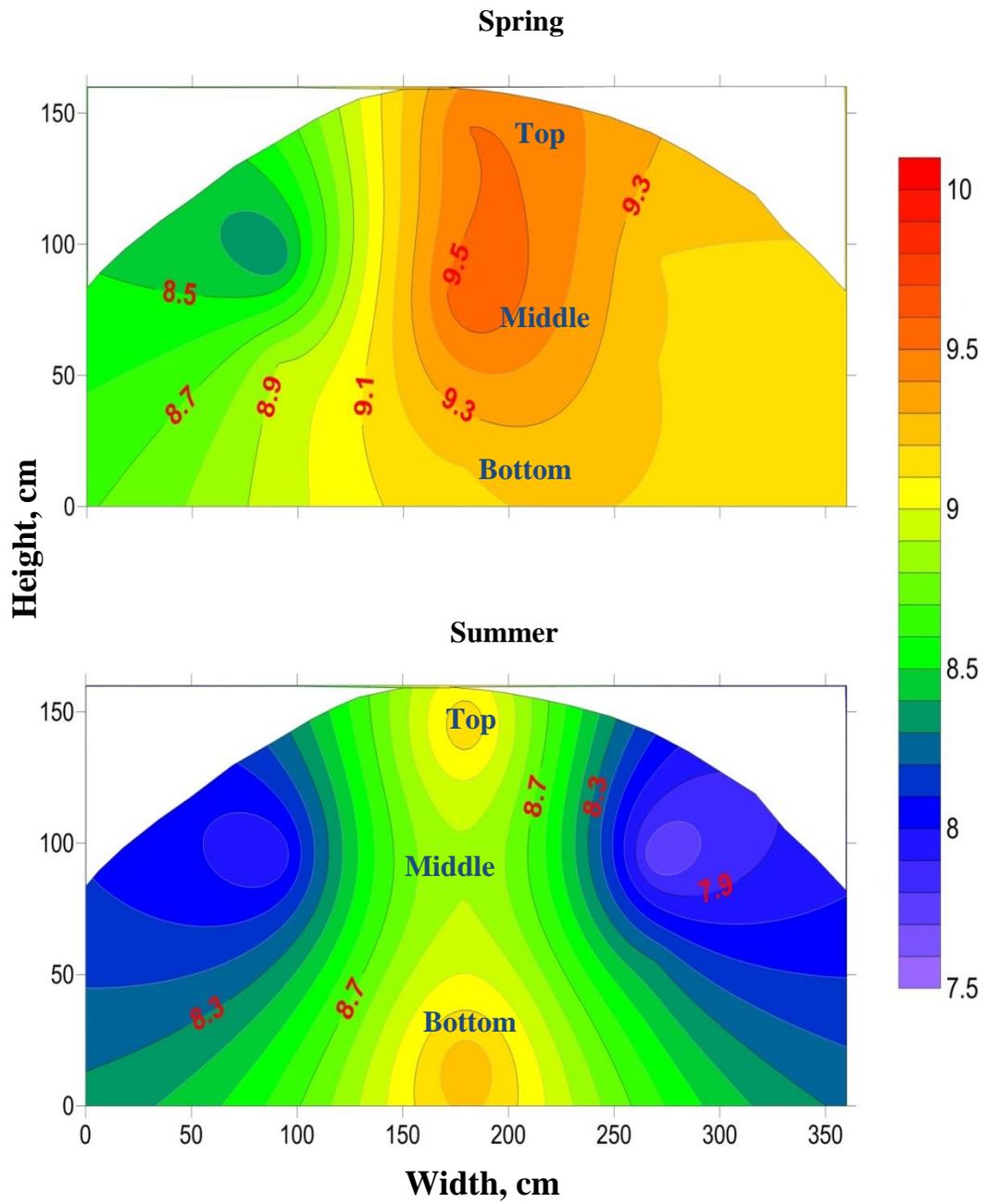


Figure 4.3 Moisture profile of silo bag with dry moisture content canola in spring and summer

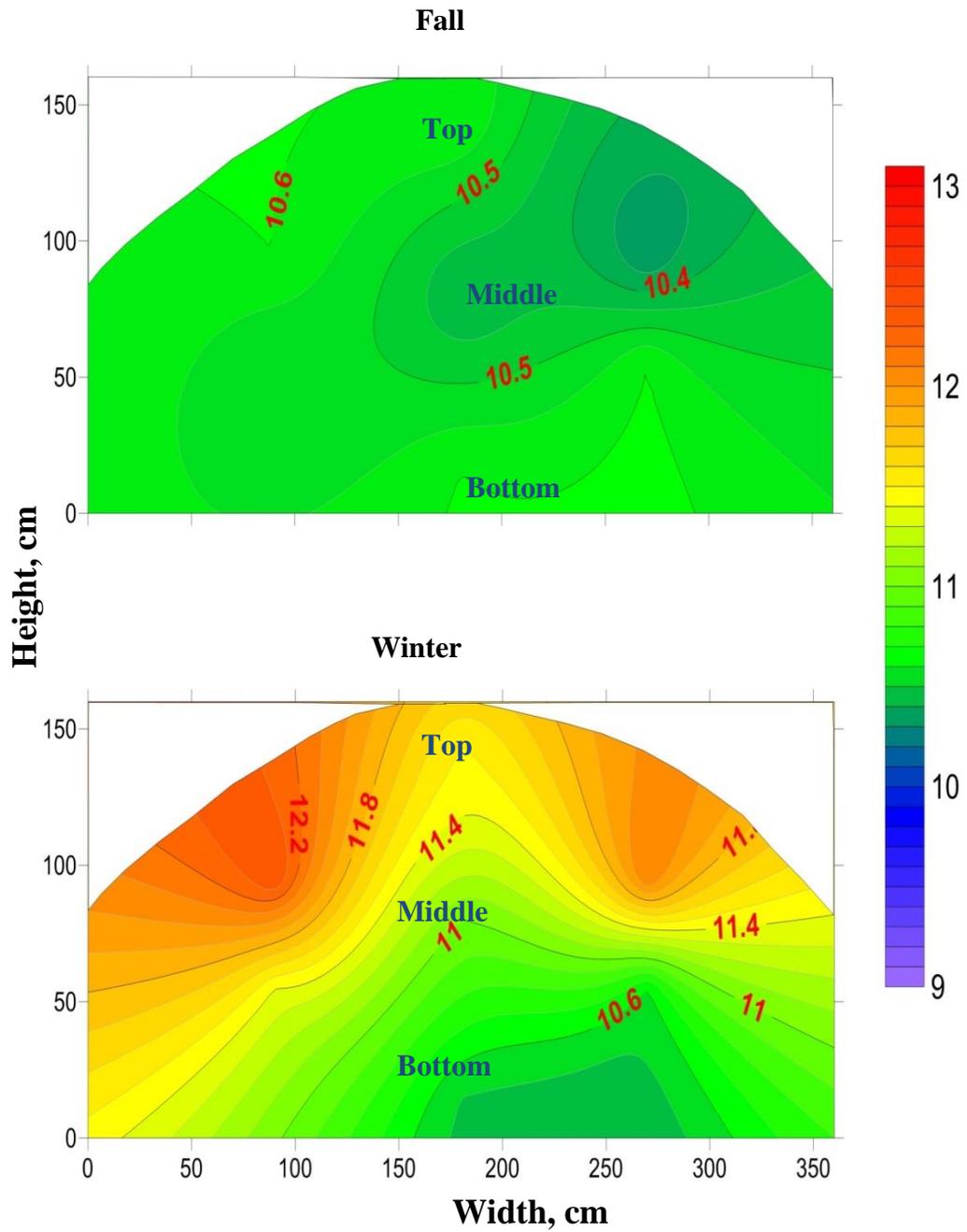


Figure 4.4 Moisture profile of silo bag with straight moisture content canola in fall and winter

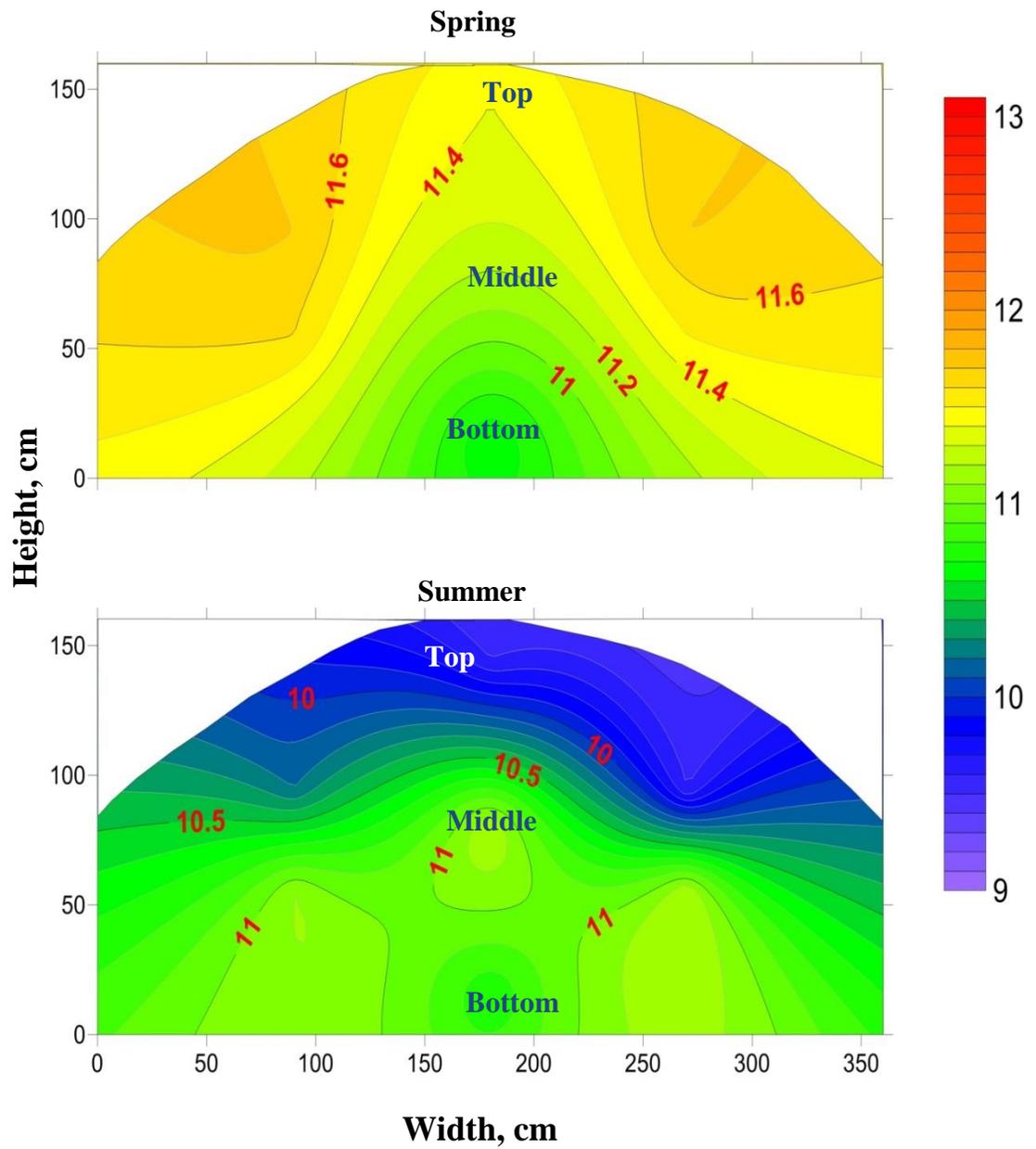


Figure 4.5 Moisture profile of silo bag with straight moisture content canola in spring and summer

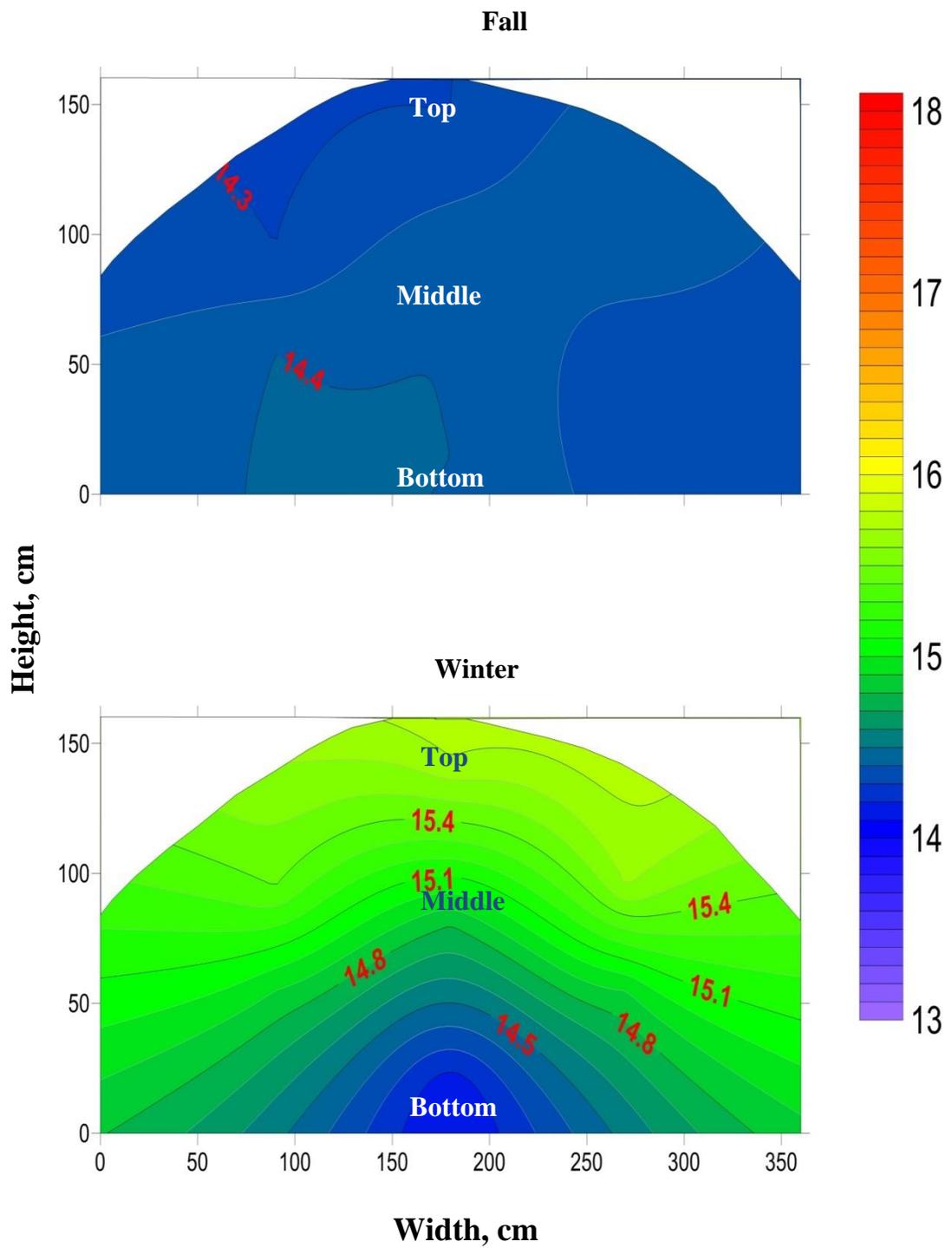


Figure 4.6 Moisture profile of silo bag with damp moisture content canola in fall and winter

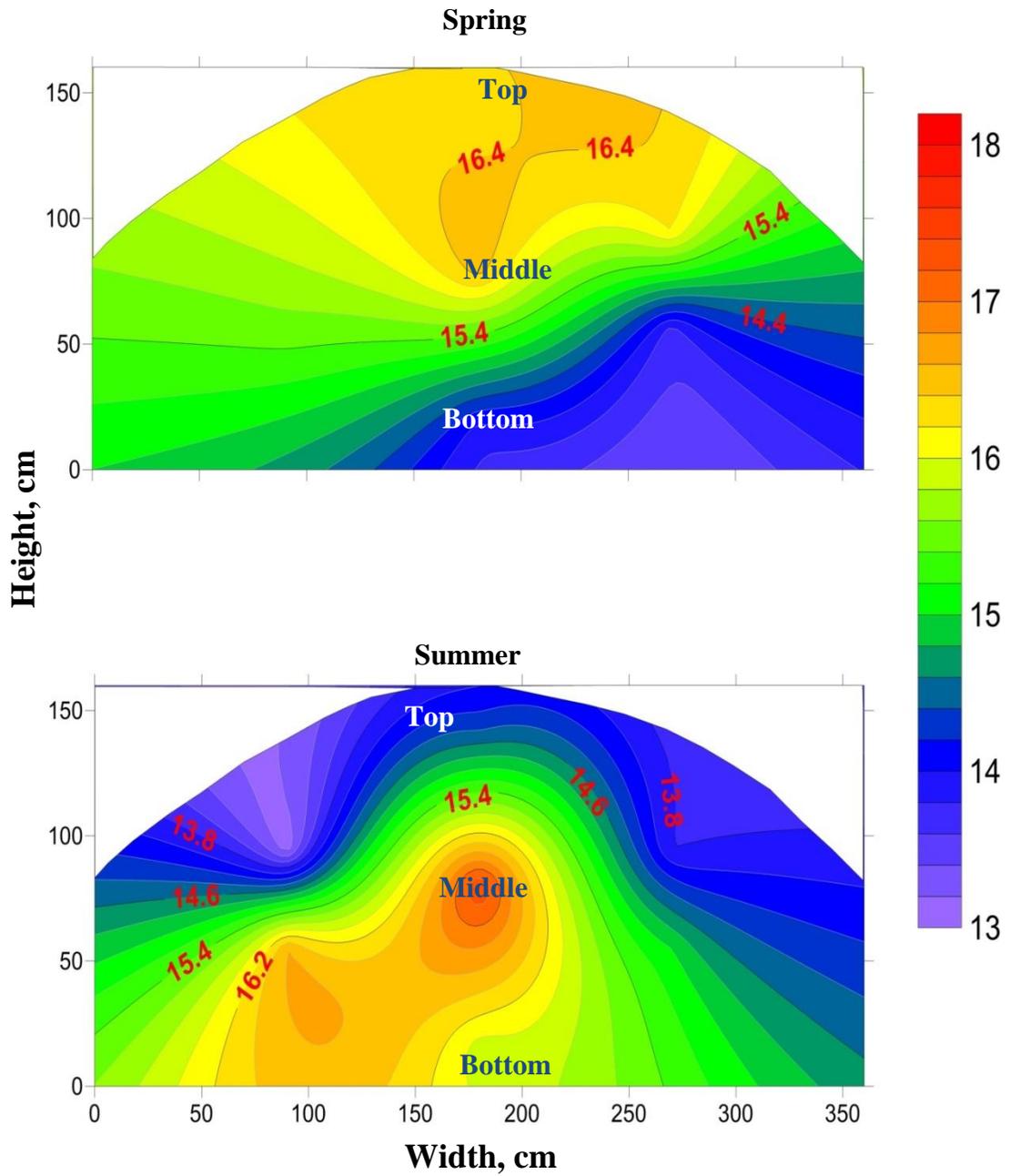


Figure 4.7 Moisture profile of silo bag with damp moisture content canola in spring and summer

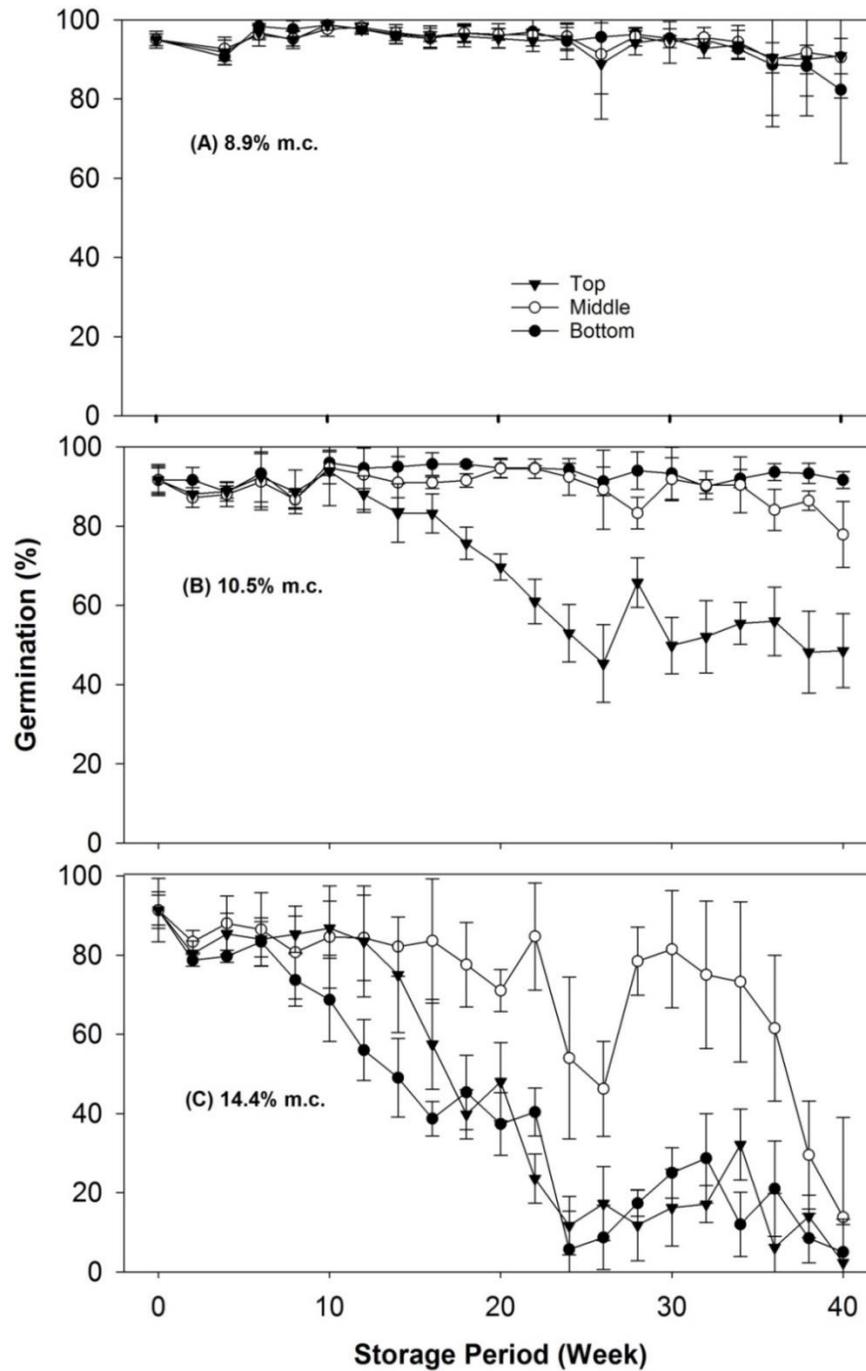


Figure 4.8 Germination of canola seed at different layers of silo bags during storage

(Loading: Oct 7&8, 2010; Unloading: Aug 10, 2011)

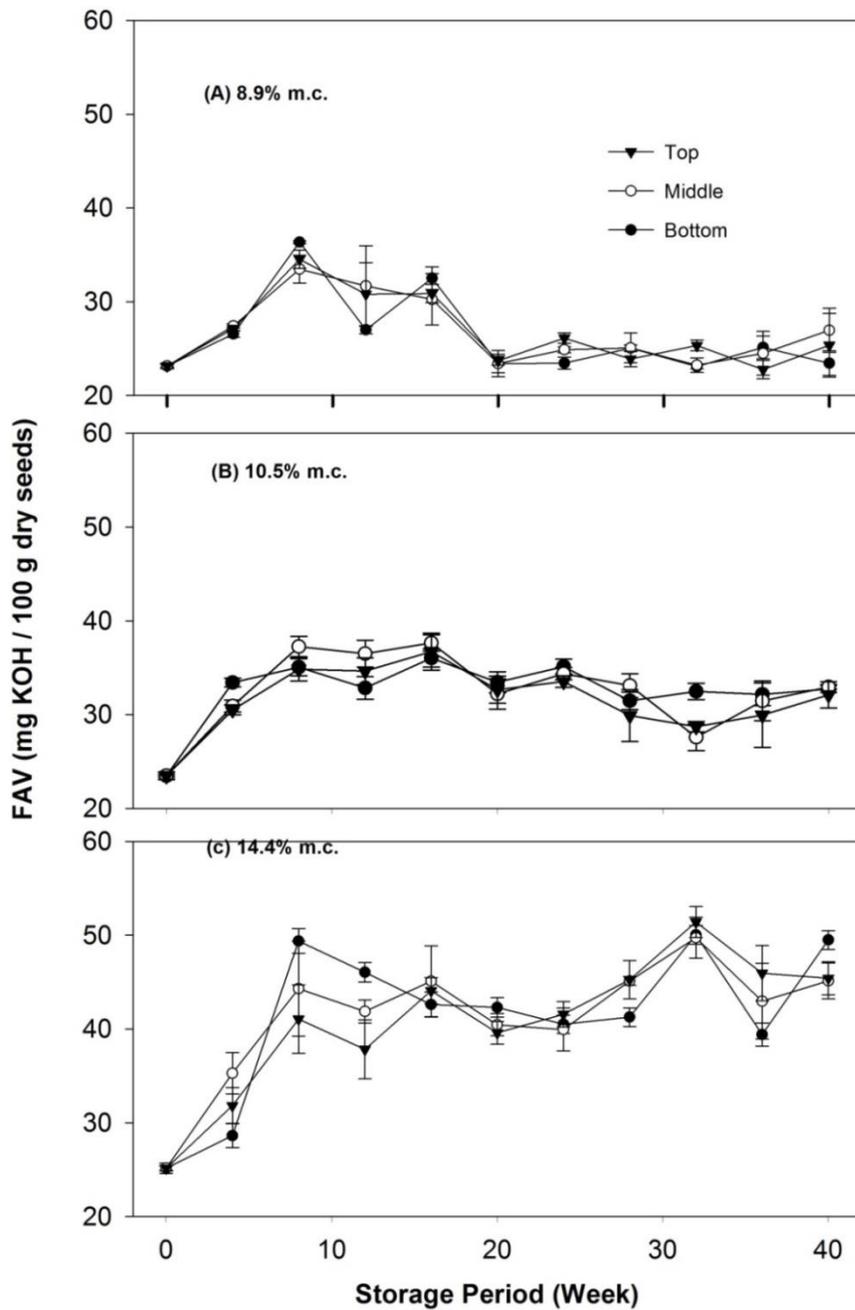


Figure 4.9 FAV of canola seed at different layers of silo bags during storage
(Loading: Oct 7&8, 2010; Unloading: Aug 10, 2011)

4.1.1.4. **Temperature**

In dry and straight grade canola bags, seed temperature near the bottom of bags was higher than other parts of the bag during autumn and winter, and seed near the top of bags was hotter than other parts of the bags during spring and summer (Fig. 4.10). Seed temperature of the top layer followed the ambient temperature changes, and most of the storage period temperature difference between bottom and top layers was about 15°C. Temperature pattern of damp grade canola bags was different than the other two drier canola bags, and hotspots developed inside the damp grade canola bags and could be the reason for this change in temperature pattern. Even in mid-winter, the temperature of the top layer of the damp grade canola bags stayed above freezing, and the middle layer of the canola followed the ambient temperature during winter. The temperature profile graphs show that temperature of the bottom layer of the grain was 4°C on January 15, 2011, and at the same time the temperature of the top layer of canola was -13°C (Figs. 4.10-4.15). This trend was the same in all three moisture content canola bags. The temperature profile map also shows a hot spot in the damp moisture canola bag, which might be the reason for caking of canola.

4.1.1.5. **Carbon dioxide concentrations**

The CO₂ concentrations of dry, straight, and damp grade canola bags were 3.6-4.0, 7.5-9.4, and 19.0-20.9%, respectively, after 4 wk of storage (Fig. 4.16). The higher CO₂ concentrations in the damp grade canola bag were probably due to the high amount of biological activity in the wet seed bulk. After 40 wk of storage, CO₂ concentrations

were 1.3% or less in the two dryer bags and above 2.9% in the damp canola bags. The storage moisture and storage time had significant effects ($p < 0.001$) on changes in CO₂ concentrations, but the sampling location did not ($p = 0.542$). The interaction between storage moisture and storage time also showed significant effects ($p < 0.001$) on CO₂ concentration changes. Some perforations made by rodents in all three moisture content bags were found during unloading, which may be the reason for drop in CO₂ concentrations after 8 wk of storage.

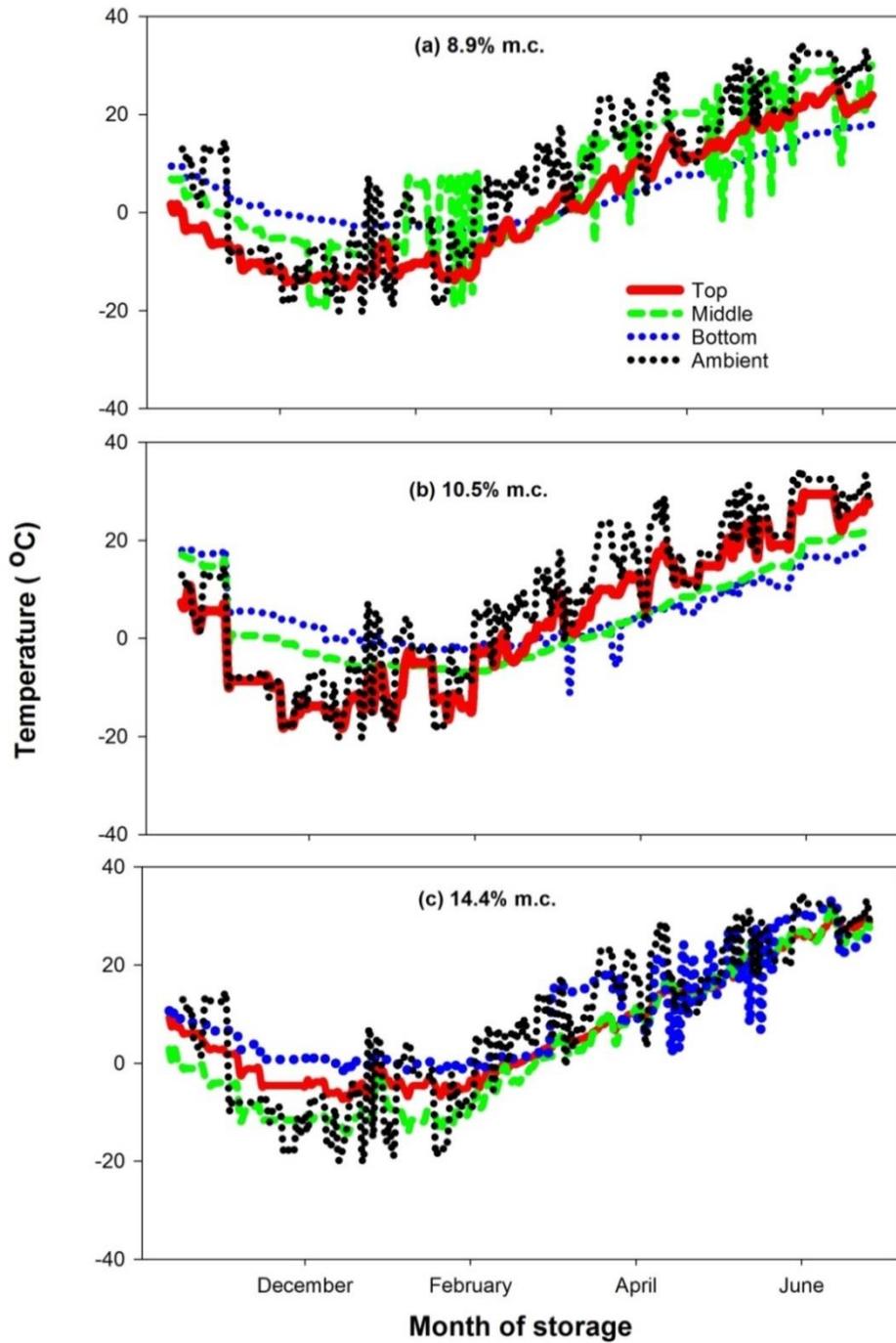


Figure 4.10 Temperature of canola seed at different layers of silo bags during storage (Loading: Oct 7&8, 2010; Unloading: Aug 10, 2011)

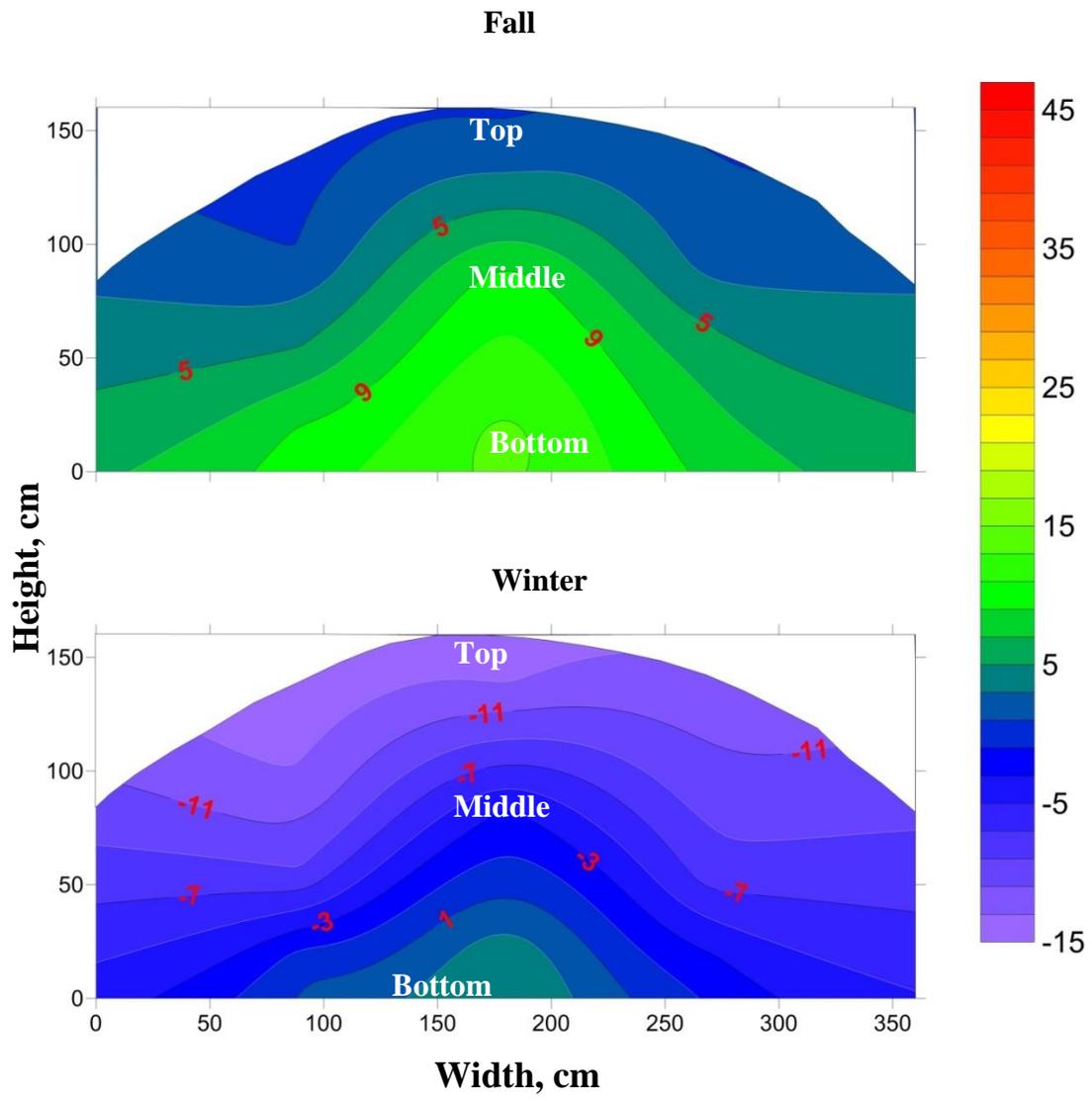


Figure 4.11 Temperature profile of silo bag with dry moisture content canola in fall and winter

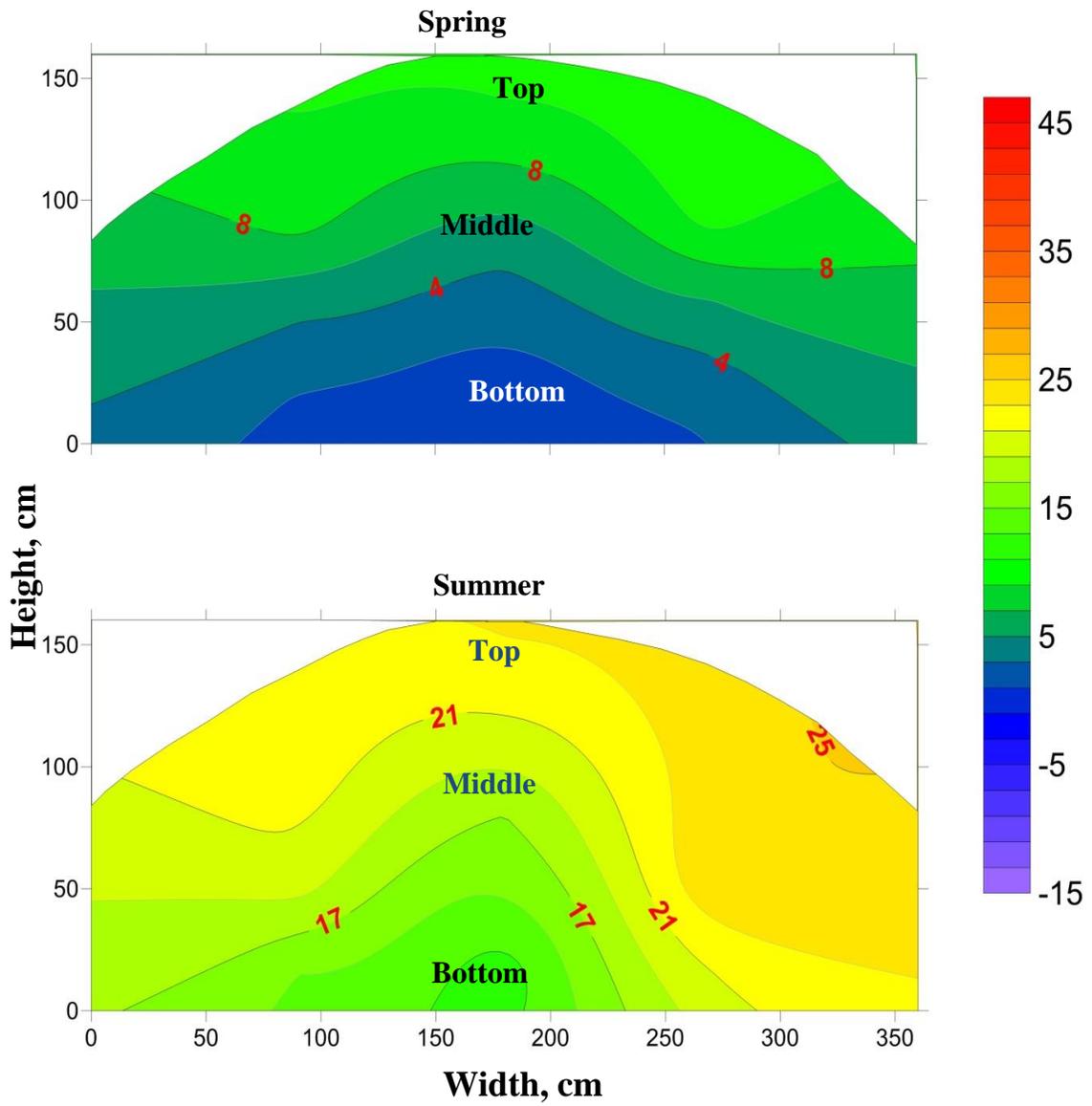


Figure 4.12 Temperature profile of silo bag with dry moisture content canola in spring and summer

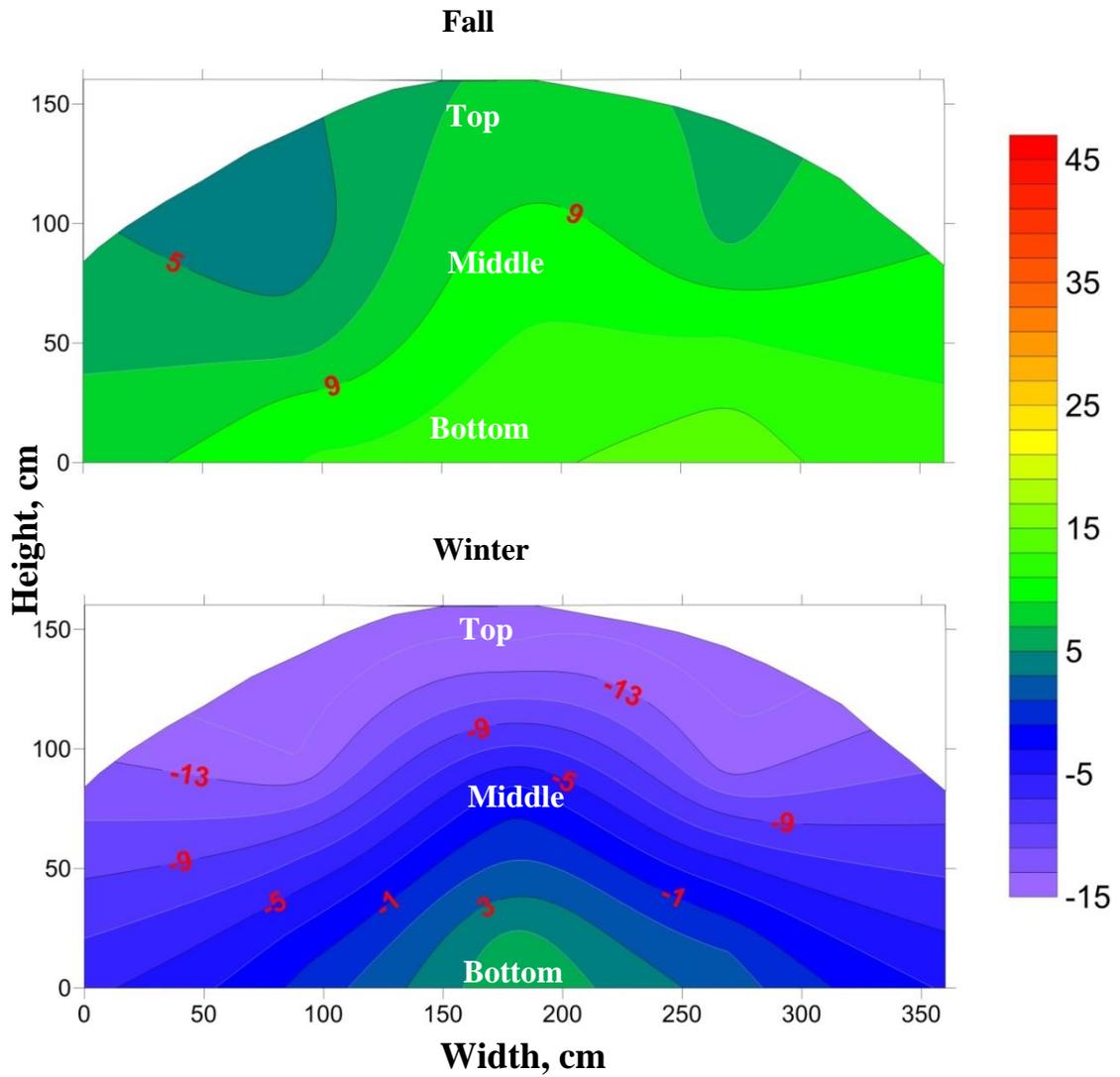


Figure 4.13 Temperature profile of silo bag with straight moisture content canola in fall and winter

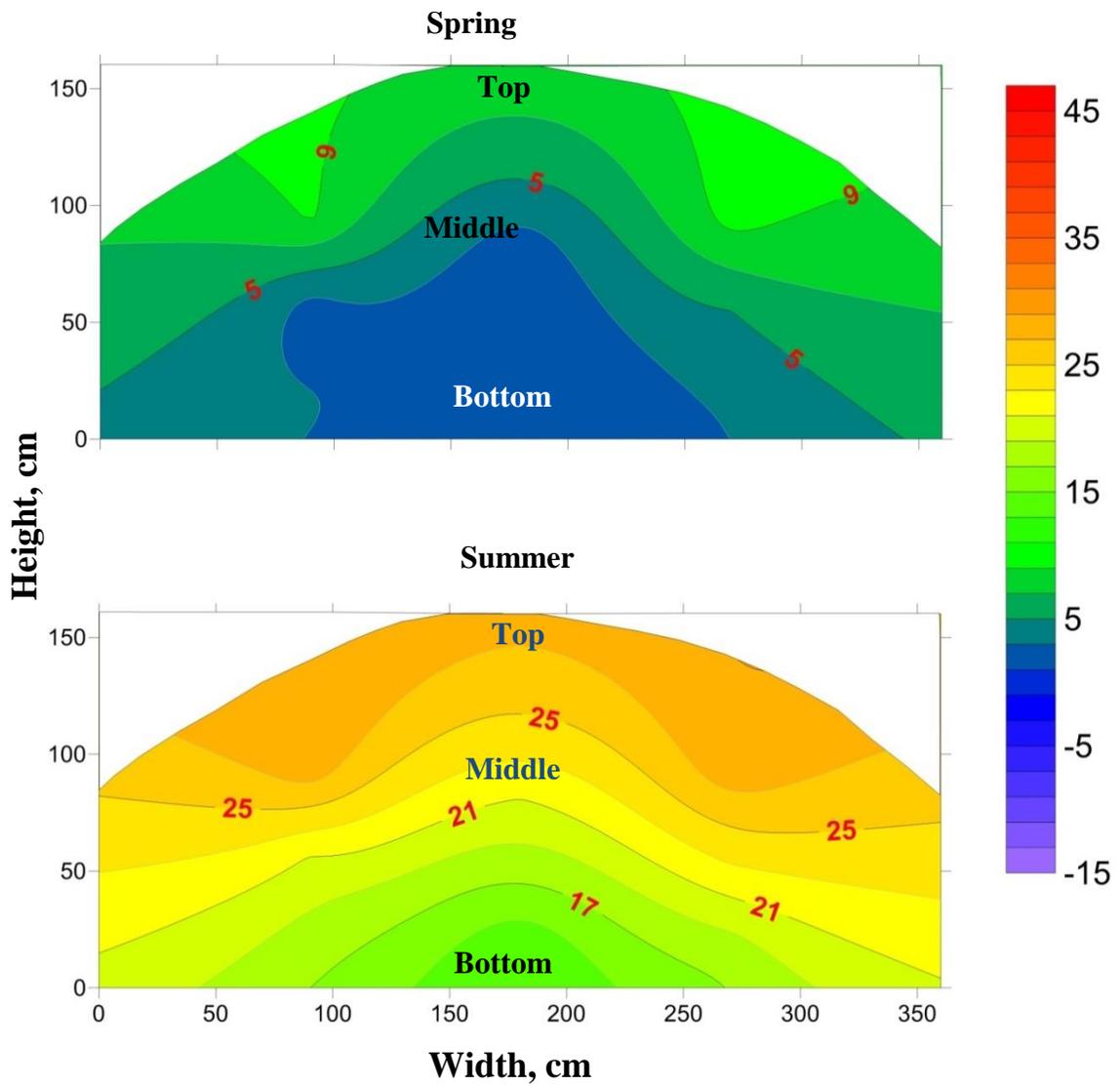


Figure 4.14 Temperature profile of silo bag with straight moisture content canola in spring and summer

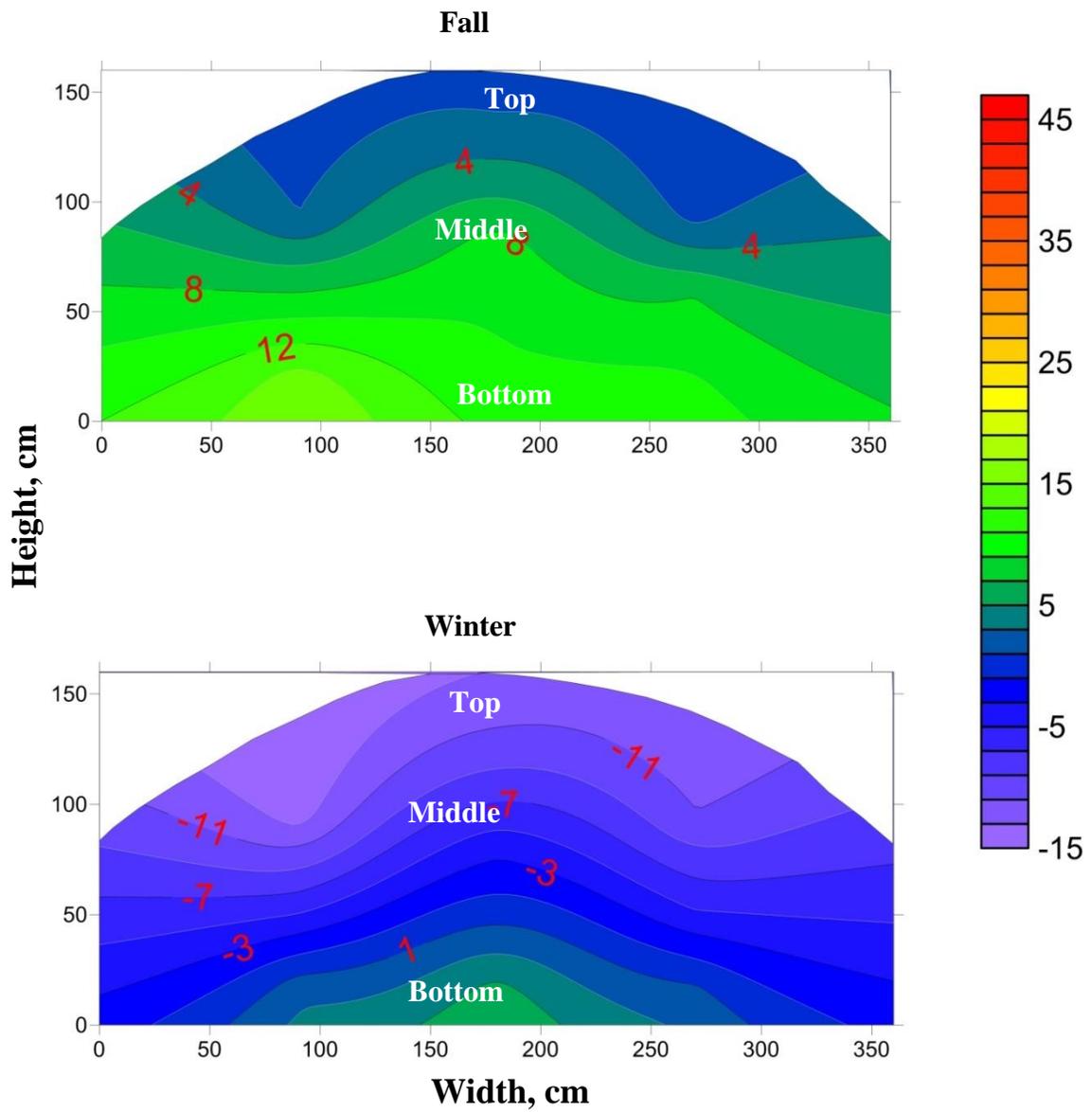


Figure 4.15 Temperature profile of silo bag with damp moisture content canola in fall and winter

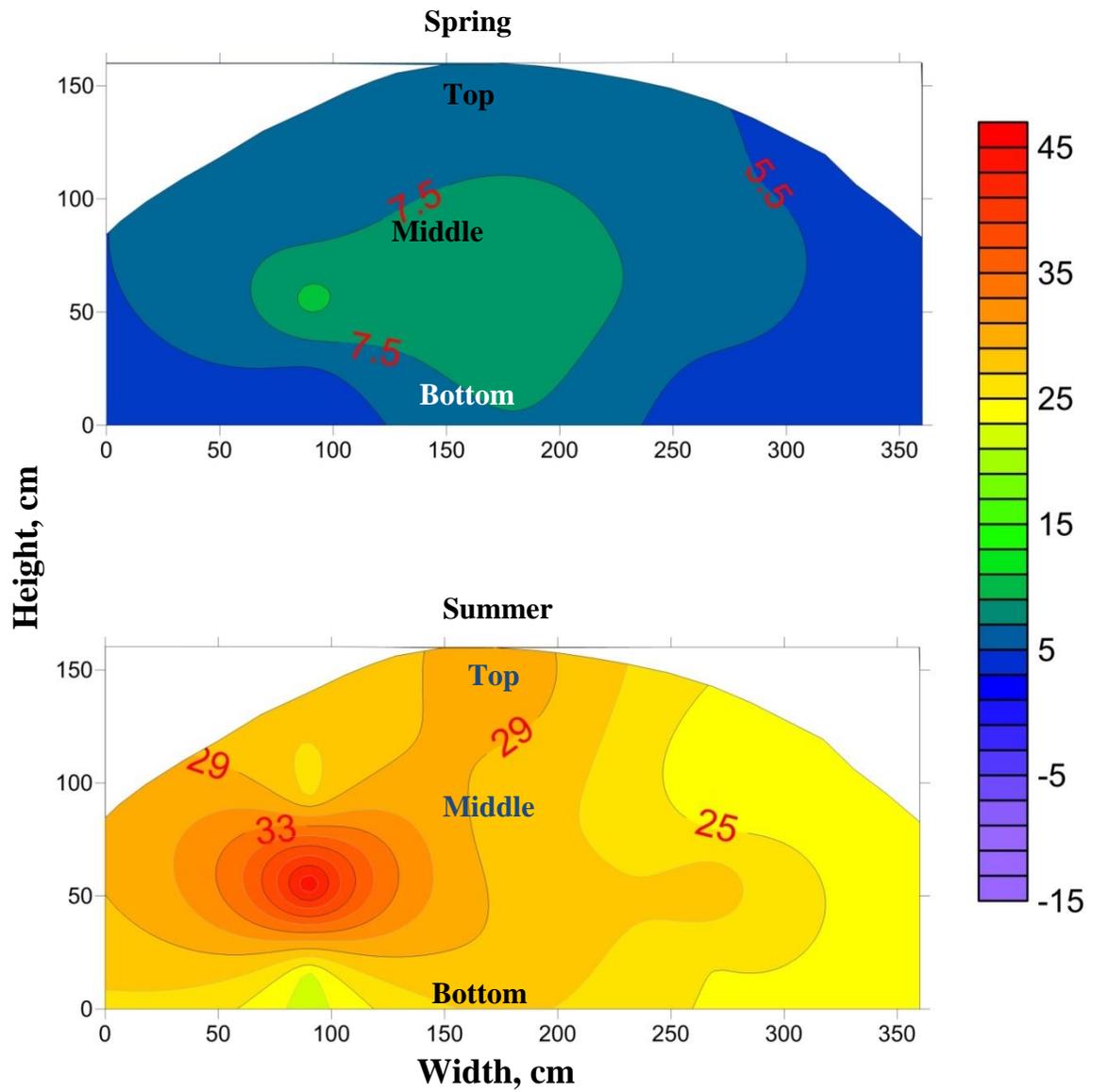


Figure 4.16 Temperature profile of silo bag with damp moisture content canola in spring and summer

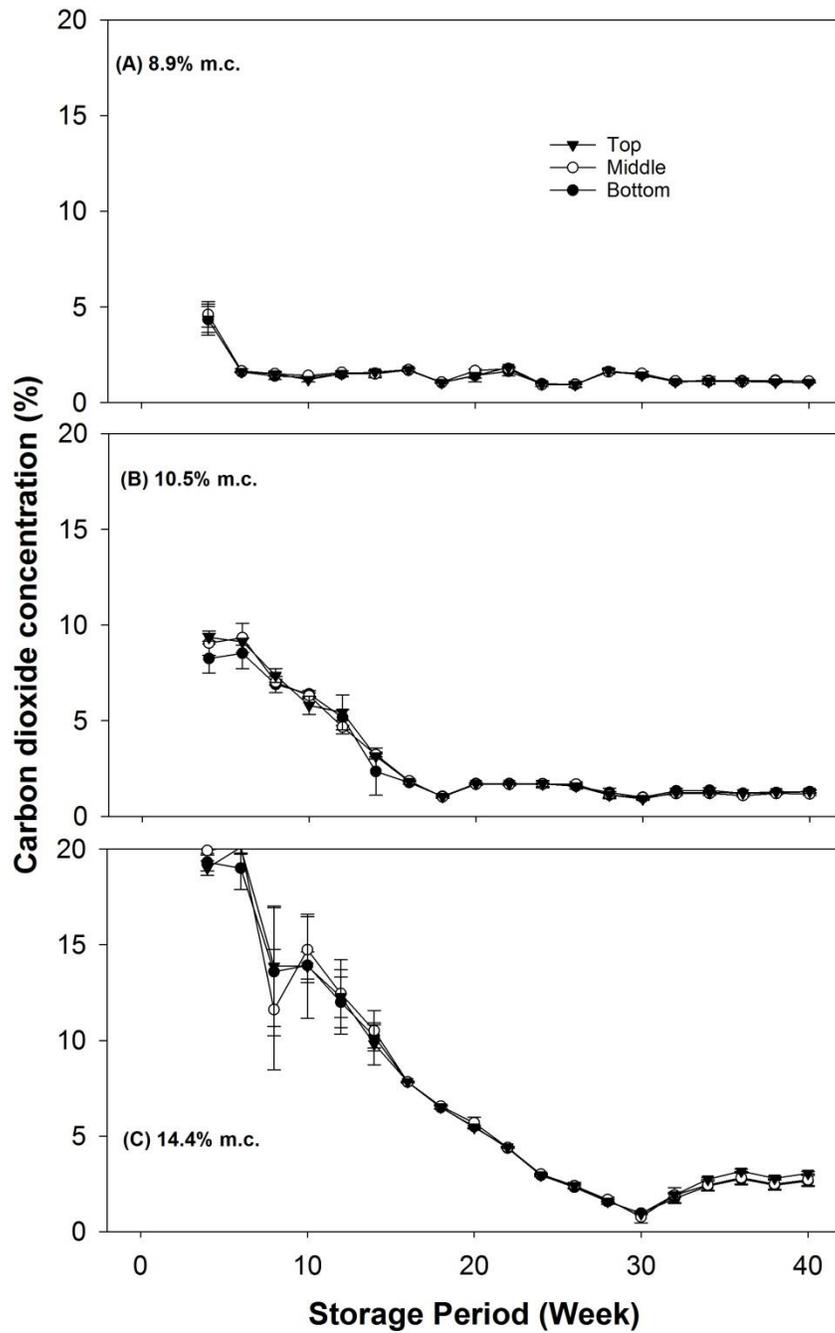


Figure 4.17 Intergranular CO₂ concentration at different layers of silo bags during storage (Loading: Oct 7&8, 2010; Unloading: Aug 10, 2011)

4.1.1.6. Commercial grading of canola

All three moisture content canola were graded as Canada Grade 1 at the beginning of the storage. After 40 wk of storage, dry and straight moisture content canola was graded as Canada Grade 1, and Canada Grade 2, respectively. Small amount of heated seeds were found in the top layer of straight grade canola bags, which reduced the grade. The damp grade canola was caked due to the high moisture and the grain bag extractor could not be used to unload the canola (Fig 4.18). The canola in damp grade bags was graded as Feed Grade.

4.1.2. Discussion

Our results show that seed moisture plays the major role in the deterioration of canola during storage. Previous studies also found that storage moisture and duration are the main parameters in determining safe storage conditions for canola and other grains (Bartosik, 2008; Sathya et al., 2009; Sun et al., 2014). According to safe storage guidelines of canola developed by Sathya et al. (2009), germination of canola with >80% of the initial germination can be considered as safe storage conditions. In our study, the dry grade canola had more than 90% germination throughout the 40 wk of storage in silo bags, which was more than 95% of initial germination. The increase in FAV content is the direct reflection of the deterioration of stored products by fungal infection (White and Jayas, 1991), and FAV has positive correlation with the storage moisture (Sathya et al., 2009). An increase of FAV by 1.5- fold of initial values was used as a threshold for deterioration of rapeseed (White and Jayas, 1991). In our study, the FAV of canola

increased more than 2-fold the initial value in damp grade canola bags, but the increase was less than 1.5-fold in dry and straight grade canola bags. The germination dropped and FAV increased in damp grade canola with hotspots and caking occurring. Christensen and Kaufmann (1969) reported that production of free fatty acid is directly proportional to the moisture content of the seeds and fungal activity. The lower FAV values of the dry moisture content canola bags indicate less biological activity in dry seeds.

The maximum CO₂ concentrations in dry, straight, and damp canola bags were 4.0, 9.4, and 20.9%, respectively, after 4 wk of storage. Canola has a high respiration rate for up to 6 wk after harvest (Thomas 1984). This may be the reason for the high concentrations of CO₂ in the first 8 wk. Ochandio et al. (2010a) also found the same trend in CO₂ concentrations of canola stored in silo bags under Argentinean weather conditions. Bartosik (2008) found that storing wet grains in silo bags created a favorable environment for mold growth and led to an increase in CO₂ and a decrease in O₂ concentrations. The mould development in grain reduced the grain quality. Barreto et al. (2013) analysed the quality changes of wheat in silo bags at three different climatic (sub-tropical, intermediate, and temperate) conditions in Argentina using mathematical modelling. Their results showed that increase in CO₂ concentration and depletion of O₂ inside the silo bags was mainly dependent on initial grain temperature and moisture content.

The moisture content of middle and bottom layers were significantly higher than the seeds at the top layer at the end of storage in dry and straight moisture bags. This

trend shows the accumulation of moisture due to condensation at the periphery of bags caused by temperature and moisture gradients during autumn and winter seasons, and in the summer the top layer grain was dried due to the hot ambient temperature. Our results also show a slight migration of moisture to the top layer of the bag in damp canola.



Figure 4.18 Canola seed with $14.4 \pm 0.1\%$ initial moisture content spoiled after 40 wk of storage

The profile graphs indicate the moisture migration between top and bottom layers of the canola due to the temperature gradient. In winter, hot and humid air from the bottom of the bag move up and get condensed at the top of the bag, which caused the increase in moisture at the top layer of the bags. This trend was similar in all three moisture content canola silo bags. The increase in moisture at the top layer might have

caused the localized hot spots, which might lead to fungal growth in damp moisture canola bags, once the temperature of the grain increased to favourable conditions during spring and summer. Even though in summer, the top layer of the grain dried up due to the moisture movement cycle and was in the opposite direction (from top layer to bottom), the hot spots developed during winter might have caused the spoilage of grain at the peripheral layer of the bag in damp moisture canola bags. This is similar to other studies. Barreto et al. (2013) found a slight increase of moisture content at the top layer of the bags filled with wet soy bean and wheat (16% m.c.) where the equilibrium relative humidity was above the safe storage levels throughout the storage period (even in colder months). Gaston et al. (2009) noticed a rise in wheat moisture content at the top layer of silo bags, and they concluded that the temperature gradients between top and other parts of the bag caused the moisture migration to the top layer of the bag. Jian et al. (2015b) found this was mainly caused by condensation and moisture migration. When compared with storage of grain in bins, a larger amount of seed is located at the periphery of the storage structure in a silo bag. So the temperature and moisture changes were large during storage, especially in the Canadian Prairie Provinces, where temperature differences between winter and summer can be 70°C (-40°C in winter and 30°C in summer). These large temperature fluctuations also lead to condensation at the top of bags (Jian et al., 2015b). Bartosik (2008) recommended that dry (below 76% ERH) grains (wheat, soybean, barley, corn, canola, and sunflower) can be stored for more than 6 months without any quality deterioration.

4.2. Effect of storage time on canola quality

4.2.1. Results

4.2.1.1. Moisture content

Initial moisture contents of canola in the first and second years were 12.1 and 12.4% (wet basis), respectively. In both years, there were no significant changes in moisture content between week 0 and week 20 in all parts of the bags. But significant changes in moisture content were noticed after 20 wk of storage between the bottom and top layers of the bags (Figs. 4.19-4.20). All the individual factors (seed layer (top, middle and bottom), and storage period) except sample location along the bag (head, centre, and tail) had significant effect on changes in moisture content ($P < 0.0001$). Means comparison proved there were significant differences between week 0 and week 20 in the second year, and also significant differences between the top and bottom layers of the bags ($\alpha = 0.05$).

The moisture content of the top layer of the silo bags was higher than the bottom layer due to moisture migration in winter at all three parts of the silo bag (head, centre, and tail), and in summer the top layer dried due to the warm autumn and summer temperatures (Figs. 4.21-4.26). These results were similar to the results of effect of moisture content study.

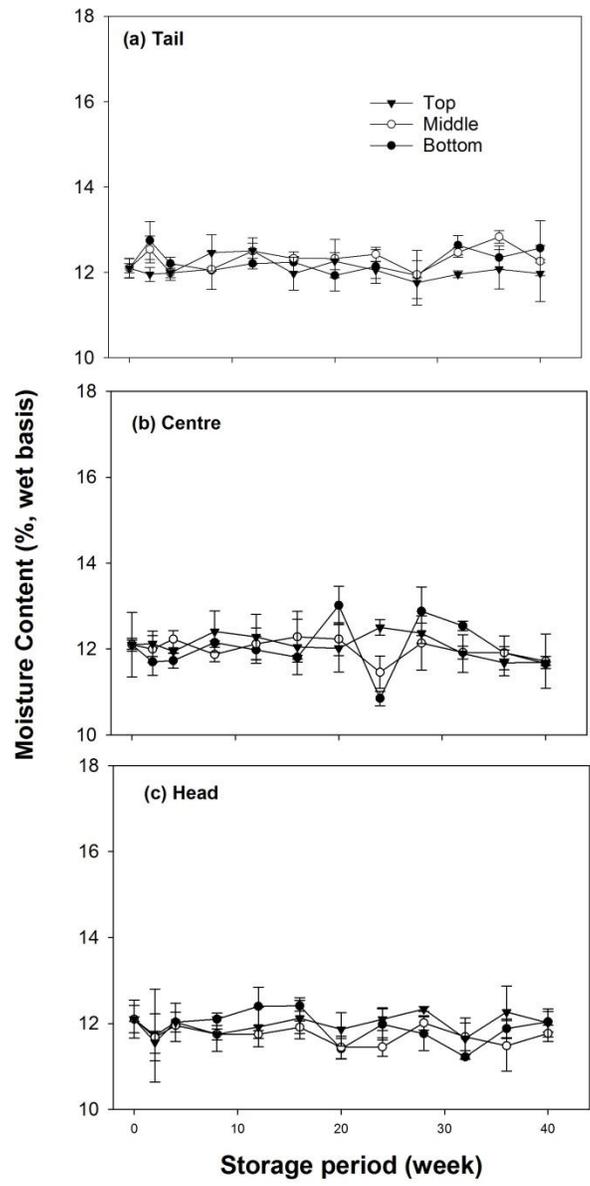


Figure 4.19 Moisture content of canola at different layers of silo bags in storage year 1 (2011-12)

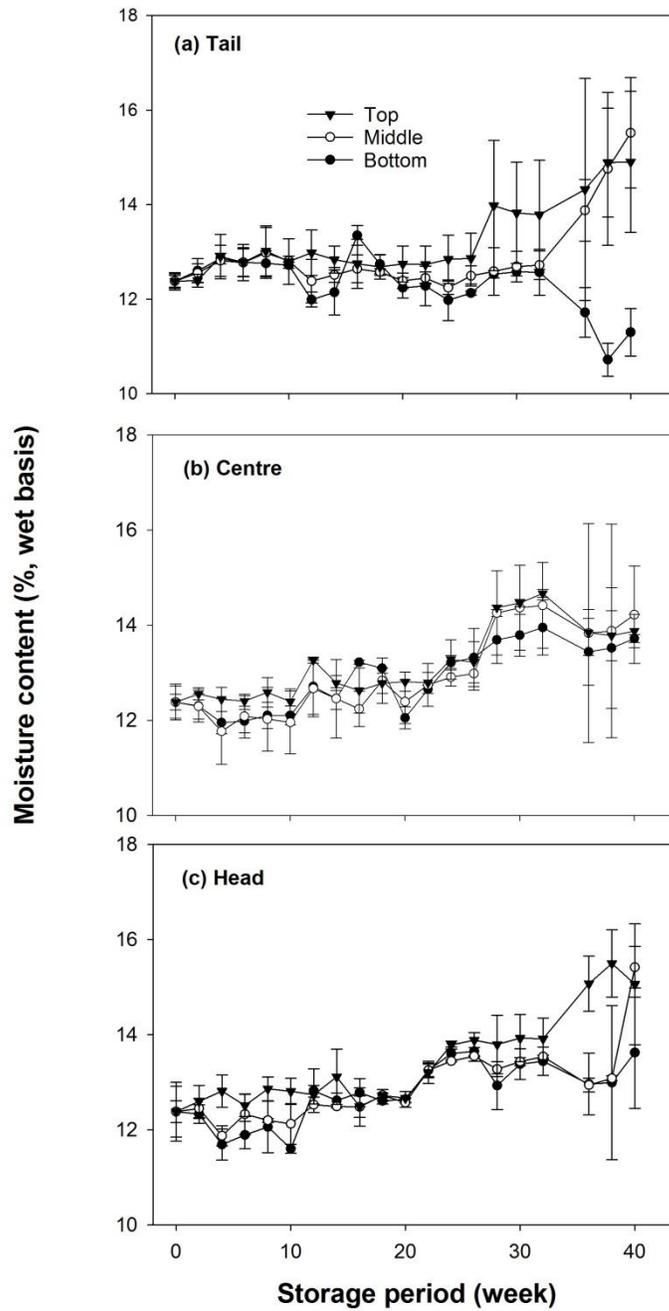


Figure 4.20 Moisture content of canola at different layers of silo bags in storage year 2 (2013-14)

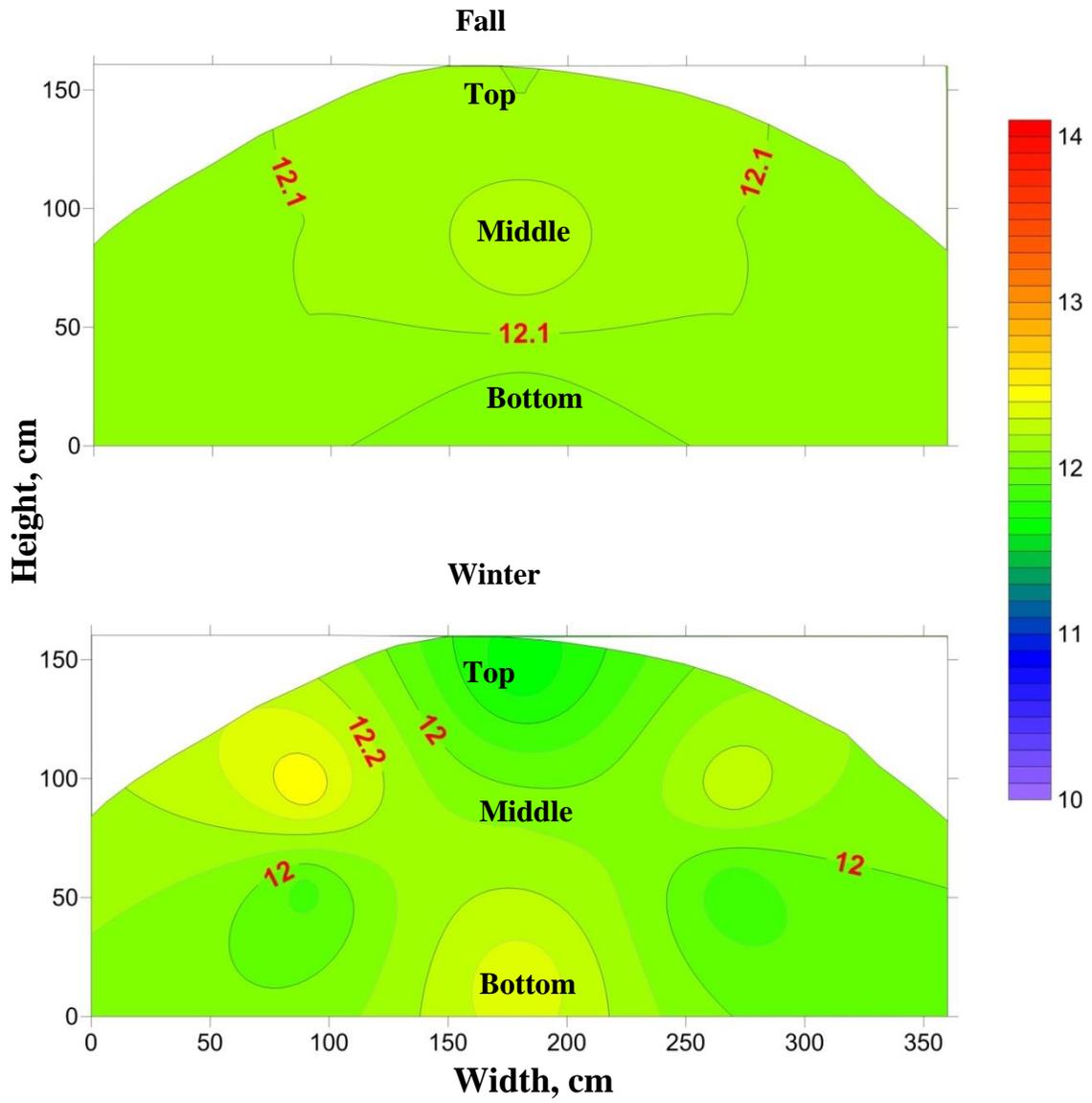


Figure 4.21 Moisture content profile of canola at different layers of head part of the silo bags in fall and winter (Fall: Oct. 10, 2011; Winter: Jan. 15, 2012)

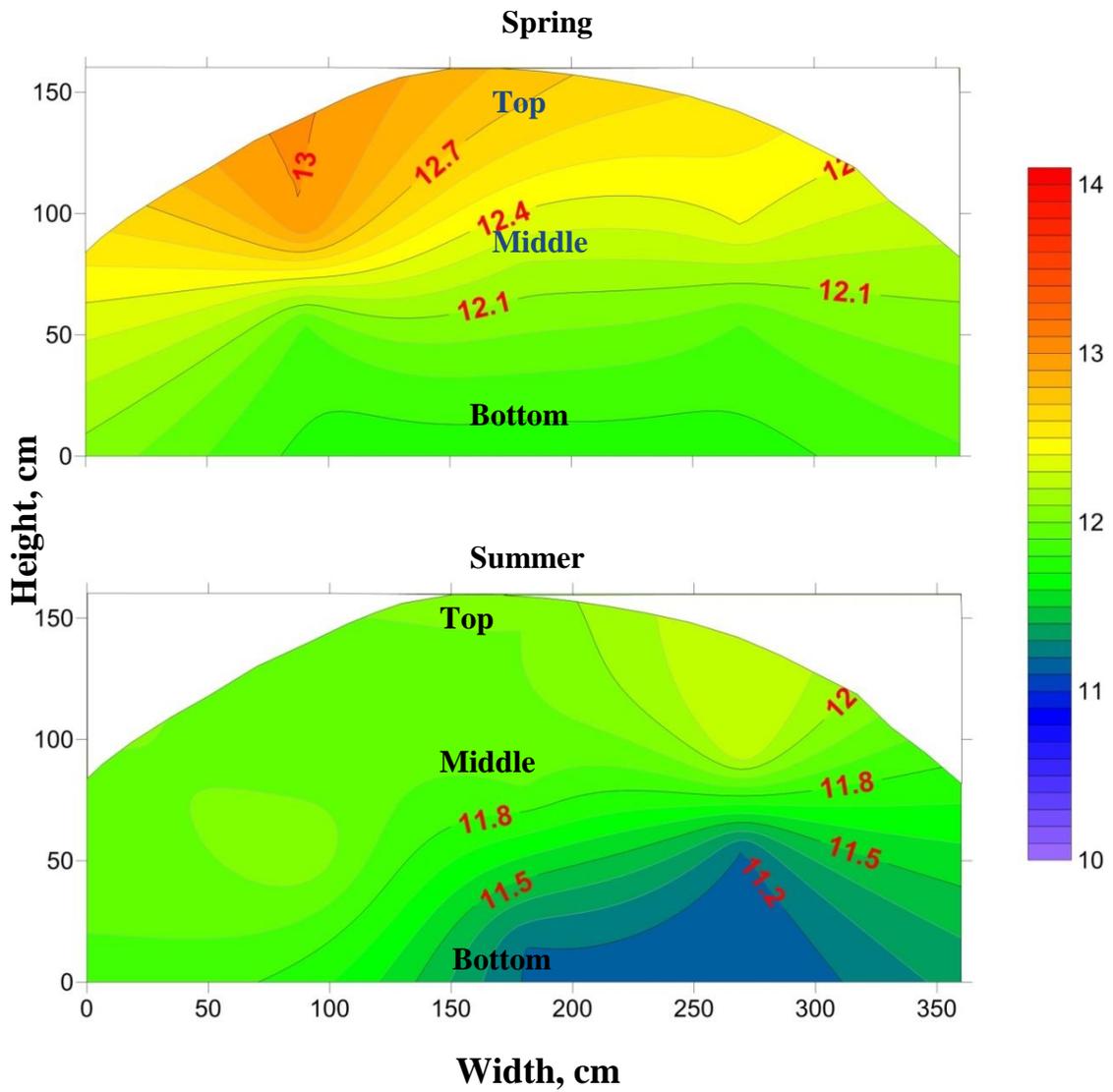


Figure 4.22 Moisture content profile of canola at different layers of head part of the silo bags in spring and summer (Spring: May 1, 2012; Summer: July 31, 2012)

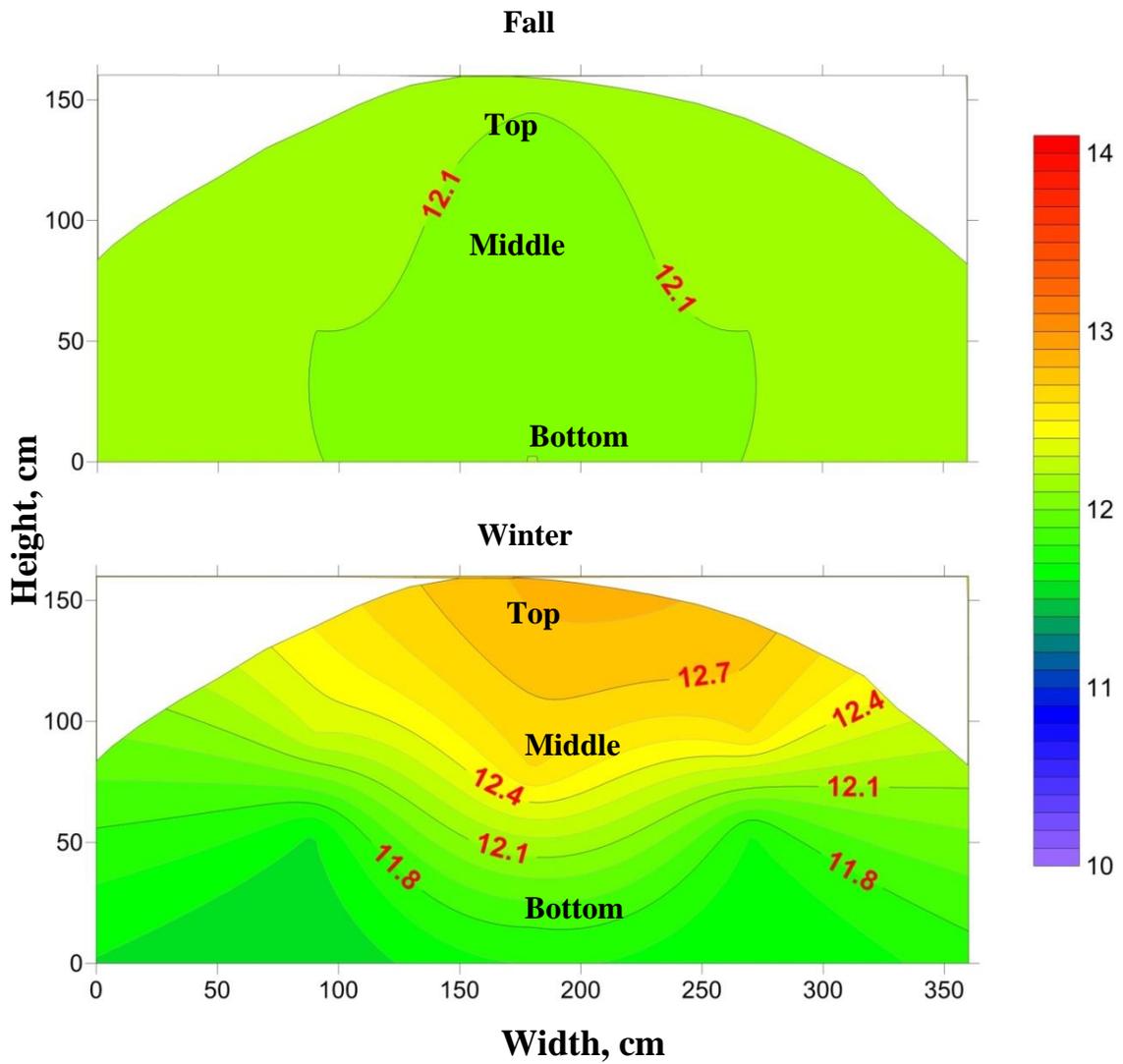


Figure 4.23 Moisture content profile of canola at different layers of centre part of the silo bags in fall and winter (Fall: Oct. 10, 2011; Winter: Jan 15, 2012)

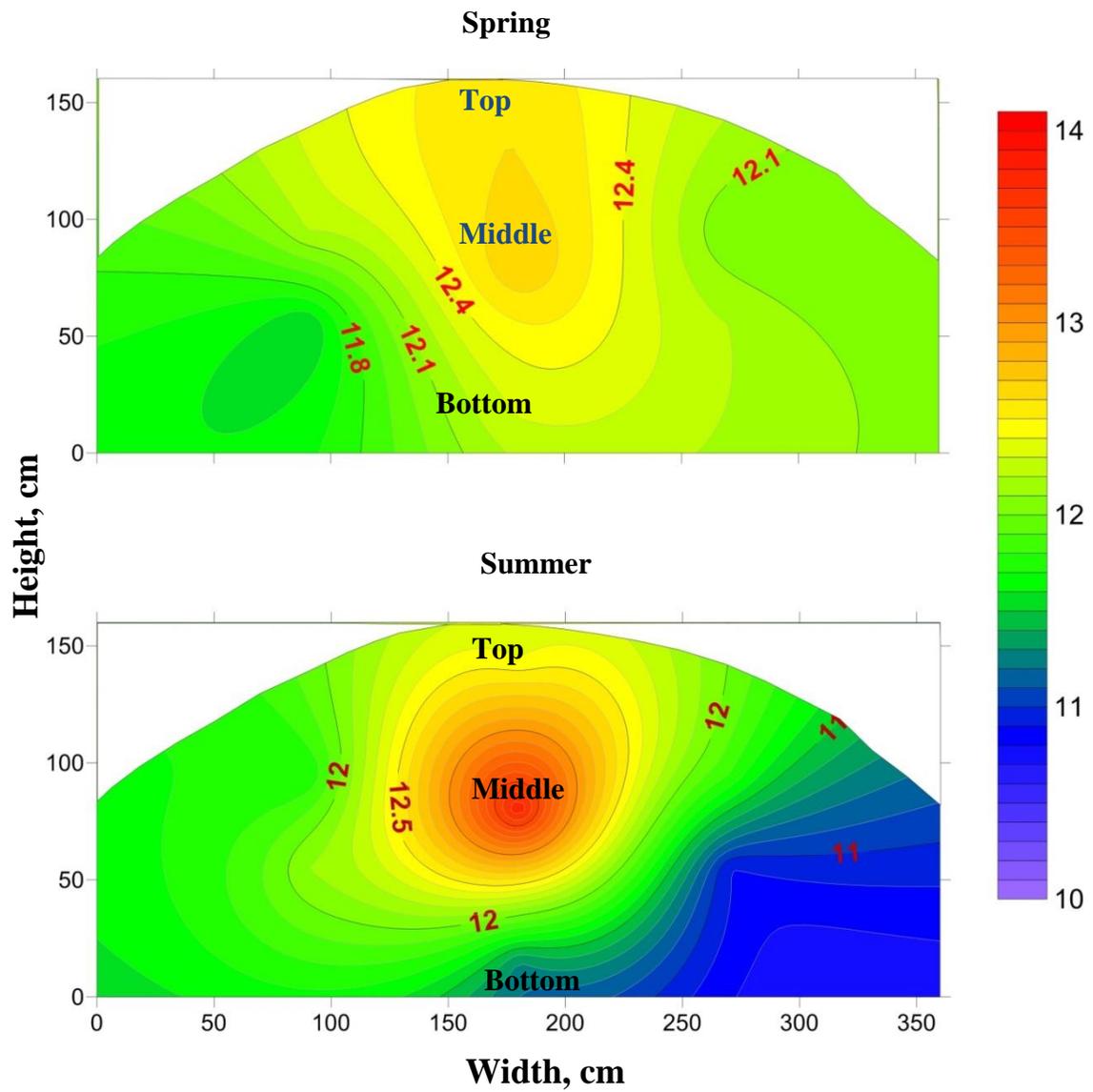


Figure 4.24 Moisture content profile of canola at different layers of centre part of the silo bags in spring and summer (Spring: May 1, 2012; Summer: July 31, 2012)

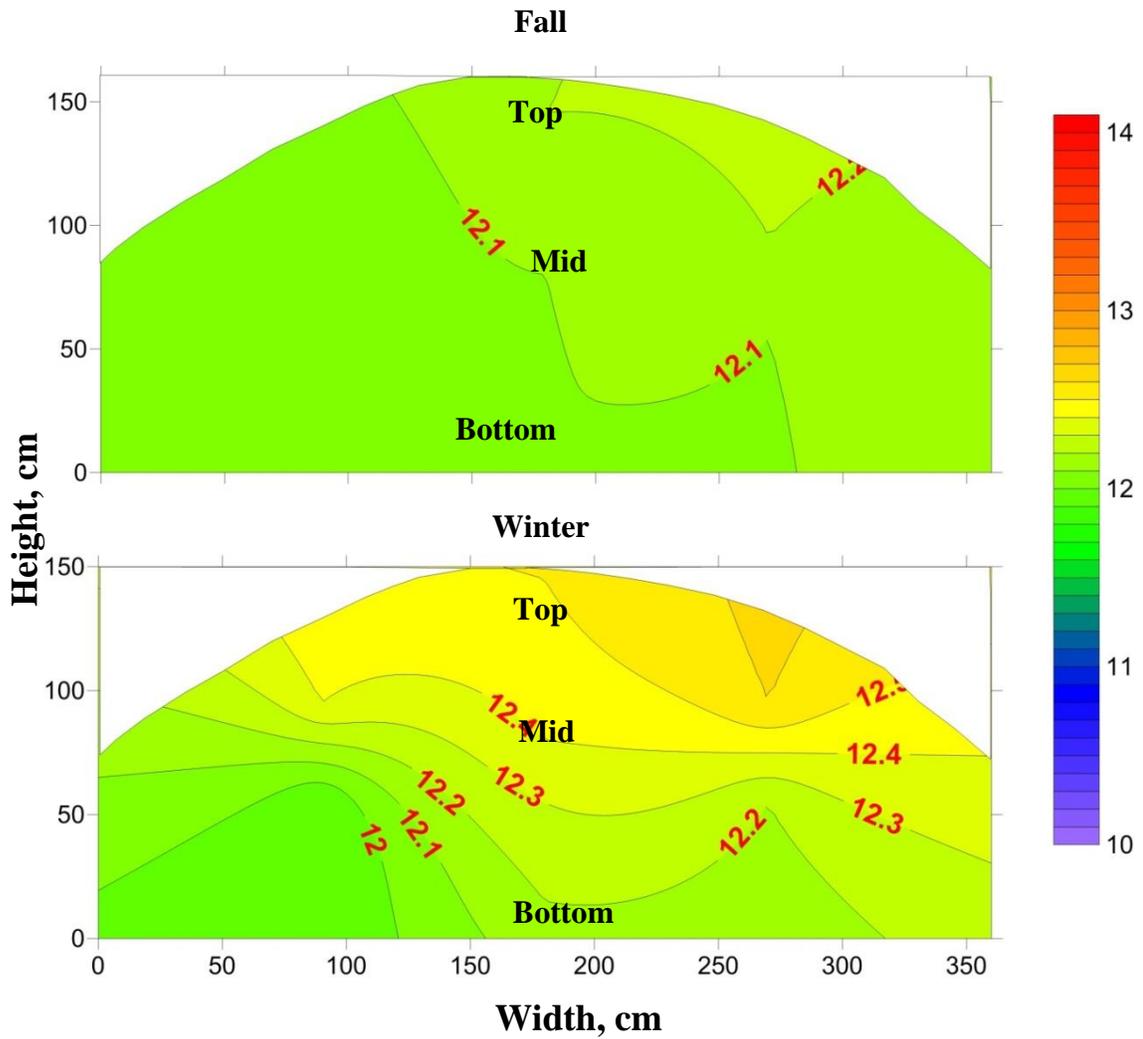


Figure 4.25 Moisture content profile of canola at different layers of tail part of the silo bags in fall and winter (Fall: Oct. 10, 2011; Winter: Jan 15, 2012)

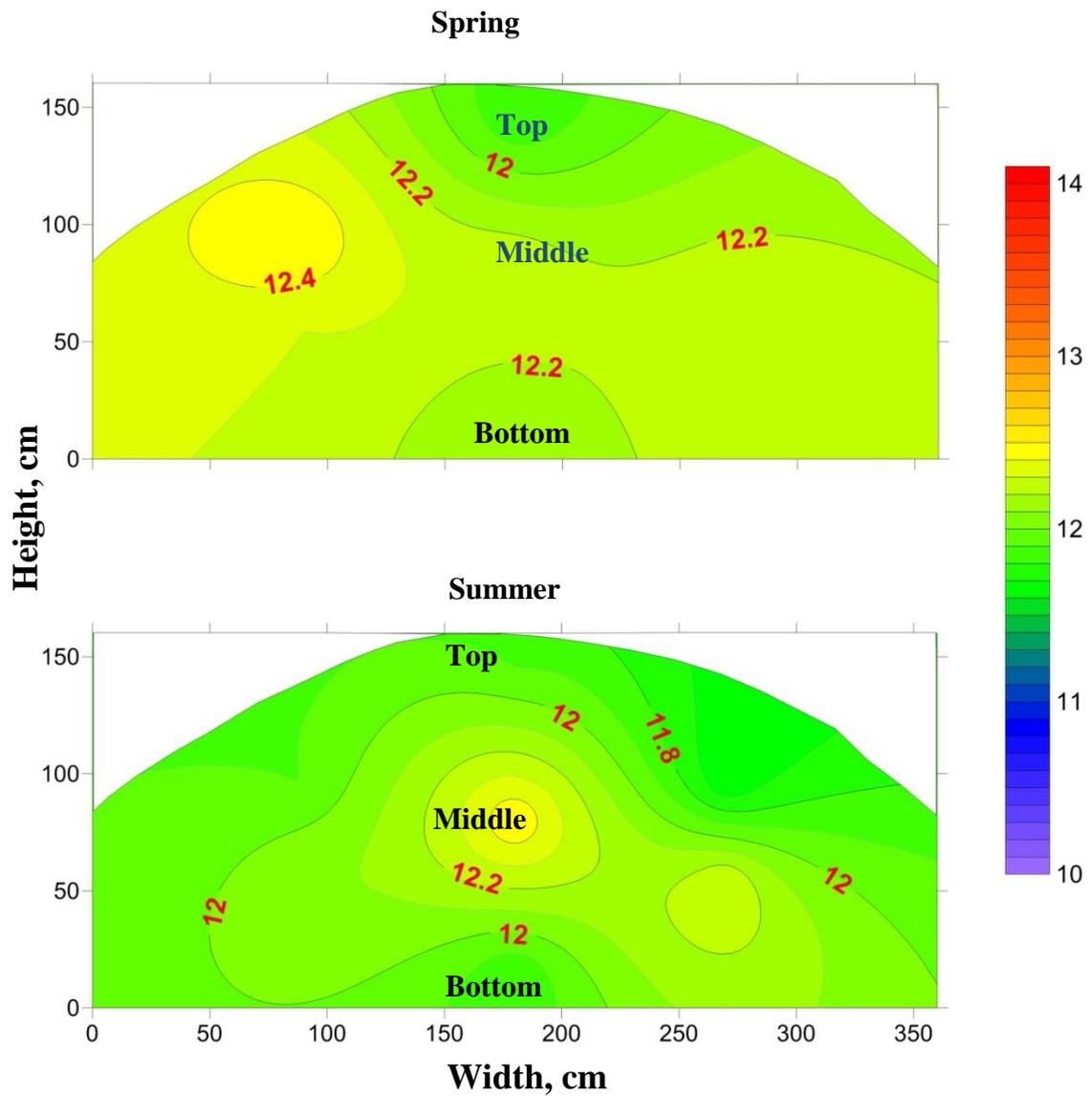


Figure 4.26 Moisture content profile of canola at different layers of tail part of the silo bags in spring and summer (Spring: May 1, 2012; Summer: July 31, 2012)

4.2.1.2. Germination

Germination decreased with increase in storage time (Figs. 4.27-4.28) in both the years. Initial germination of the first year seeds was $94.3 \pm 2.3\%$, and average germination of the canola in the silo bag was above 80% after 20 wk of storage (at the time of first unloading). The top layer of the canola and tail portion seeds had lower germination when compared to other parts of the silo bag. After 28 wk of storage (second unloading), the germination of the canola seeds at the top location of tail part was below 50%, and germination of the top layer seeds at all portions of silo bag decreased below 20% after 40 wk of storage. In the second year, initial germination was $92.5 \pm 2.1\%$ and maintained above 80% up to 24 wk at most parts of the silo bag (first unloading time). Similar to the first year, the tail portion had lower germination. Germination in most of the bags was above 69% except the top layers up to the second unloading time, and the same trend (decrease of germination of top layer seeds with increase in time during summer storage) was noticed in the second year of storage.

Different sampling locations and layers of canola had significant effects on changes in germination during storage in both the storage years ($P < 0.0001$). There was a significant change in germination from initial germination after 8 wk of storage, and germination of canola in the tail portion was significantly different than the seeds in centre and head portions of the silo bag. There were significant differences in germination between top, middle, and bottom layers of the seeds in both storage years ($\alpha = 0.05$).

4.2.1.3. Free fatty acid values

The initial FAV levels were 31.3-33.7 and 31.9-32.4 mg KOH/100 g of dry seed for the first and second year at different parts of the bags, respectively and FAV increased with increase in storage period in both the years. FAV of canola were 34.3-40.5 and 40.6-44.5 mg KOH/100 g of dry seed at different parts of the bag during first unloading (after 20 wk of storage), and it increased to 51.2-61.3 and 62.1-76.9 mg KOH/100 g of dry seed during the third unloading time (40 wk of storage) in the first and second year of storage, respectively (Figs. 4.29-4.30). In both years, FAV increased more than 2-fold of initial values at the time of the third unloading (after summer storage). Sampling location along the length, and the seed layer and storage period had significant effects ($P \leq 0.007$) on changes in FAV during storage in both the years. Significant differences of FAV were also noticed between top and bottom layers of canola by means comparison tests. There were significant differences in FAV between the tail portion, and centre or head portions of the silo bag, but there were no significant differences between centre and head portions of the bag.

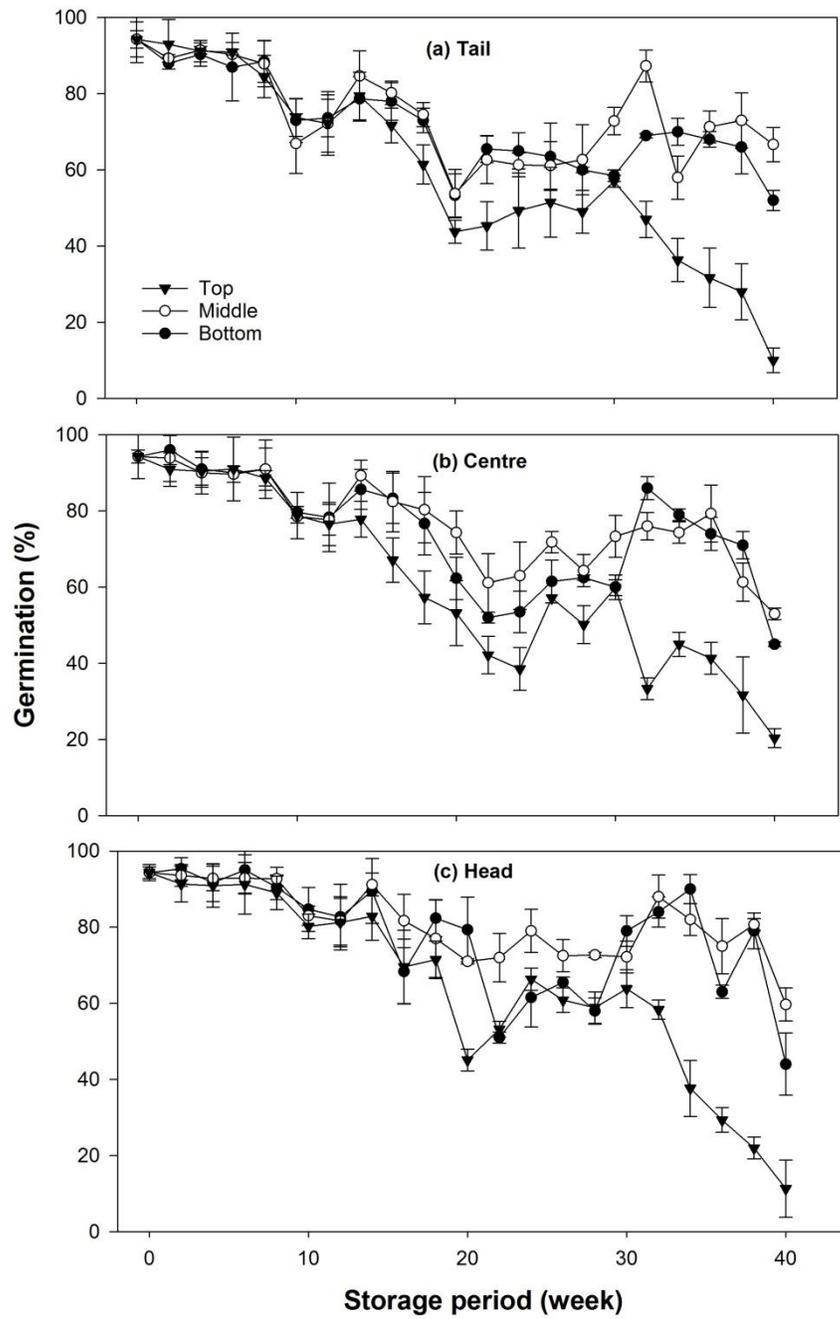


Figure 4.27 Germination of canola at different layers of silo bags in storage year 1 (2011-12)

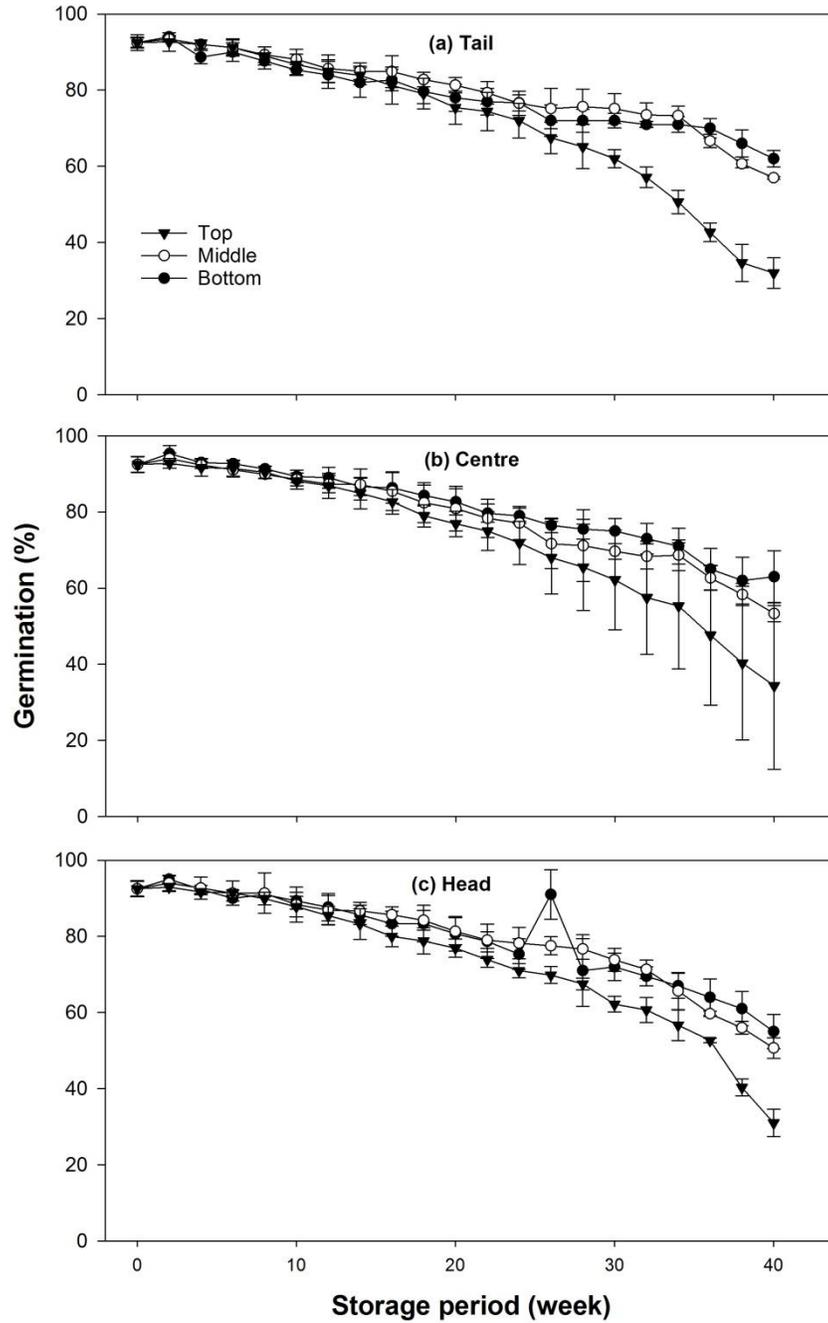


Figure 4.28 Germination of canola at different layers of silo bags in storage year 2 (2013-14)

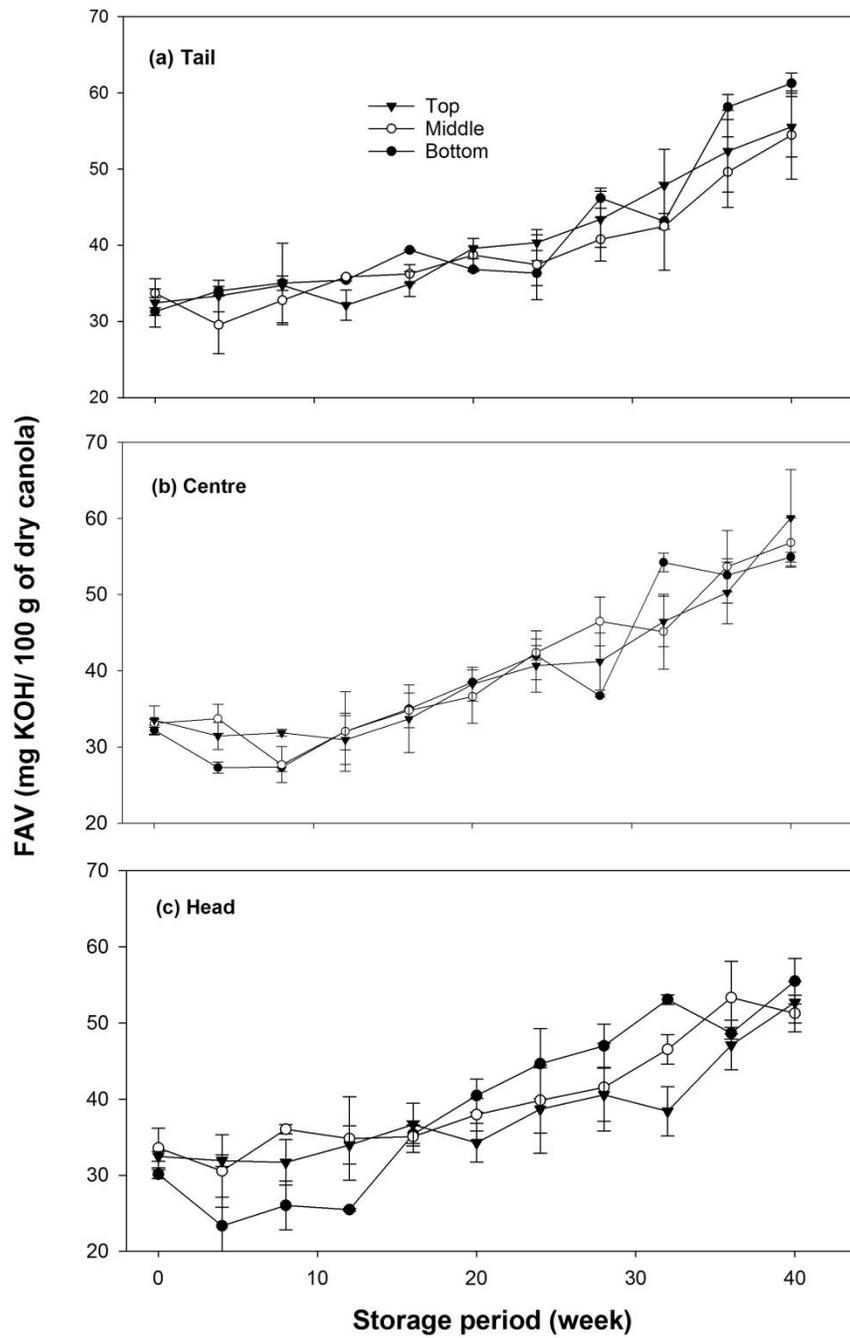


Figure 4.29 FAV of canola at different layers of silo bags in storage year 1 (2011-12)

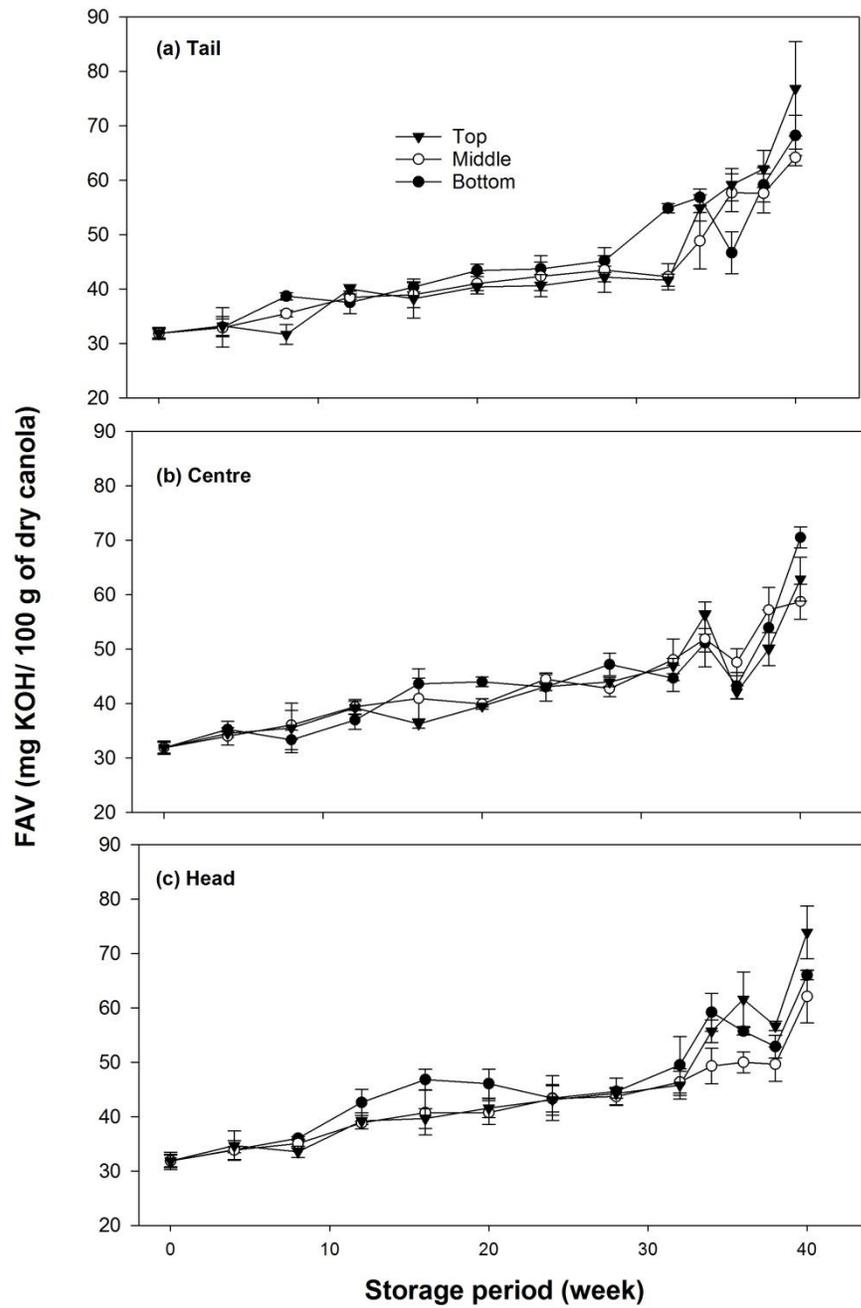


Figure 4.30 FAV of canola at different layers of silo bags in storage year 2 (2013-14)

4.2.1.4. CO₂ levels

Initial gas samples were collected at different parts of the bag 2 days after loading and then samples were collected every 2 wk. Initial CO₂ levels were 1.2±0.1 and 1.9±0.2% for the first and second years of storage, and increased to 10.5 ±0.8 and 9.5±0.5% after 4 wk of storage (Fig. 4.31-4.32), respectively. A drop in CO₂ levels was noticed after 6 wk of storage, and CO₂ levels were maintained around 6% until the second unloading time (after 28 wk of storage) in both the storage years. During summer storage, increases in CO₂ levels were noticed in both the years and CO₂ levels were around 12 and 19% after 40 wk of storage in year 1 and 2, respectively.

There were significant differences in CO₂ levels at different locations along the length of the silo bag in both storage years ($P \leq 0.008$), and storage period also had significant effects on CO₂ levels inside the silo bags ($P < 0.0001$). But there were no significant differences between different layers of the silo bag ($P = 0.46$). Means comparison test showed that there was a significant difference in CO₂ levels at the tail portion of silo bags than other portions of the silo bags in both the storage years ($P < 0.001$). The mean's comparison results also showed significant differences in CO₂ levels at the top layer of the bag than at other parts of the bag in the first year of storage ($P < 0.0001$), but in the second year there were no significant differences in CO₂ levels at top, middle, and bottom layers of the bags ($P \geq 0.43$).

4.2.1.5. O₂ levels

In the first year of storage, initial O₂ levels were between 19.0 and 20.1% at different parts of the bags and dropped to 15.6-18.5% after 4 wk of storage (Figs. 4.33-

4.34). The O₂ concentration maintained around 15% until the second unloading time (28 wk of storage), and started declining during summer storage. After 40 wk of storage, the O₂ concentrations were 9.1-11.5% at different parts of the bag. In the second year of storage, initial O₂ concentration was 20.1% and decreased rapidly to 8.4-12.0% within 4 wk of storage. The O₂ concentrations at different parts of the bags at the time of first unloading (24 wk of storage) were 13.6-14.5%. Similar to the first year storage, decrease of O₂ levels during summer storage was found in the second year and after 40 wk of storage O₂ levels were 4.0-6.3%.

Statistical analysis of first and second years of data showed that storage period and sampling location along the length of the bags had significant effects on O₂ levels inside the silo bag ($P \leq 0.04$), but the layer in the silo bags had no significant effects on O₂ level ($P \geq 0.15$). There were no significant differences between O₂ levels in the top, middle, and bottom layers of the grain. The O₂ levels increased from the second week of storage and there were no significant differences between O₂ levels in the tail and centre portions of the bag; and the head portion had the lowest O₂ concentration.

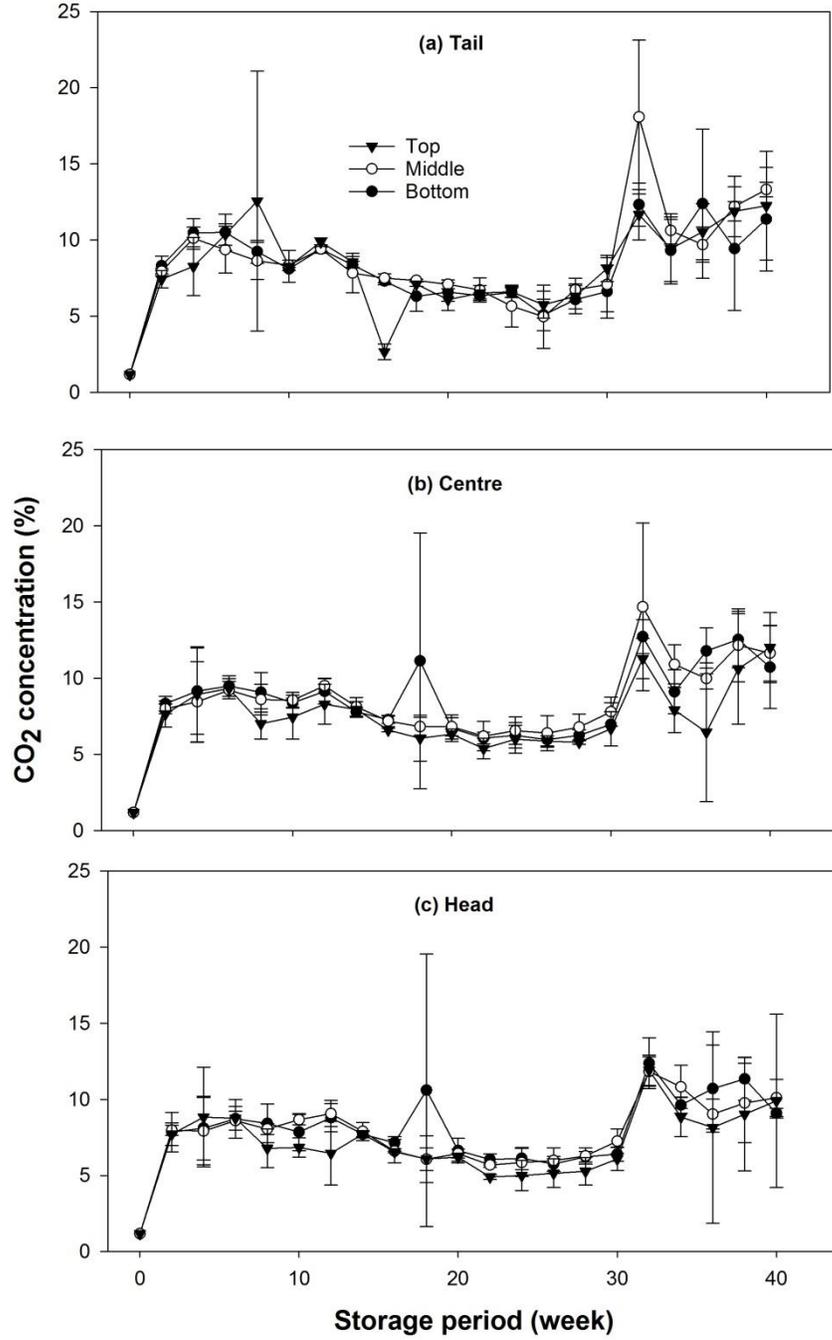


Figure 4.31 Intergranular CO₂ concentration at different layers of silo bags during storage in storage year 1 (2011-12)

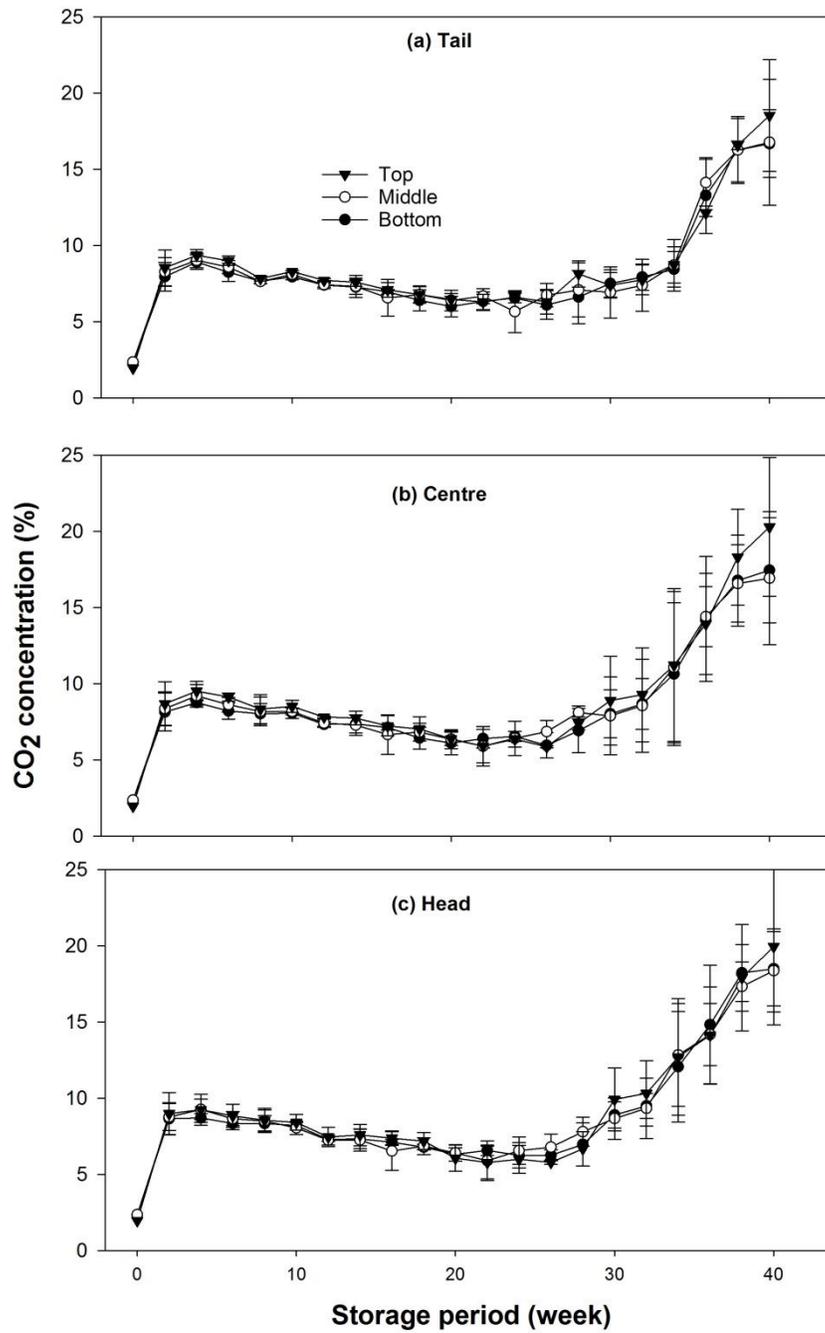


Figure 4.32 Intergranular CO₂ concentration at different layers of silo bags during storage in storage year 2 (2013-14)

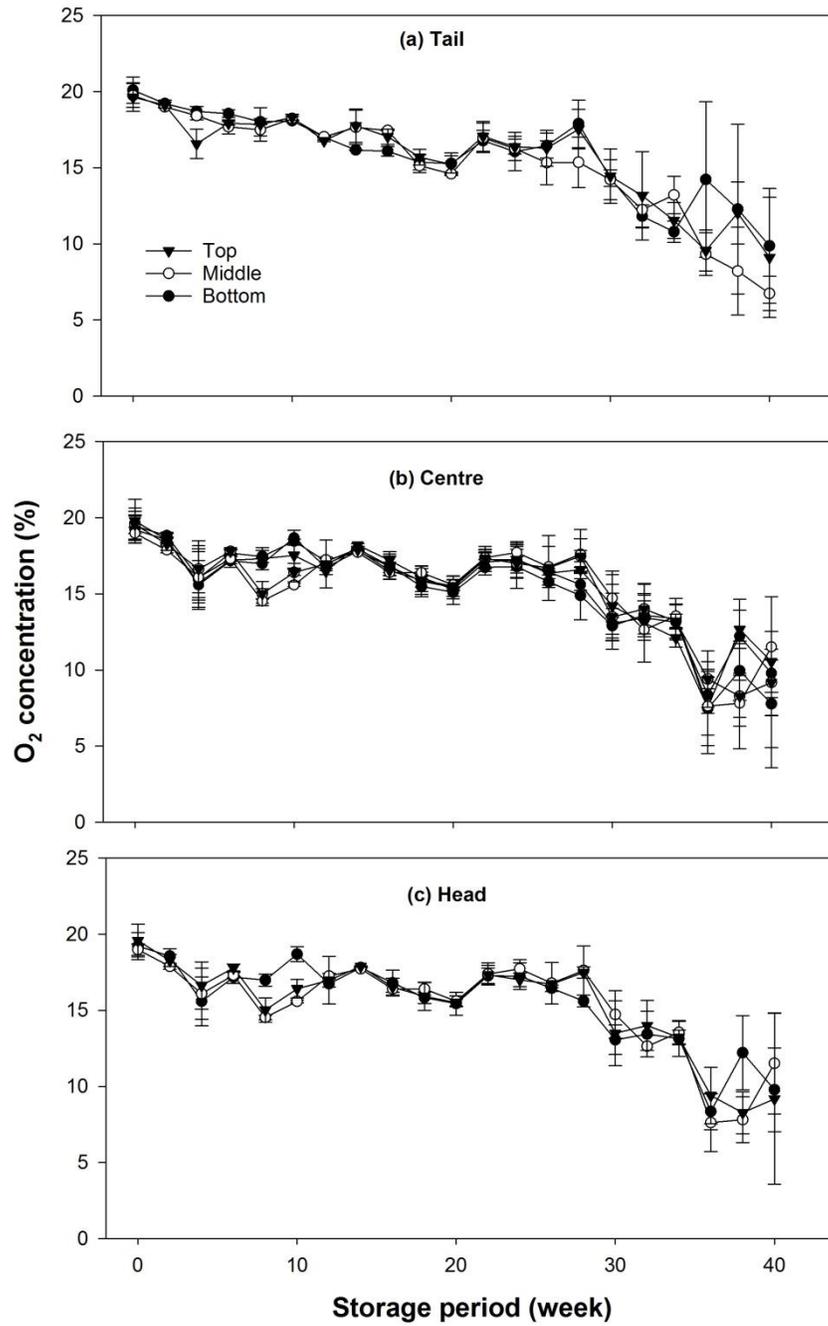


Figure 4.33 Intergranular O₂ concentration at different layers of silo bags during storage year 1 (2011-12)

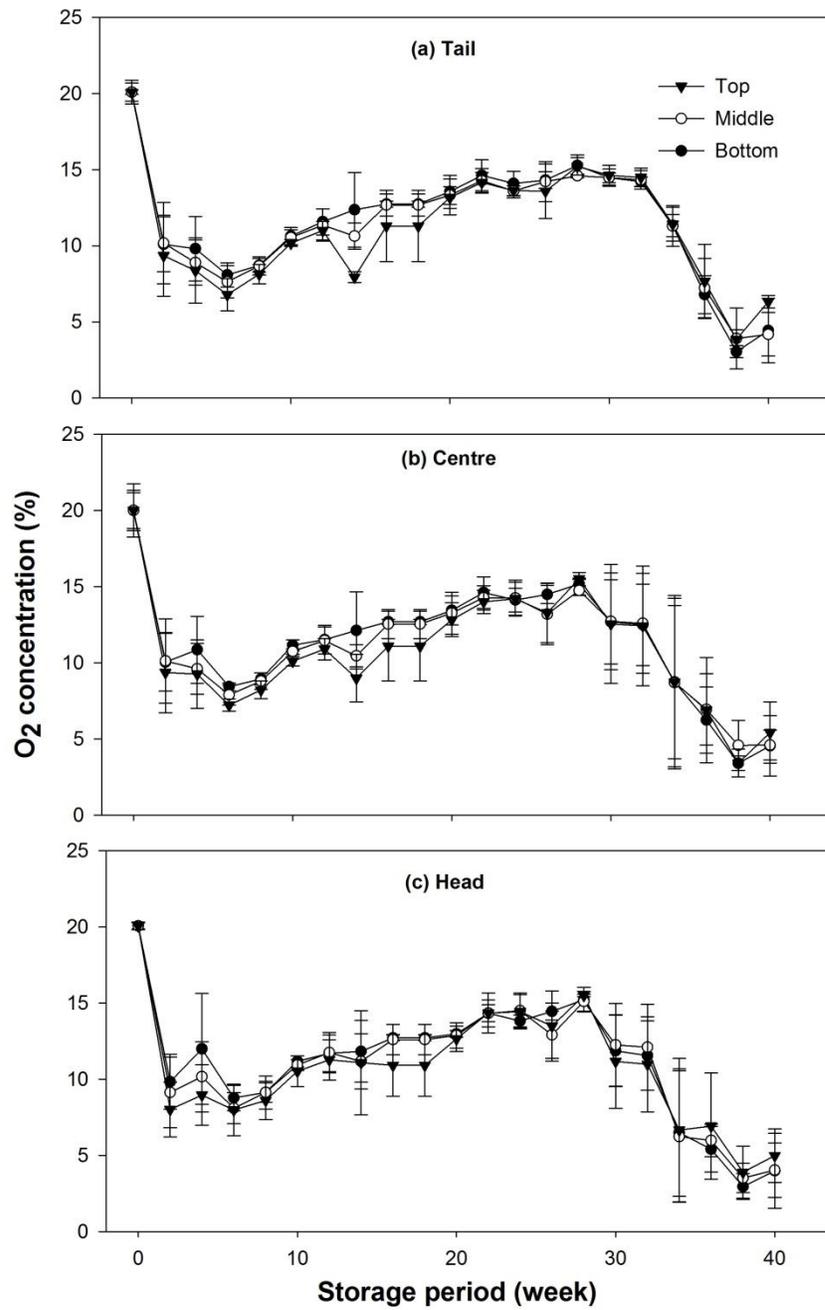


Figure 4.34 Intergranular O₂ concentration at different layers of silo bags during storage year 2 (2013-14)

4.2.1.6. Temperature

Temperatures of canola during loading were 14.5°C and 16.2°C, and temperatures measured at different parts of the silo bags are given in Figures 4.35 and 4.36. For the first storage year, temperatures at different parts of the bag were measured until the third unloading time (40 wk of storage), but in the second year of the storage study, temperature was measured only up to the end of June due to the damage of thermocouples. In both years of storage, the top layer of grain at the tail, centre and head portions stayed close to ambient temperatures (Figs. 4.37-4.42). In autumn and winter, the top layer was the coolest zone and the bottom layer was the warmest part of the silo bags. But in summer, the top layer became the warmest portion of the silo bag. In the second year of storage, the monthly average temperatures were lower than in the first year of storage (Table 4.1). Temperatures of all parts of the silo bags in the second year of storage were below freezing until April, an indication of the cold and long winter of 2013-14. For the most of the storage time, the temperature gradient between the bottom and top layers of the silo bag was 4 to 7°C, and this large gradient in temperature caused moisture migration between bottom and top layers of the silo bag. Water condensation at the top layer might occur when ambient air was colder than the intergranular air (Jian et al., 2015b). Condensation at the inner portion of sampling port cap was noticed during winter (Fig. 4.43) in both years. Temperature profile maps show that, the temperatures of bottom and top layers of silo bag were 4°C and -13°C, respectively in winter time (on January 15, 2012), and in summer (July 31, 2012) the temperatures were 20°C and 39°C in bottom and top layers, respectively.

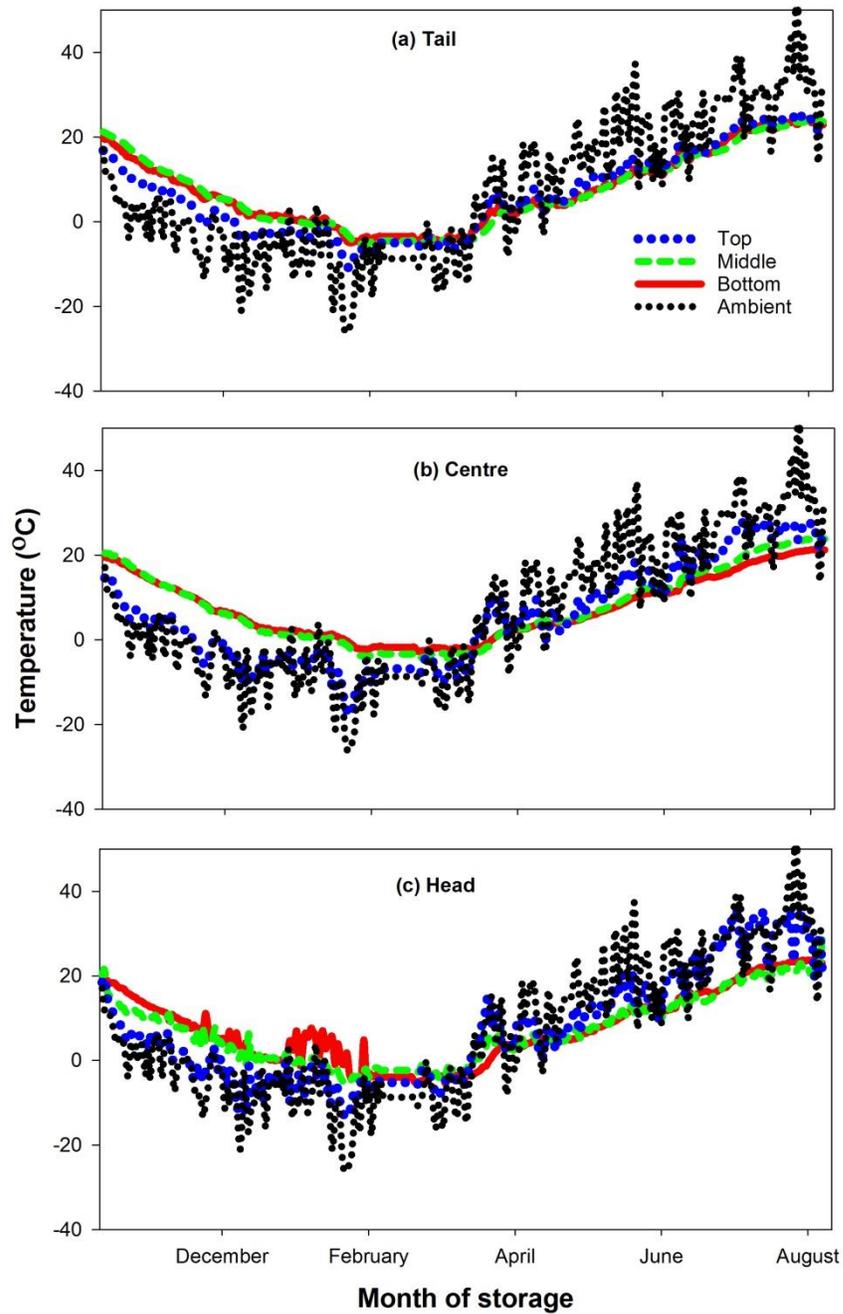


Figure 4.35 Temperature of canola at different layers of silo bags in storage year 1 (2011-12)

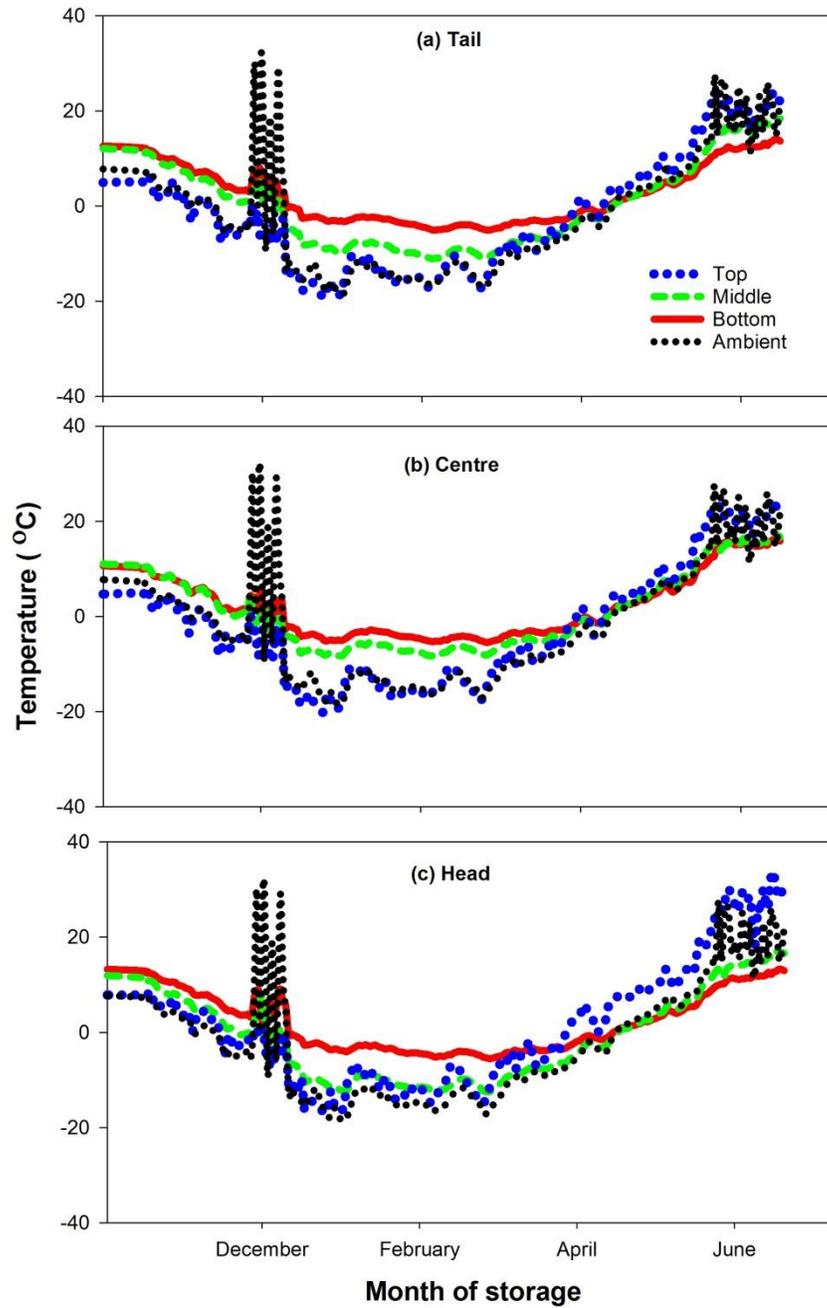


Figure 4.36 Temperature of canola at different layers of silo bags in storage year 2 (2013-14)

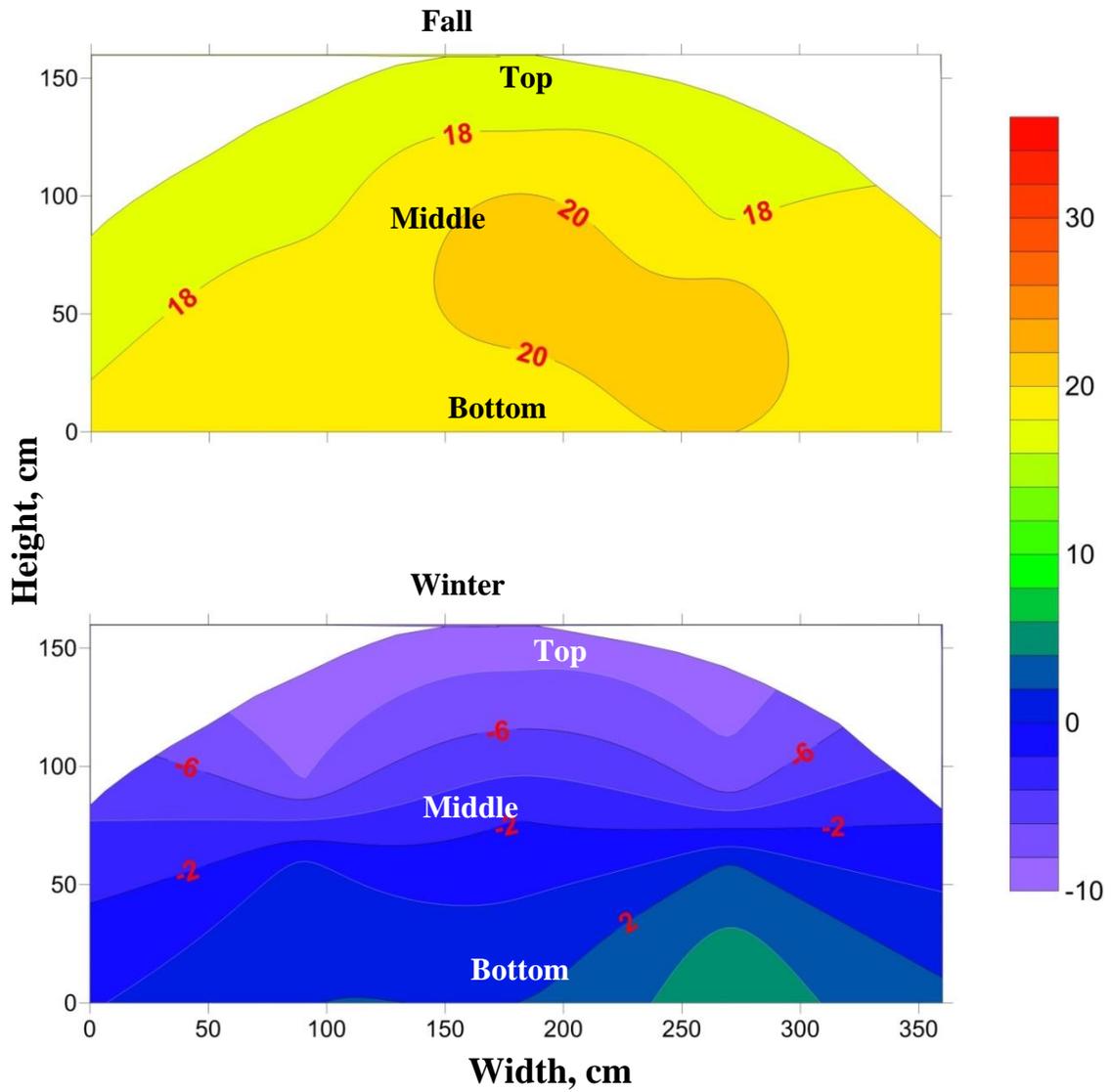


Figure 4.37 Temperature profile of canola at head part of silo bag at different seasons (A) Fall: Oct. 10, 2011; (B) Winter: Jan. 15, 2012

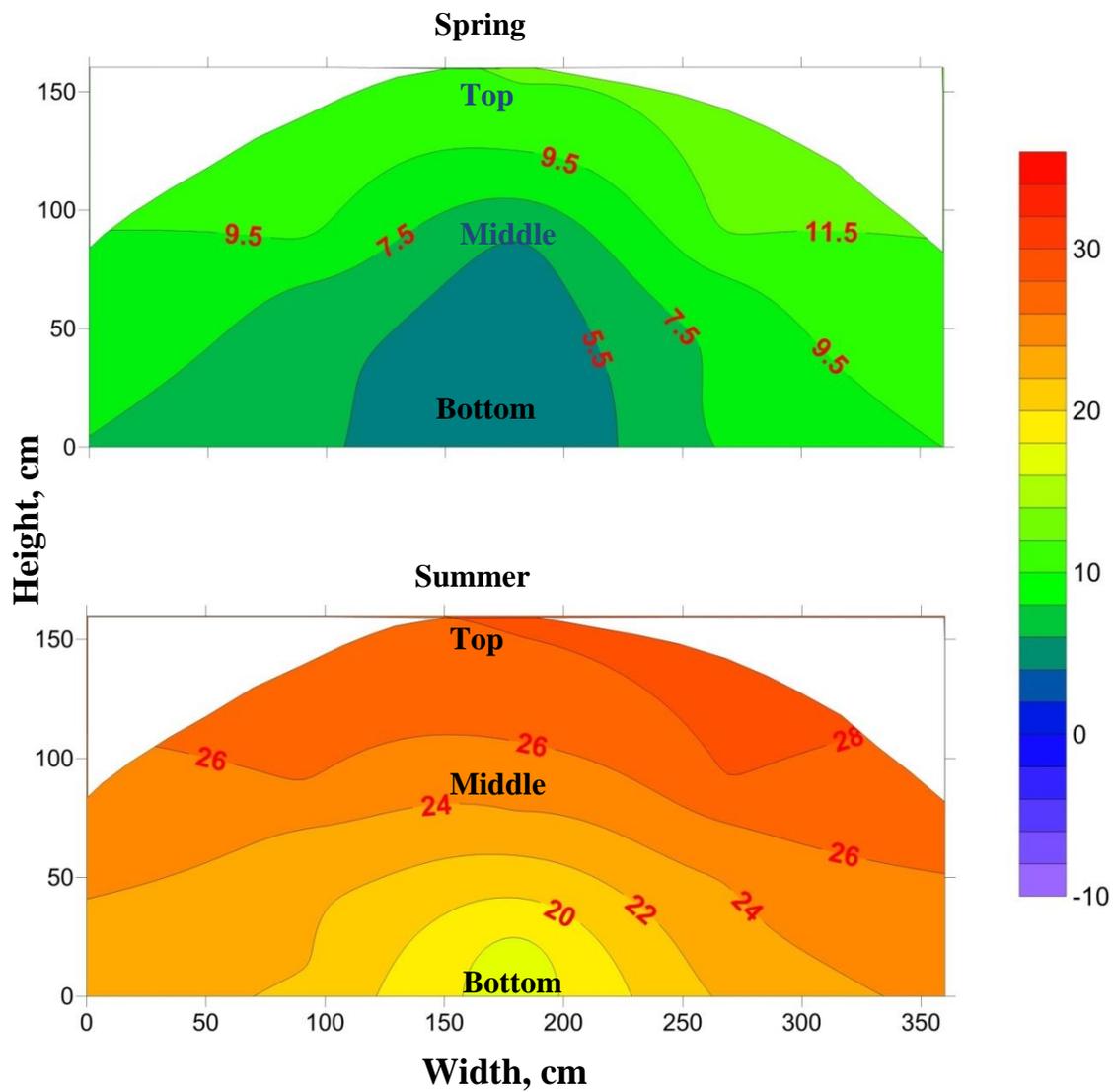


Figure 4.38 Temperature profile of canola at head part of silo bag at different seasons (A) Spring: May1, 2012; (B) Summer: July31, 2012

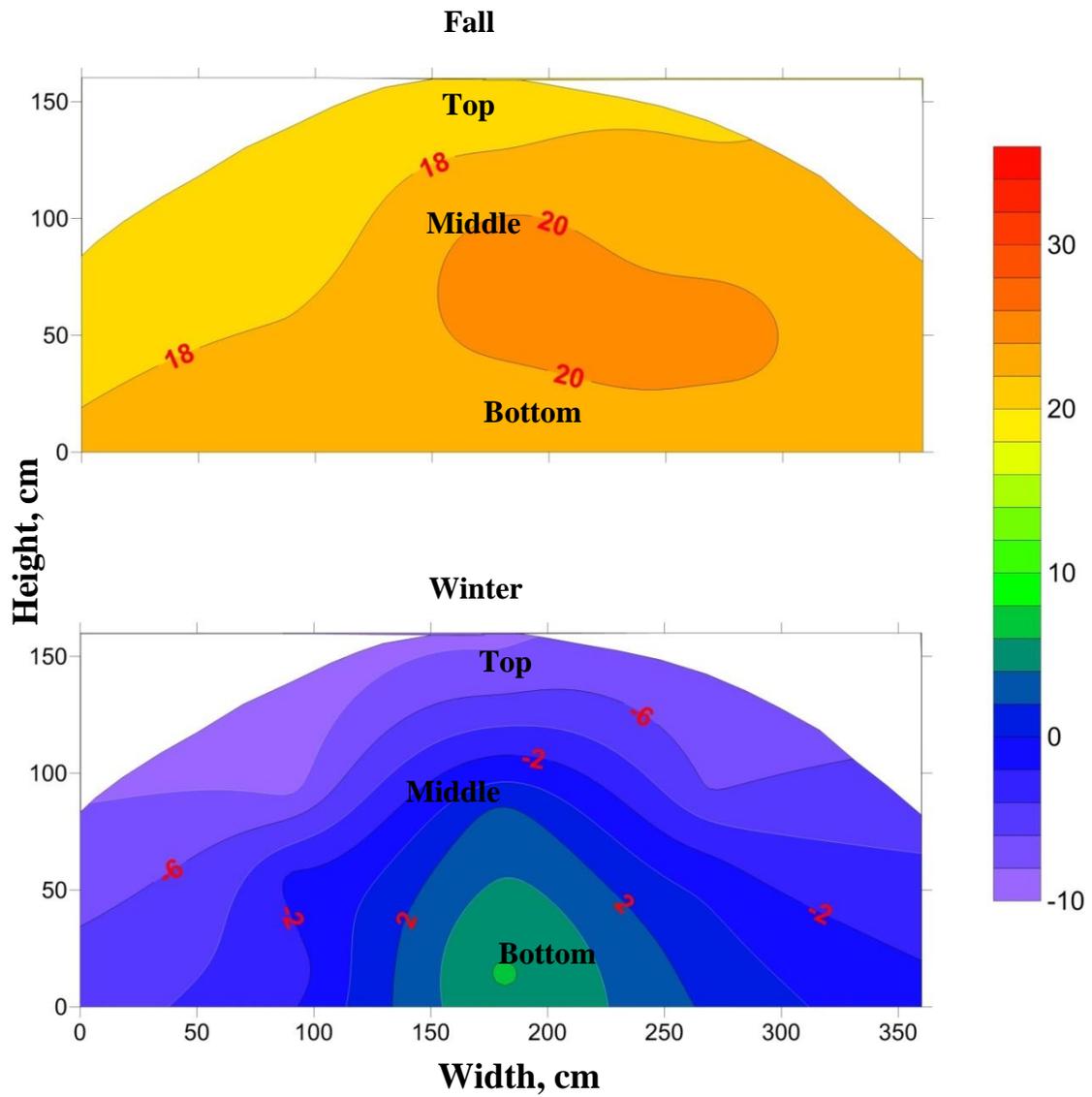


Figure 4.39 Temperature profile of canola at centre part of silo bag at different seasons (A) Fall: Oct10, 2011; (B) Winter: Jan 15, 2012

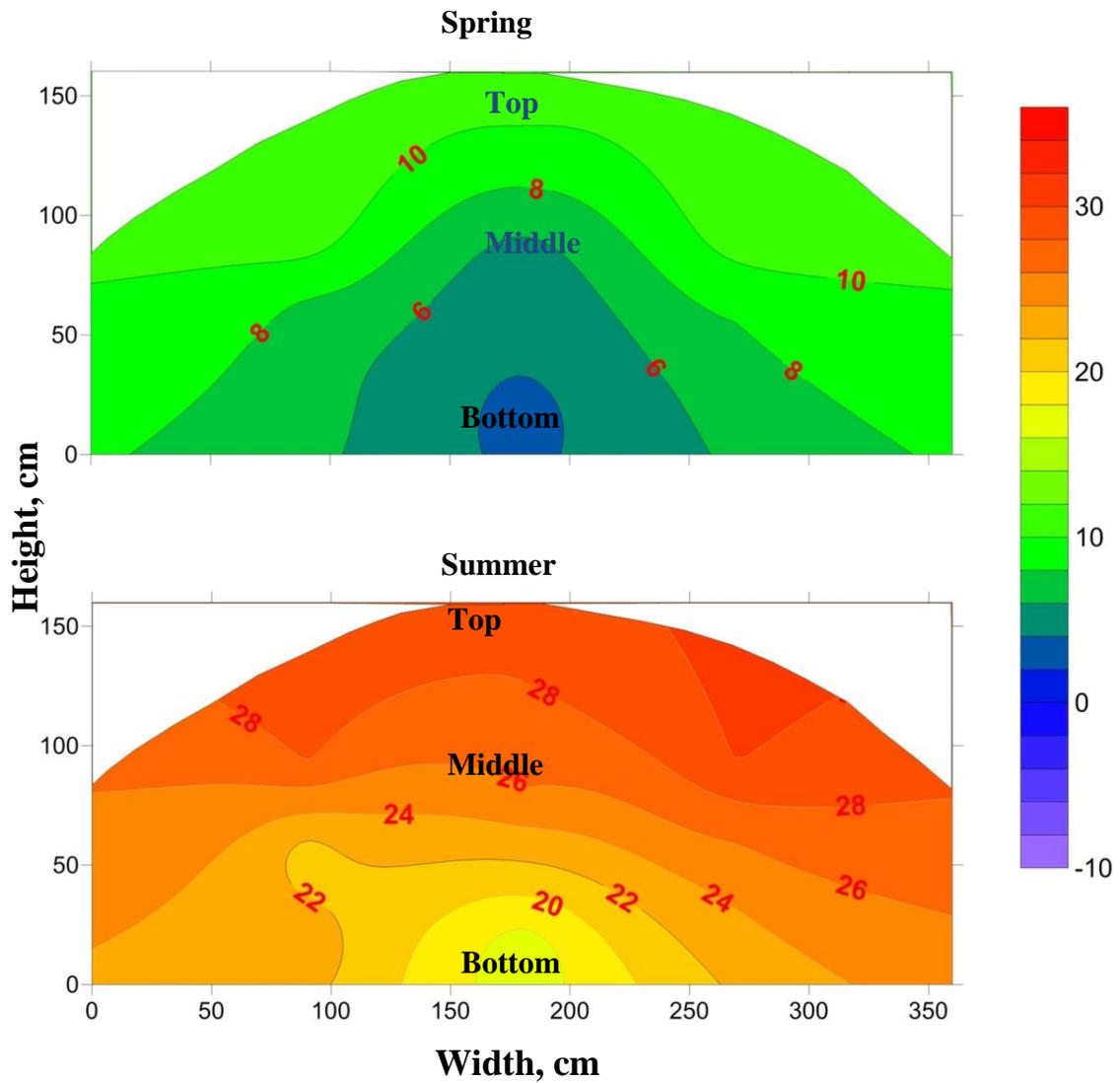


Figure 4.40 Temperature profile of canola at centre part of silo bag at different seasons (A) Spring: May 1, 2012; (B) Summer: July 31, 2012

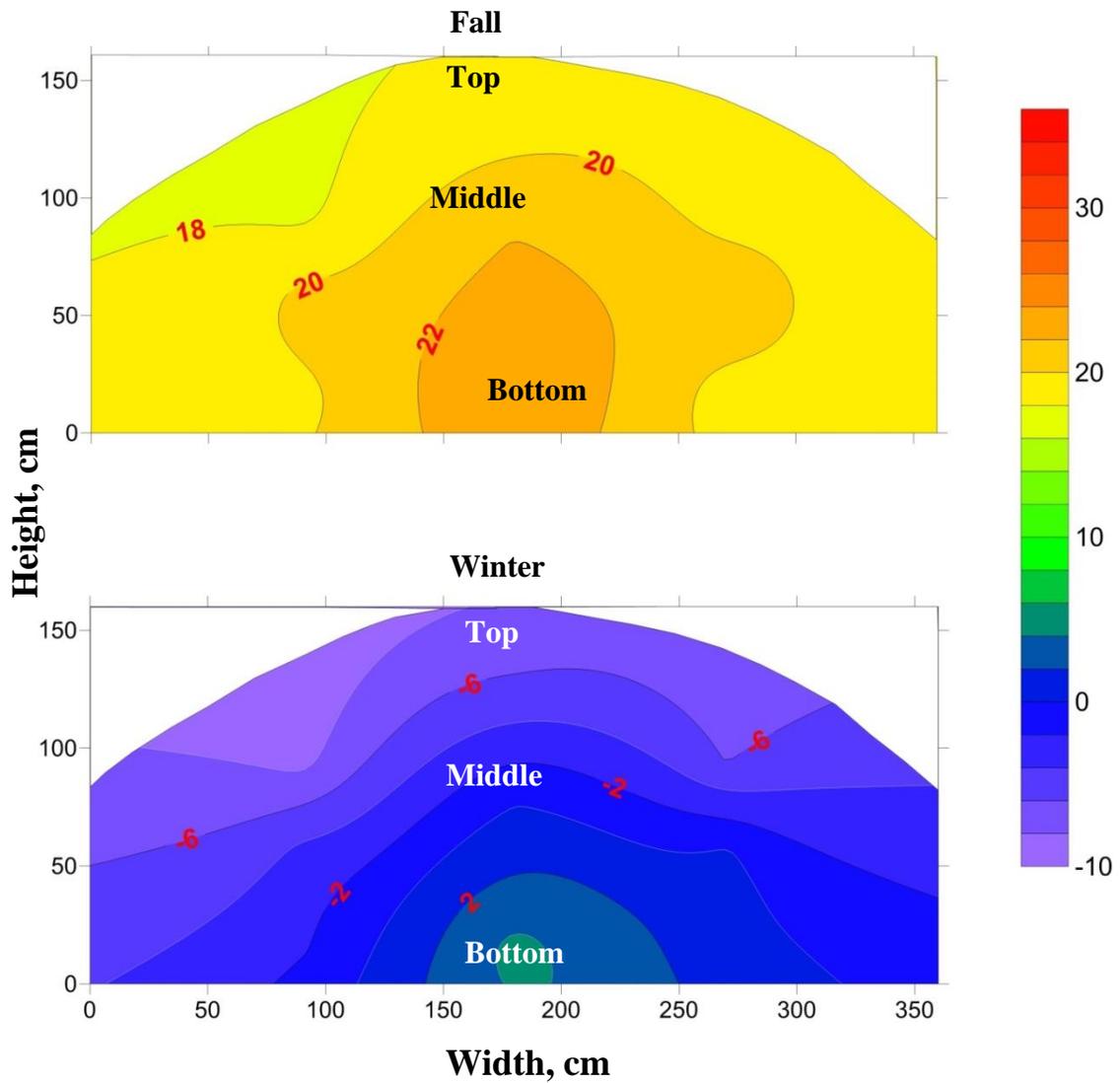


Figure 4.41 Temperature profile of canola at tail part of silo bag at different seasons

(A) Fall: Oct. 10, 2011; (B) Winter: Jan 15, 2012

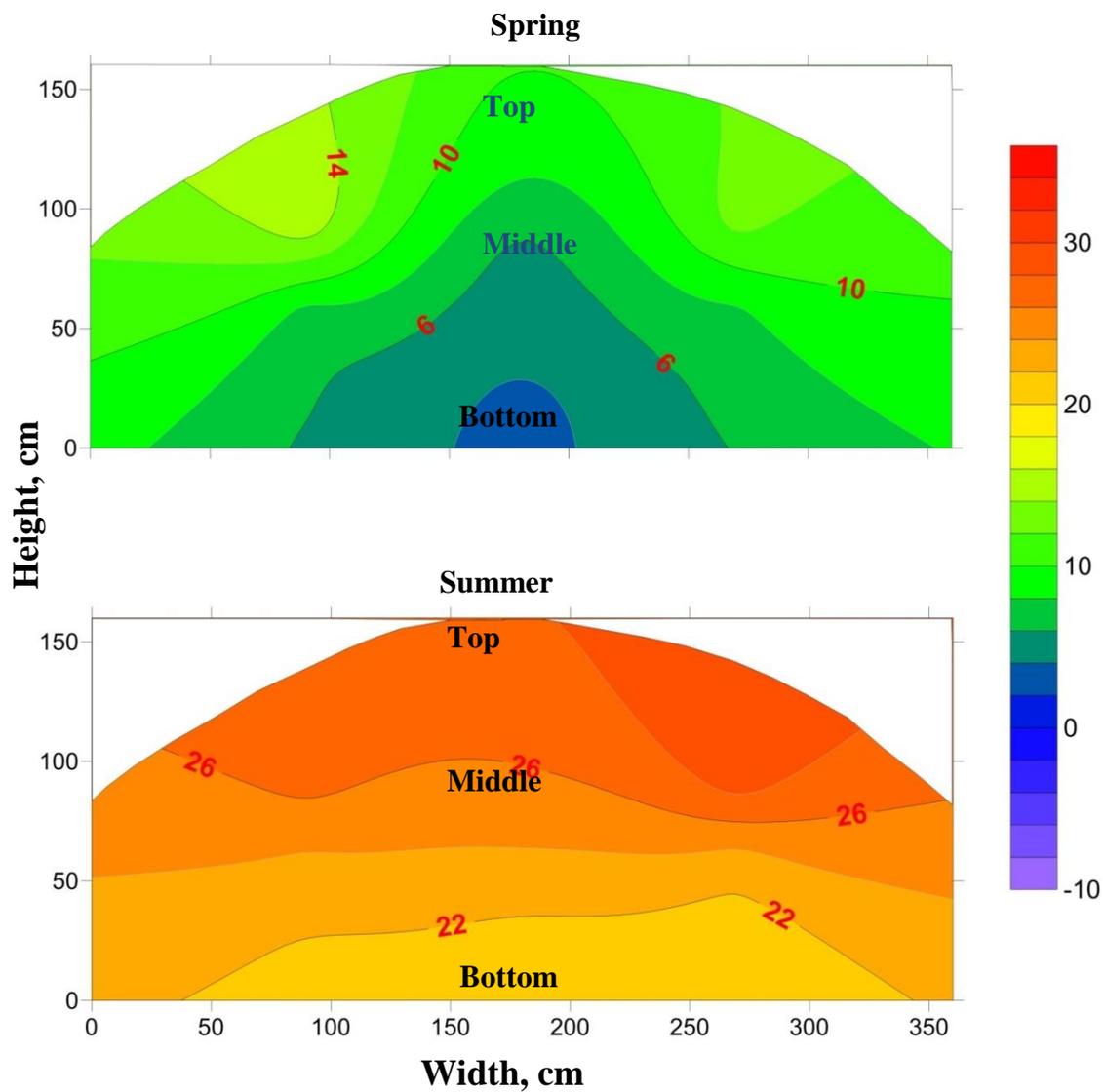


Figure 4.42 Temperature profile of canola at tail part of silo bag at different seasons

(A) Spring: May 1, 2012; (B) Summer: July 31, 2012

Table 4.1 Monthly average temperatures at Winnipeg, Manitoba (49.9°N, 47.1°E)

Month	Average Temperature (°C)	
	2011-12	2013-14
October	14.4	10.6
November	2.3	0.4
December	-3.2	-15.4
January	-9.5	-17.8
February	-8.1	-17.5
March	3.2	-10.4
April	6.8	1.3
May	13.1	12.5
June	18.9	17.9
July	24.1	20.1



Figure 4.43 Condensation of water droplets at the sampling port cap during winter

4.2.1.7. Commercial grading of canola

In the first year of storage, canola were graded as Canada Grade 1, Canada Grade 2 and Feed grade at the first, second and third unloading times, respectively. However, in the second year of storage, canola were graded as Canada Grade 1 at first and second unloading times, Canada Grade 2 at the third unloading time.

4.2.2. Discussion

The results of both storage years showed that there were significant changes in moisture content of the top layer of canola. The accumulation of moisture due to condensation of water at the inner wall of the silo bag might have caused the increase of moisture content of the top layer of the canola during storage (Jian et al. 2015b). Temperature profile maps of our study clearly show the large temperature gradients between top and bottom layers of canola in all parts of silo bag (Figs. 4.37-4.42). In winter, the temperature of bottom layer of the canola was 6.1°C, and at the same time temperature of top layer of canola was -6.8°C at the centre part of the bag. Temperature differences between bottom and top layers of the silo bag might have caused the moisture migration between bottom and top layers of canola. Bartosik et al. (2008a) noticed increase of moisture content (from 16.4 to 20.8%) at the top layer of sunflower seeds in the silo bags stored in Argentina. This was probably due to the repeated cycles of water condensation due to the temperature gradients. Gaston et al. (2009) and Jian et al. (2015b) predicted moisture content increases in the top layer of the silo bag using mathematical models, and they mentioned that the temperature difference between the

top layer and rest of the grain in the bag caused the moisture migration, which would create condensation near the top of the bag.

In both the years, the FAV values at different parts of the silo bags remained at safe levels until the second unloading (28 wk of storage). At the third unloading (40 wk of storage), FAV increased more than 2-fold from the initial value. Increase of FAV more than 1.5-fold of initial value is considered as an indicator of grain spoilage (White and Jayas, 1991). Ochandio et al. (2010) found 0.7 percentage point increase of fat acidity after 1 year of storage of 6% moisture content (wet basis) canola in Argentina. They also found the fat acidity remained close to the initial value for 6 months and started the increasing trend after 6 months of storage. Bartosik et al. (2008a) also found the increase of fat acidity more than 2-fold after 150 days of storage of wet sunflower seeds (16.4% m.c.). The increase in moisture content at the top layer of the seeds might have created damp spots and led to mould development and heated seeds, which caused hydrolysis of the fats, which caused the increase in FAV with increase in storage time. Sathya et al. (2009) also reported FAV of canola (12.5% m.c.) at 10°C increased from 22.0 ± 1.5 to 66.4 ± 0.1 (4 wks) and 89.9 ± 1.5 (16 wks) mg KOH/100 g. Seed germination of canola at most of parts of silo bags remained at safe levels up to the first unloading time and a rapid drop in germination at the top layer of the silo bags was noticed in both the storage years during summer. Reduced germination was also noted by Bartosik et al. (2008a) while storing wet (16.4% m.c.) wheat in silo bags. Germination of wet wheat dropped from 95 to 40% after 150 days of storage, but germination of dry (12.5% m.c.) wheat decreased from 93 to 87% only. They also noticed a drop of germination (74 to 55%) of

soybean at wet conditions (15.6% m.c.) for 150 days. Mould formation due to localized hot spots in the top layer of the silo bags might have caused the drop in germination when compared to the other parts of the bag during summer storage.

The application of agricultural lime (calcium carbonate) eliminated the rodent activity at the storage site, and there were no perforations in bags during unloading in both the storage years. Concentration of CO₂ is one of the best indicators of grain spoilage during storage, and Bartosik et al. (2008b) used intergranular CO₂ concentration to detect the spoilage of grain in silo bags. They classified silo bags into three categories based on CO₂ concentration: safe (CO₂ concentration <4.0%), risky (CO₂ concentration >4.0 and <11.5%), and unsafe (CO₂ concentration ≥11.5%). In both storage years and after loading the canola into silo bags, drastic increases in CO₂ (from 1.2% to 10.5%) and decreases in O₂ levels (from 20.1% to 15.6%) were observed. Canola have a tendency of higher respiration rate for 6 to 8 wk after harvesting, which might have caused this large increase in CO₂ concentrations inside the bags after 2 wk of storage. After 8 wk of storage, a slight drop in CO₂ concentration was noticed in both years. Cereal grains and oilseeds absorb some CO₂ during storage, and the absorption rate is higher for canola when compared with cereal grains (Cofie-Agblor et al. 1998). Lower CO₂ and higher O₂ levels were observed in the tail portion of the silo bags. Usually the tail portion is the weakest portion of a silo bag in terms of air tightness, because of the sealing practice (sealed using two wooden pieces) used for closing the end of the bag. The absorption of CO₂ by the canola, permeation of CO₂ through bag material and leaks through the tail portion's seal may be the reason for the slight drop in CO₂ levels. During summer storage,

increase of CO₂ and decrease of O₂ levels were observed in both years. This indicated higher biological activity during summer storage. Bartosik et al. (2008a) also noticed the same trend of increased CO₂ and decreased O₂ concentrations over a storage period while storing wheat, soybean, and sunflower seeds in silo bags. They also noticed higher rates of reduction in O₂ and increases in CO₂ concentrations while storing wet grains compared to dry grains. Bartosik et al. (2008b) also found a trend of increase in CO₂ concentration inside silo bags with soybeans during the spring in Argentina, and the bags with higher CO₂ concentrations had more spoilage.

The commercial grading test results indicated that damp canola (m.c. around 12%) can be stored up to 5 months (or up to the middle of winter) without any quality deterioration under western Canadian conditions. In the first year of storage, canola lost one grade by the second unloading but there was no grade change in the second year storage. The cooler temperature over a long duration during the second year (2013-14) of storage might have maintained the canola quality for a longer time than in the first year of storage.

The results of our study is in alignment with the findings of Sun et al. (2014) and Sathya et al. (2009), and proved storage temperature plays a major role in quality deterioration during canola storage. Both the studies showed 12% m.c. canola can be stored up to 20 wk without spoilage at $\leq 10^{\circ}\text{C}$, and canola quality declines rapidly if it is stored at $\geq 20^{\circ}\text{C}$. In this experiment, the ambient temperature in autumn and winter was $\leq 10^{\circ}\text{C}$, and in summer temperature was $\geq 20^{\circ}\text{C}$.

4.3. Prediction of CO₂ concentration

Data collected from the silo bags with canola at 8.9, 10.5 and 12.1% moisture contents (storage moisture, monthly mean temperature, day of storage and CO₂ concentration) were analyzed by SAS 9.3 software using PROC REG procedure and a regression model was obtained by the backward selection method (when all the independent variables had significant effect on CO₂ concentration at P<0.001):

$$\text{Log}_{10}(Y_{\text{CO}_2}) = -1.6286 + 0.00616 T + 0.228278 M - 0.00213 \emptyset + 0.00009T^2$$

Where,

Y_{CO_2} = CO₂ concentration (%)

T = Storage temperature (°C)

M= Moisture content of the grain (% , wet basis)

\emptyset = Storage time (days)

The R² value for this model was 0.76 and the root mean square error (RMSE) was 0.196. This regression model indicates that, initial moisture content of the canola plays a major role in production of CO₂ inside the canola bulk. In this model, the β coefficients for temperature, temperature², moisture content, and the storage time and were 0.195, 0.032, 0.763, and -0.434, respectively. The β coefficient value also demonstrates that moisture content of canola is 3.9 times more important than storage temperature for prediction of CO₂ concentration. White et al. (1982) also found storage moisture of wheat was 1.5 times as important as the storage temperature for prediction of

CO₂ production rate. The β coefficient value also indicates that the length of storage period (day of storage) is the major factor in CO₂ production rate prediction (1.9 times as important as storage moisture). But in prediction of CO₂ production rate of wheat, the day of storage had very small effect (White et al., 1982).

The CO₂ concentration was high at initial stages of storage (first 4-6 wk of storage) irrespective of storage moisture and temperature. The higher respiration rate of canola after harvest produces higher amounts of CO₂ during initial stages of storage, which might be the cause for this higher CO₂ concentration at the initial storage periods. It was noticed that CO₂ concentration decreased after 6 wk of storage. Canola tends to go to a quiescent stage after 6 to 8 wk of storage, in that period respiration of canola is almost negligible (Mills, 1989). Jian et al. (2014) found the similar trend of higher interstitial CO₂ concentration at early stages of storage and decrease in CO₂ concentration after reaching the maximum concentrations in stored canola in small glass (300 mL capacity) flasks at 10, 20, 30 and 40°C.

For dry moisture canola, CO₂ concentration stayed around the 1.5% level for most of the storage time (up to 280 days of storage) and the RMSE of prediction for dry moisture canola was 0.12. The error of prediction was high at initial storage time (around 1.3% CO₂), but at later stages of storage the predictability of CO₂ concentration using the regression model was good. The RMSE for prediction of interstitial CO₂ concentration in high moisture canola bags using the model was 0.23, which accounts for 1.7% of CO₂. The prediction error was higher at the summer storage time (after 200 days of storage), and prediction and measured curves were similar at most parts of the storage period (Fig

4.44). These results showed that, wet seeds had a higher rate of increase in CO₂ concentration when compared to dry grains, aligned with the finding of Bartosik et al. (2008a, b) while storing wheat, soybean, and sunflower seeds in silo bags. The increase of CO₂ concentration during spring was also noticed by Bartosik et al. (2008b) inside silo bags with soybeans in Argentina.

For validation of the model, data collected from the year 2 of the effect of storage period (12.4% initial moisture content) on the canola quality experiment were used. The root mean square error of prediction of the model was 0.16 and it accounted for 1.4% CO₂. The measured vs predicted curve for validation data set showed that prediction model had good agreement with measured CO₂ concentrations until 200 days of storage, and had more error during the summer (Fig 4.45). Higher amounts of CO₂ produced by the heated seeds during the summer might have been the reason for higher amount of measured CO₂ after 200 days of storage. The model was developed with the assumption that, average storage moisture remained the same as initial moisture content of canola and the CO₂ production by fungi or insect was not taken into account. But an increase of moisture content of canola at the top layer of the silo bag by condensation of water at the silo bag walls due to the moisture migration was seen in our field experiments (both the studies: an effect of storage moisture and effect of storage time sections 4.1 and 4.2). These wet pockets of canola might have created localized hot spots and mould growth in those spots, which increased CO₂ production during summer due to the higher amount of biological activity.

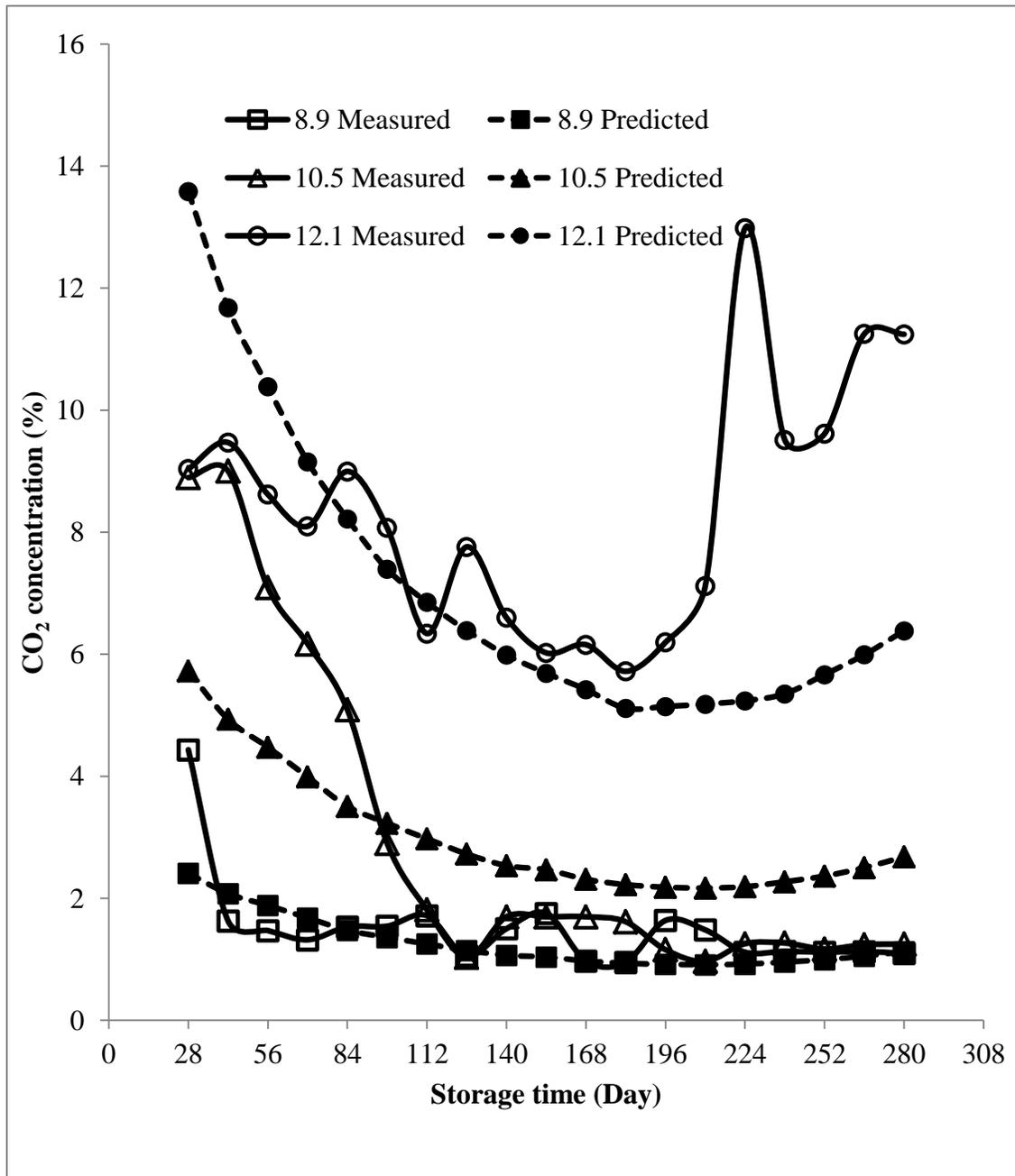


Figure 4.44 Concentration of CO₂ inside the silo bags with 8.9, 10.5, and 12.1% moisture content canola stored for 40 wk (8.9 and 10.5% MC canola stored from Oct 2010 to July 2011, and 12.1% MC canola stored from Oct 2011 to July 2012)

The sensitivity of the developed regression model was compared with the previously developed CO₂ production rate prediction models (White et al., 1982). Data collected from 8.9% and 12.1% moisture content canola silo bags were used as input for these models and compared with measured CO₂ production rate. The measured cumulative CO₂ production of canola in 8.9% moisture content silo bags was 58.48 mg/kg of canola after 28 days of storage, and the predicted CO₂ productions were 32.17 and 0.73 mg/kg of canola from the current and White et al. (1982) models, respectively (Fig 4.46). The predicted CO₂ productions were very low with the White et al. model until 200 days of storage, and rapid increase in CO₂ production was noticed with the increase in temperature in summer (after 200 days of storage). After 250 days of storage, measured cumulative CO₂ was 14.74 mg/ kg of canola, but the predicted CO₂ amount was 119.15 mg/ kg of canola with the White et al. (1982) model. From our model, the predicted amount of CO₂ after 250 days was 13.05 mg/kg of canola. The same trend of lower CO₂ at most parts of the storage and sudden increase after 200 days of storage (or summer time) was noticed in prediction of CO₂ production of 12.1% moisture content canola with the White et al. (1982) model (Fig 4.47). After 14 days of storage, measured CO₂ was 104.77 mg/ kg of canola, and predicted CO₂ with the current and White et al. (1982) models were 140.71 and 3.40 mg/kg of canola, respectively. After 240 days, measured and predicted CO₂ amounts with our developed model and White et al. (1982) were 125.43, 93.91, and 223.37, respectively.

The regression model developed by White et al. (1982) used the CO₂ production rate of wheat stored at four temperatures (10 to 40°C) for developing the prediction

model. The respiration of wheat is totally different than that of canola, where canola produced large amount of CO₂ during the early storage period due to the sweating process (Cofie-Agblor et al, 1998; Proynk et al., 2004). This difference in rate of CO₂ production during the first 4 to 6 wk of storage might have caused the large error in prediction of CO₂ production in canola using White et al. (1982) regression model at early stages of storage. The storage moisture and temperature have significant effect on CO₂ rate prediction with the White et al. (1982) model (standardized regression coefficients (β coefficients) of temperature and moisture were 0.179 and 0.719, respectively), and storage time had a negative effect on CO₂ production rate prediction at early stages of storage. This negative effect of storage time at the start and storage temperature below 10°C up to 200 days of storage might have been the reasons for lower CO₂ production prediction. The rapid increase in CO₂ production prediction after 200 days may have been caused by storage temperature above 10°C and the positive effect of storage time on predicting CO₂ production rate. Abalone et al. (2011a) found that the predictability of the regression model developed by White et al. (1982) was significantly affected by the respiration rate of grain especially in hermetic storage conditions.

Jian et al. (2014) reported 305.6±5.4, 418.3±24.3 mg CO₂ / kg of canola while storing 8 and 12% m.c. canola in 300 mL glass flasks after 30 days of storage at 10°C. These reported values were higher than that measured from the silo bags of this study. They stored canola at airtight conditions for 3 days, and after 3 days canola was transferred to a new flask where fresh air was added in the storage structure. Airtightness of silo bags was totally different than the flasks, as well as absorption CO₂ by canola and

CO₂ diffusion through grain and bag might be the reasons for the difference in accumulated CO₂ production. Proynk et al. (2004) found that, canola with 12% moisture content produced more than 100 mg of CO₂/ kg of canola in 24 hours of storage in 25 to 30°C temperature regime. The regression model developed by Proynk et al. (2004) was tested with the current data from 12.1% moisture content canola silo bag, but it yielded large error in predicting CO₂ production ($\text{Log}(Y_{\text{CO}_2})=2264.75$; where Y_{CO_2} is the mg of CO₂ produced/kg of canola in day 1 (temperature 11.3°C)). Storage moisture of canola had a large effect on predictability of the Proynk et al. (2004) model (parameter estimate of moisture was 181M (Eqn. 3)), which might cause the large error in predicting daily CO₂ production.

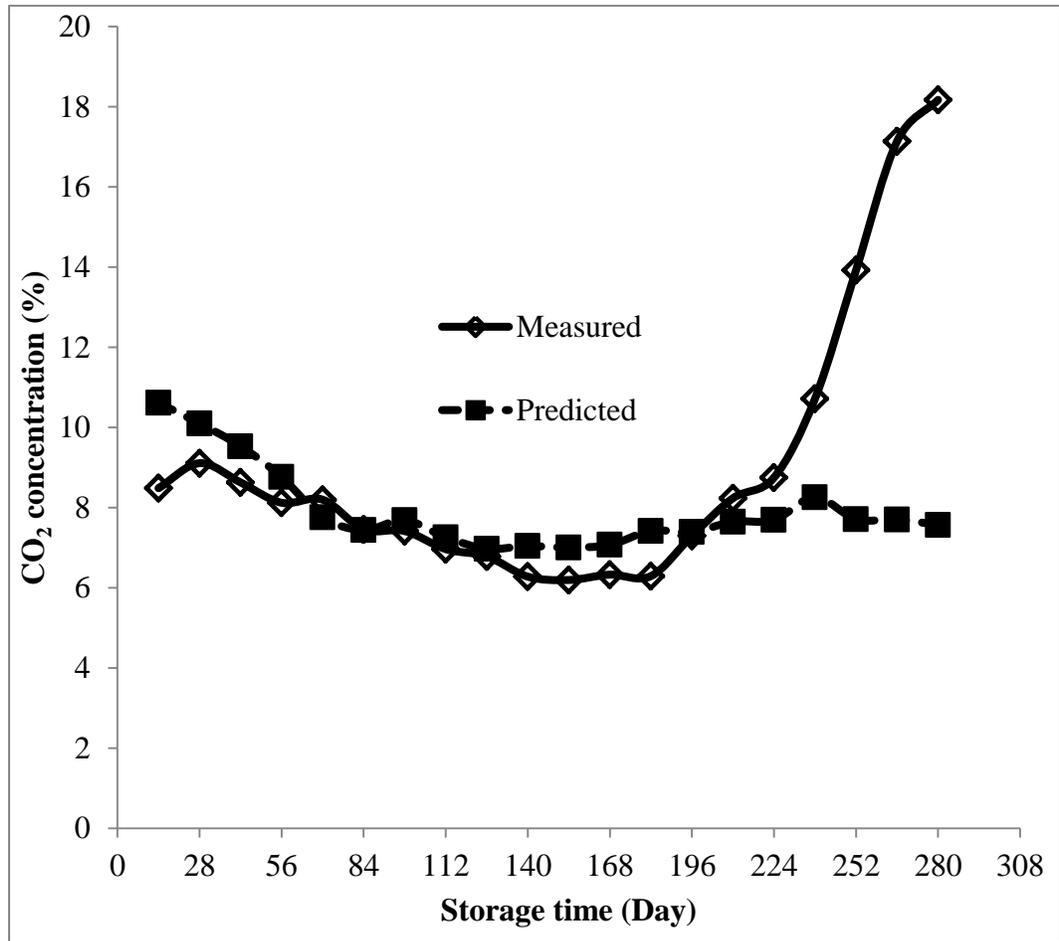


Figure 4.45 Concentration of CO₂ inside the silo bags with 12.4% moisture content canola stored for 40 weeks (from Oct 2013 to July 2014)

Carbon dioxide is the one of the major parameters used to determine the quality of the grain during storage. Bartosik et al. (2008b) measured CO₂ concentration inside the silo bags with wheat using a portable gas analyzer (Model: PBI Dan Sensor, CheckPoint, Denmark) and used these CO₂ concentration values to detect the spoilage of wheat at very early stages. They found positive correlation between CO₂ concentration inside silo bags and spoilage of grain. They perforated the silo bag with needle to collect the air samples for analysis. The air tightness is the major factor for success of silo bag storage, and making perforations affects the integrity of air tightness of the silo bags. The developed prediction model uses moisture content of canola during loading, monthly average temperature during storage and the day of storage to predict CO₂ concentration inside the silo bags. So there is no need to perforate the silo bags for CO₂ concentration measurement. Abalone et al. (2011) developed a lumped-capacity differential model to predict the in the interstitial gas concentrations of a silo bag with wheat, and they used the White et al. (1982) model for calculation of CO₂ production. Our results showed that there were large difference between CO₂ production rates of wheat and canola, and large error while calculation CO₂ production using the White et al. (1982) model.

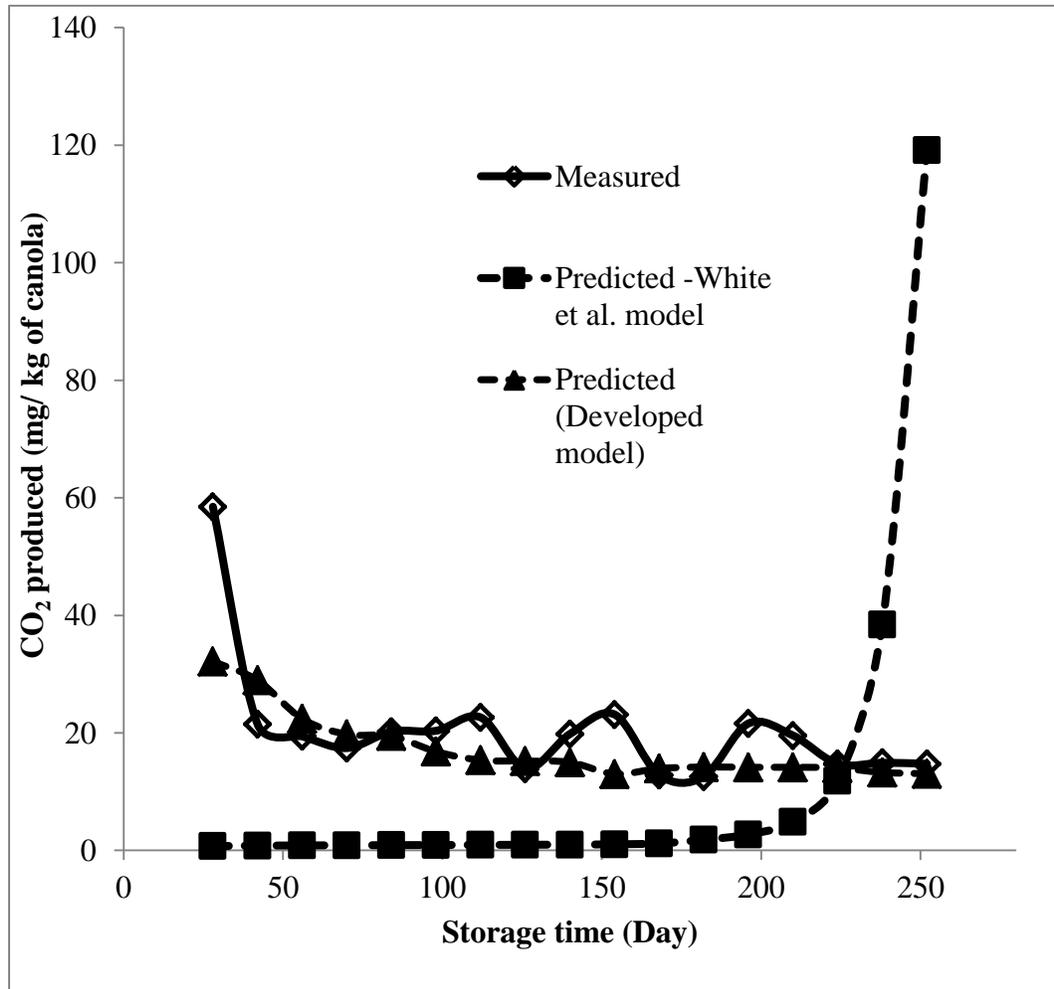


Figure 4.46 Measured and predicted CO₂ production of 8.9% moisture content canola at different times of storage in silo bags

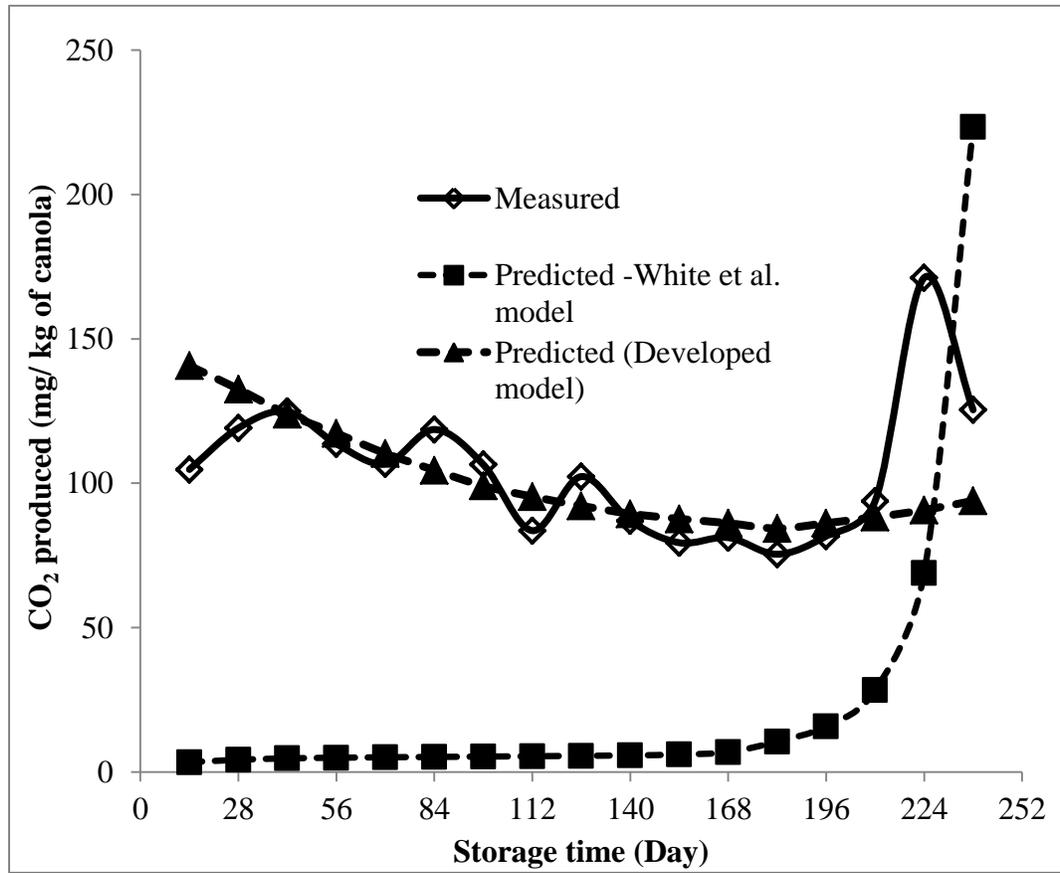


Figure 4.47 Measured and predicted CO₂ production of 12.1% moisture content canola at different times of storage in silo bags

4.4. Permeability of the silo bag

Permeability of new silo bag material for carbon dioxide was $21.64 \pm 0.54 \times 10^{-6}$ $\text{m}^3\text{md}^{-1}\text{m}^{-2}\text{atm}^{-1}$ and after 7 and 10 months of using it in the field with canola the permeability of the material was $23.04 \pm 0.24 \times 10^{-6}$ $\text{m}^3\text{md}^{-1}\text{m}^{-2}\text{atm}^{-1}$, and $29.20 \pm 0.40 \times 10^{-6}$ $\text{m}^3\text{md}^{-1}\text{m}^{-2}\text{atm}^{-1}$, respectively. Permeability for CO_2 of stretched and unstretched bag materials were $29.20 \pm 0.40 \times 10^{-6}$, and $30.70 \pm 1.04 \times 10^{-6}$ $\text{m}^3\text{md}^{-1}\text{m}^{-2}\text{atm}^{-1}$, respectively, after 10 months. Tukey's means comparison test of the data showed that there was significant difference between new and after 10 months used stretched materials for permeability for CO_2 ($\alpha = 0.05$).

Permeability for O_2 for new and 10 months used bag materials were $1.95 \pm 0.36 \times 10^{-6}$ and $3.79 \pm 0.33 \times 10^{-6}$ $\text{m}^3\text{md}^{-1}\text{m}^{-2}\text{atm}^{-1}$, respectively. After 10 months of using the silo bags in the field, permeability of O_2 for stretched and unstretched bag materials were $3.54 \pm 0.80 \times 10^{-6}$, and $3.79 \pm 0.33 \times 10^{-6}$, respectively. Tukey's test proved that, there were significant differences between new and after 10 months silo bag materials. Permeability measurement tests were carried out at the same temperature, but the silo bags were used in the field for 10 months, and were affected by the different environmental factors (temperature, precipitation, snow, UV radiation, and so on). Combination of these effects might have caused the difference in permeability to O_2 and CO_2 .

Guiseng et al. (1995) demonstrated that the permeability of polymer materials could change depending on several factors like the temperature, relative humidity, and properties of polymer materials. Galic and Cikovic (2001) reported that, the water activity of food inside a packaging material also was affected the permeability of the

polymer material. The polymer's structure would change because of the swelling of polymer due to the water activity and it changes the diffusion of gases through it. Mujica-Paz and Gontard (1997) also reported that relative humidity had a large role in change of permeability to O₂ and CO₂ of wheat gluten films, when compared to the effect of temperature. Most of the packaging films (low density polyethylene, polyvinyl chloride, and Polypropylene) used in modified atmospheric packaging have 3 to 7 times higher permeability for CO₂ than O₂ (Mattos et al., 2012). The tested silo bag material is combination of HDPE and LDPE and permeability for CO₂ was 8 to 11 times higher than the permeability for O₂. The mix ratio of HDPE and LDPE might be the reason for higher permeability for CO₂. Abalone et al. (2011a) estimated equivalent permeability of the silo bag plastic material from the permeability of HDPE and LDPE to CO₂ and O₂ values reported by Osborn and Jenkins (1992). The estimated equivalent permeability to O₂ and CO₂ were $9.75 \times 10^{-8} \text{ m}^3 \text{ md}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$ and $3.22 \times 10^{-7} \text{ m}^3 \text{ md}^{-1} \text{ m}^{-2} \text{ atm}^{-1}$, respectively. They made an assumption of half of the plastic layer was HDPE and the other half was LDPE. But the actual composition of HDPE and LDPE for manufacture of silo bag material is unknown, and this might be the reason for the difference in permeability to O₂ and CO₂ values. Abalone et al. (2011b) reported that, even a small perforation in a silo bag had a significant effect on permeability to the gases and they also reported that, filling and sealing of silo bags also had significant effects on permeability to O₂ and CO₂.

Table 4.2 Permeability of the silo bag (n=5)

S. No	Silo bag Material	Permeability for CO ₂ (m ³ md ⁻¹ m ⁻² atm ⁻¹)	Permeability for O ₂ (m ³ md ⁻¹ m ⁻² atm ⁻¹)
1	New (unstretched)	21.61 ±1.50× 10 ⁻⁶ a	1.95 ±0.36× 10 ⁻⁶ a
2	After 7 months- unstretched	23.04 ±2.04× 10 ⁻⁶ a,b	2.49 ±0.24× 10 ⁻⁶ a,b
3	After 7 months- stretched	23.38 ±3.72× 10 ⁻⁶ a,b	2.54 ±0.31× 10 ⁻⁶ a,b
4	After 10 months- unstretched	29.20 ±3.40× 10 ⁻⁶ a,b	3.54 ±0.80× 10 ⁻⁶ b,c
5	After 10 months- stretched	30.70 ±2.47× 10 ⁻⁶ b	3.79± 0.33× 10 ⁻⁶ c

* Numbers marked with same character (a,b,c) in same column is statistically same using Tukey's means comparison test ($\alpha=0.05$)

Chapter 6

Conclusions

Canola was stored at 8.9% (dry), 10.5% (straight) and 14.4% (damp) moisture contents for 40 wk in silo bags. For dry grade canola, germination and FAV stayed at an acceptable level of quality and there was no change in commercial grade for up to 40 wk of storage in silo bags under western Canadian conditions. Germination of straight grade canola stayed at safe levels in most parts of the silo bags, but it did drop below 80% of its initial germination in the top layer of the silo bags. The commercial grade of the straight grade canola was downgraded by one grade after 40 wk of storage. Inside the damp grade bags, the germination dropped below safe storage levels and FAV increased twice of its initial value within 8 wk of storage. Damp canola was downgraded to animal feed grade after 40 wk of storage and also caked up due to mould growth during storage in silo bags which made use of a grain bag unloader impossible for unloading. Moisture migration due to temperature gradients inside silo bags (between bottom and top layers of the grain) was noticed in all three moisture contents. In winter, moisture content of the top layer of the grain in silo bags was high due to the condensation and at the same time temperature

of top layer was lower than the other parts of the bag. Carbon dioxide levels inside the silo bags were constant after higher CO₂ production due to higher respiration rate in first 6 to 8 wk of storage, and increase in CO₂ levels were noticed during summer storage in straight and damp moisture content canola due to higher biological activity. The results from the effect of storage moisture content, and storage time experiments indicated that, dry (8% m.c.), straight (10% m.c.), and damp (14% m.c.) grade canola can be stored up to 40, 24, and 4 wk, respectively after harvest without any quality loss under Canadian Prairie conditions.

When storing 12% moisture content canola in silo bags, moisture content of canola at the top layer of the silo bags increased during summer storage. The germination also dropped below 30% at the top layer during summer storage. The FAV values increased more than 2-fold of its initial values after 40 wk of storage. Germination and FAV values stayed within safe storage levels up to the late winter. Carbon dioxide and O₂ levels inside the silo bags indicated higher levels of biological activity during summer. Commercial grading results proved that, temperature rise in the spring and summer period had a significant role in changes in canola quality parameters during storage in silo bags. Canola with 12% moisture content could be stored up to 20 wk (or up to the late winter) without any quality deterioration under the western Canadian conditions. Storing wet canola in the summer season might cause increase in biological activity inside the silo bag resulting in increased deterioration of the canola.

A polynomial quadratic regression model was developed to predict the carbon dioxide concentration inside silo bags with canola from the experimental data collected in

this study. The R^2 value of the developed model was 0.76, and root mean square errors for predicting CO_2 concentration inside silo bags with 8.9%, and 12.1% moisture content canola were 0.12, and 0.23, respectively. Validation of the model with field data collected from silo bags with 12.4% m.c. canola yielded a RMSE of 0.16. This quadratic model performed well when compared with the previously developed prediction models. Since CO_2 is a good indicator of grain spoilage, this developed model can be used to predict grain quality inside silo bags without damaging the bag integrity. The CO_2 concentration inside a silo bag can be predicted with this model using average monthly temperature, initial moisture content of the canola and days of storage as inputs. Predictability of CO_2 concentration with the current model was low after 200 days of storage (especially in summer storage).

Permeability of the silo bag material to CO_2 and O_2 were measured using a specially designed testing unit. At room temperature (24°C), permeability of new silo bag material to CO_2 and O_2 were $21.61 \pm 1.50 \times 10^{-6}$, and $1.95 \pm 0.36 \times 10^{-6} \text{ m}^3\text{md}^{-1}\text{m}^{-2}\text{atm}^{-1}$, respectively. Exposure to the environment and stretching of bags while loading the grain into silo bags had significant effects on the permeability to CO_2 and O_2 .

Chapter 7

Future Studies

Analysis of gas samples from the silo bags showed a significant difference in gas concentration at the tail portion than the other portions of the silo bag. Sealing of the tail portion (end part) of the silo bag might have resulted in leaking of some air even if the bag had no perforations, which affected the CO₂ concentration inside the bags. Alternate sealing techniques should be tested to get proper sealing of the ends to protect a silo bag's air tightness during storage.

Jian et al. (2015a, b) developed three-dimensional transient models to predict temperature of the soil covered by a silo bag, temperature, and moisture content of the canola inside the silo bag. These models can be coupled with the CO₂ concentration prediction models and a new three-dimensional model can be developed to predict CO₂ levels inside silo bags. This model will help farmers and grain storage managers as a tool to predict the quality of grain inside silo bags without damaging the silo bags' integrity for air tightness.

Insect infestation is not a big issue in storage of canola, and there were no insect infestations found in our experiments. Most of the quality losses occurred in our experiments caused by mould growth. But in cereal grains and pulses storage, insect infestation is one of the major issues along with fungal infection. Feasibility of silo bags to store cereal grains and pulses under Canadian Prairie conditions have to be studied in order to find the adaptability of these silo bags on western Canadian farms.

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Appendix

Table A. 2 Factorial test statistics of moisture content and germination data of effect of storage moisture content experiment

Analysis	Parameter	F value	P value
Moisture content	Initial moisture content (%)	672.71	<.0001
	Storage time (week)	3.05	0.0008
	Sampling location	8.06	<.0001
	Initial moisture content and storage time	1.27	0.1869*
	Storage time and sampling location	1.33	0.0491
	Initial moisture content and storage time and sampling location	1.16	0.1319*
Germination	Initial moisture content (%)	646.26	<.0001
	Storage time (week)	70.62	<.0001
	Sampling location	126.77	<.0001
	Initial moisture content and storage time	26.46	<.0001
	Storage time and sampling location	53.53	<.0001
	Initial moisture content and storage time and sampling location	6.19	<.0001

* No significant difference at P=0.05 level

Table A. 3 Factorial test statistics of FAV and CO₂ concentration data of effect of storage moisture content experiment

Analysis	Parameter	F value	P value
FAV	Initial moisture content (%)	91.42	<.0001
	Storage time (week)	37.87	<.0001
	Sampling location	1.72	0.1787
	Initial moisture content and storage time	4.96	<.0001
	Storage time and sampling location	3.64	0.0059
	Initial moisture content and storage time and sampling location	1.73	0.0005
	CO ₂ concentration	Initial moisture content (%)	0.61
	Storage time (week)	1592.23	<.0001
	Sampling location	345.24	<.0001
	Initial moisture content and storage time	90.13	<.0001
	Storage time and sampling location	2.00	0.0941
	Initial moisture content and storage time and sampling location	0.27	1.0000

* No significant difference at P=0.05 level

Table A. 4 Factorial test statistics of moisture content and germination data of effect of storage time experiment

Analysis Parameter	Parameter	F value	P value
Moisture content	Storage year	223.26	<0.001
	Storage time (week)	20.35	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	19.66	<.0001
	Sampling location along length of bag (Head, Centre, and Tail)	0.94	0.3925*
	Germination	Storage year	276.93
	Storage time (week)	294.90	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	8.87	0.0002
	Sampling location along length of bag (Head, Centre, and Tail)	51.89	<.0001

* No significant difference at P=0.05 level

Table A. 5 Factorial test statistics of FAV and CO₂ concentration of effect of storage time experiment

Analysis Parameter	Parameter	F value	P value
FAV	Storage year	2.26	0.1333*
	Storage time (week)	12.95	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	2.25	0.1062*
	Sampling location along length of bag (Head, Centre, and Tail)	0.60	0.5498*

* No significant difference at P=0.05 level

Table A. 6 Factorial test statistics of CO₂ data of effect of storage time experiment

Analysis Parameter	Parameter	F value	P value
Year1(2011-12)	Storage time (week)	194.37	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	4.87	0.0080
	Sampling location along length of bag (Head, Centre, and Tail)	6.24	0.0021
Year2 (2013-14)	Storage time (week)	330.25	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	50.49	<.0001
	Sampling location along length of bag (Head, Centre, and Tail)	0.32	0.7232*

* No significant difference at P=0.05 level

Table A. 7 Factorial test statistics of O₂ data of effect of storage time experiment

Analysis Parameter	Parameter	F value	P value
Year1(2011-12)	Storage time (week)	23.88	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	3.32	0.0368
	Sampling location along length of bag (Head, Centre, and Tail)	0.06	0.9385*
Year2 (2013-14)	Storage time (week)	197.10	<.0001
	Sampling location along height of bag (Top, middle, and Bottom)	18.67	<.0001
	Sampling location along length of bag (Head, Centre, and Tail)	1.73	0.1779*

* No significant difference at P=0.05 level

Table A. 8 Summary of regression analysis statistics of CO₂ prediction model development

Variable	Parameter Estimate	Standard Error	Type II SS	F Value	P Value	Standardized Estimate (β coefficient)
Intercept	-1.62860	0.13421	5.62299	147.26	<.0001	0
Temperature	0.00616	0.00150	0.64667	16.94	<.0001	0.19466
Moisture Content	0.22828	0.01174	14.44378	378.27	<.0001	0.76252
Day	-0.00213	0.00024517	2.86999	75.16	<.0001	-0.43408
Temperature²	0.00009385	0.00012655	0.02100	8.84	0.0034	0.03237

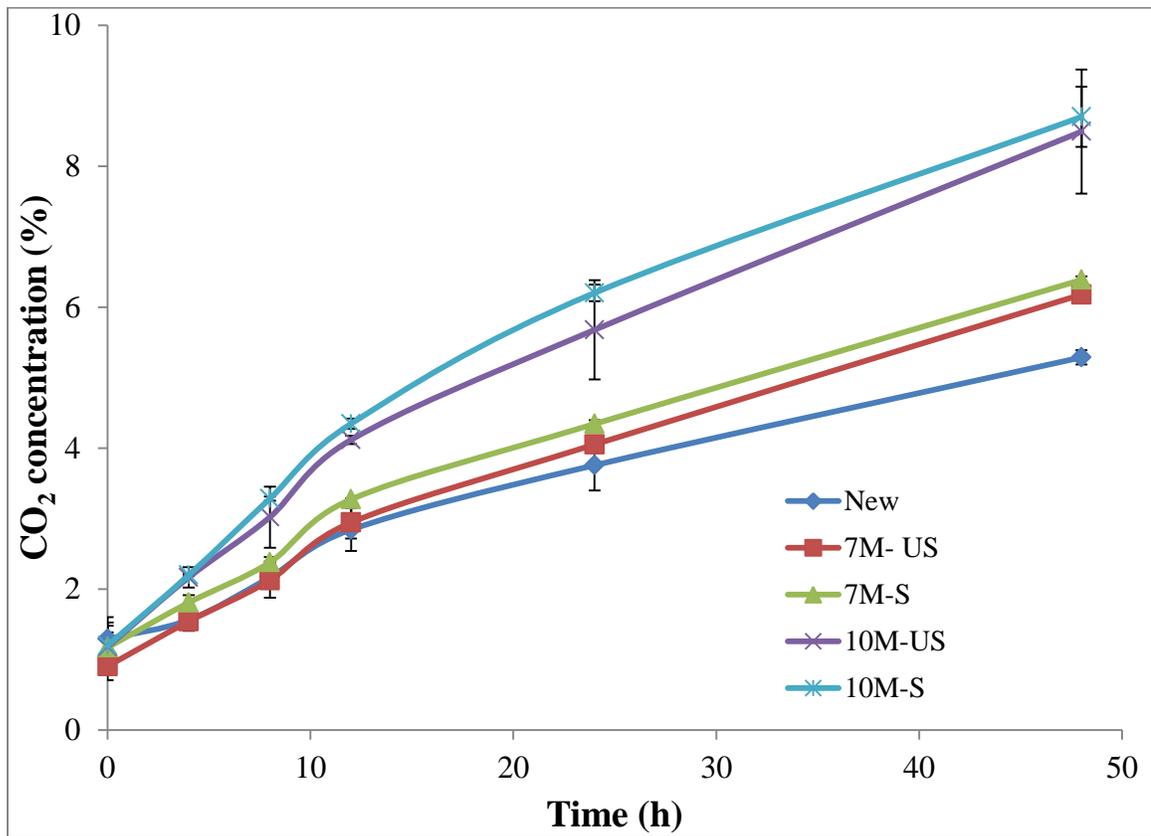


Figure A. 1 Carbon dioxide concentration in testing chamber permeability testing unit with different bag materials (New, 7M-US: 7 month used-Unstretched, 7M-S: 7 month used- Stretched, 10M-US: 10 month used-Unstretched, 10M-S: 10 month used- Stretched)

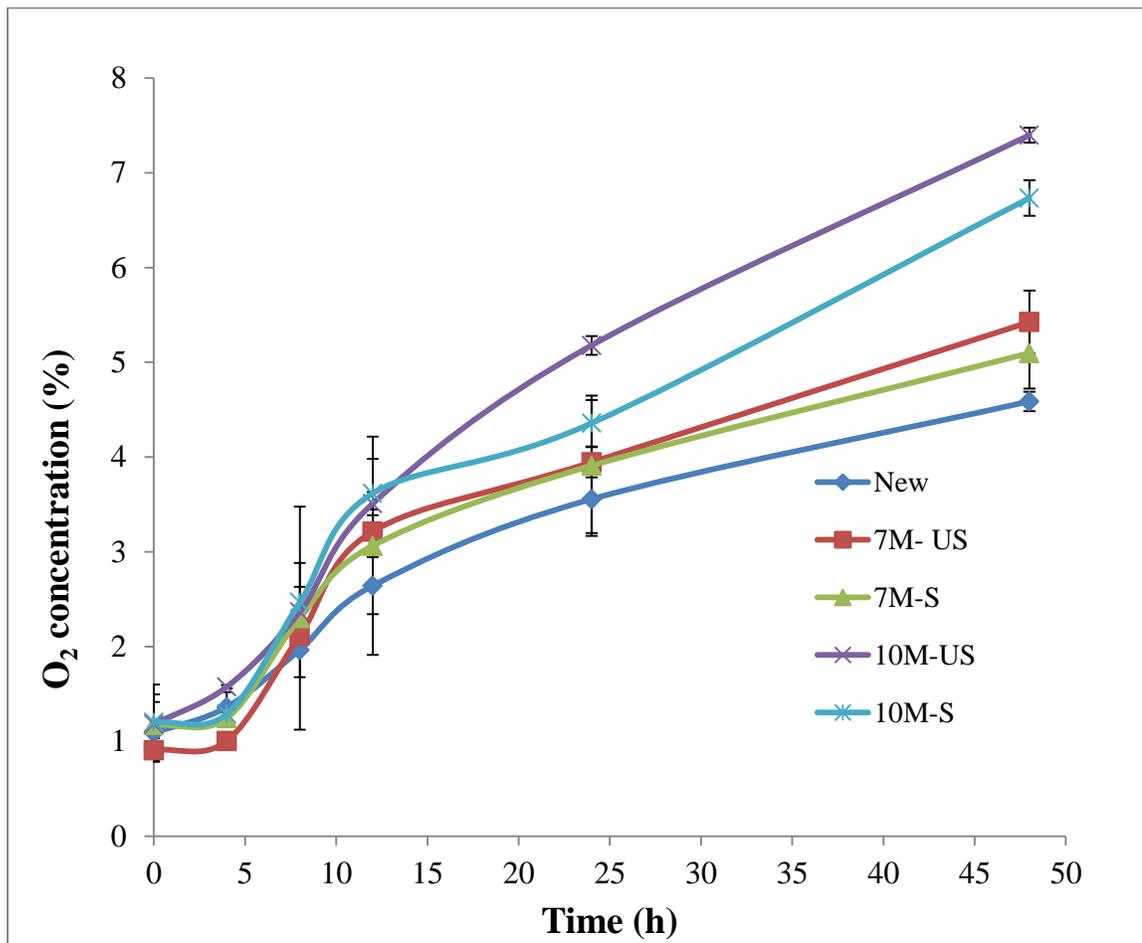


Figure A. 2 Oxygen concentration in testing chamber permeability testing unit with different bag materials (New, 7M-US: 7 month used- Unstretched, 7M-S: 7 month used- Stretched, 10M-US: 10 month used- Unstretched, 10M-S: 10 month used- Stretched)