Gas leakage through samples of wall seams of bolted-metal bins

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Lukasiewicz, M., Jayas, D.S., Muir, W.E. and White, N.D.G. 1999. **Gas leakage through samples of wall seams of bolted-metal bins.** Can. Agric. Eng. 41:065-071. Laboratory experiments were conducted to quantify the leakage of carbon dioxide (CO_2) through samples of wall seams of bolted-metal bins by determining pressure decay times and CO_2 retention rates from a sealed container. Samples of vertical and horizontal seams of a typical grain bin were tested with different methods of sealing and a range of initial CO_2 concentrations. The performances of vertical seams sealed with a single or double rows of butyl caulking were similar, but they were significantly different from uncaulked horizontal seams. An additional protective coating of a flexible membrane resin was not effective when other caulking had been applied previously but was effective when no caulking was used. Pressure decay times were not affected by initial CO_2 concentrations in the bin, but CO_2 retention coefficients were lower for higher concentrations. Pressure decay times were shorter and CO_2 retention coefficients were lower in containers filled with grain than in empty ones.

Des expériences ont été faites en laboratoire pour quantifier les fuites de dioxyde de carbone (CO_2) à travers différents types de joints de parois de silo en métal boulonné, en déterminant le temps de dépressurisation et le coefficient de rétention du CO_2 de conteneurs scellés. Des échantillons de joints verticaux et horizontaux d'un silo à grains typique furent testés en utilisant différentes méthodes de colmatage et une gamme de concentrations initiales de CO_2. Les performances de joints horizontaux scellés avec une ou deux couches de calfeutrage au butyle ont été semblables, mais ont été significativement différentes de celles de joints horizontaux non-calfeutrés. L'addition d'une couche protectrice constituée d'une membrane flexible en résine n'a eu aucun effet lorsque du calfeutrage avait déjà été appliqué, mais a été efficace lorsqu'il n'y avait pas eu d'application de calfeutrage. Les temps de dépressurisation n'ont pas été influencés par la concentration initiale de CO_2 dans le silo. Par contre les coefficients de rétention du CO_2 étaient plus faibles pour des concentrations plus élevées. Dans des conteneurs remplis de grains, les temps de dépressurisation étaient plus courts et les coefficients de rétention du CO_2 plus faibles.

INTRODUCTION

Controlled atmosphere (CA) storage is a potential alternative to synthetic chemical fumigation for insect control in stored grain. In the CA treatment, the intergranular gas composition in a grain bulk is altered by injecting either carbon dioxide (CO_2) to create high-CO_2 atmospheres, or nitrogen (N_2) to create low-oxygen (O_2) atmospheres lethal to stored-product pests (Banks and Annis 1977). The objective of the CA treatment is to kill all insects and mites with a minimal use of gases in the shortest time. The effectiveness of CA for controlling various stored-product pests depends on the temperature and moisture content of the grain, species and life stages of pests, gaseous composition, uniformity of gas distribution, and exposure time of the CA treatment.

Based on a review of 70 years of literature (1900-1970), Annis (1986) stated that the majority of species showed 95% or greater mortality in less than 10 d at CO_2 levels of 40 to 60% at an optimum temperature range 20-29°C. Declines in CO_2 concentration during treatment occur in non-airtight structures, but even under such conditions 95% mortality can be obtained in 15 d when concentrations decline from 70 to 35% (Jay 1980). The Australian recommendation for the control of stored-product insects is the maintenance of a CO_2 concentration of more than 35% for a period of 10 to 14 d (Barry 1984).

Leakage of the CA gases from the bins is the main obstacle to practical implementation of the CA technology. There are generally two ways to manage the leakage problem: seal the structures or optimize the CA technology.

In Australia, many of the grain storage structures are 'airtight,' which means they meet a pressurization test criterion. A structure is regarded as satisfactorily sealed, if a structure has a half-life time of at least 5 min, i.e., it takes at least 5 min for an initial increased air pressure to drop from 2500 to 1250 Pa (Banks and Annis 1984). The on-farm storage bins in Canada are not airtight (White and Jayas 1993). In North America, two thirds of all grain storage facilities are bolted, galvanized-steel bins which contain many leaks and do not meet the Australian pressurization criterion. To avoid CA-fumigation failures in non-airtight bins without resorting to unnecessarily high standards of sealing and costly modifications to storages, the CA technology can be optimized. McGaughey and Akins (1989) successfully carried out a two stage process for controlling insects in leaky grain bins. The initial stage purges the bin to the required CA composition. In the second, maintenance stage, gas is added to compensate for any gas loss during the required exposure time. An understanding of the gas distribution in the grain bulk and loss of CA gases should assist in successful application of CA (Alagusundaram 1993). From predictions of gas losses expected under particular environmental conditions for any bin, guidelines for compensation of the CA-gas losses could be developed.

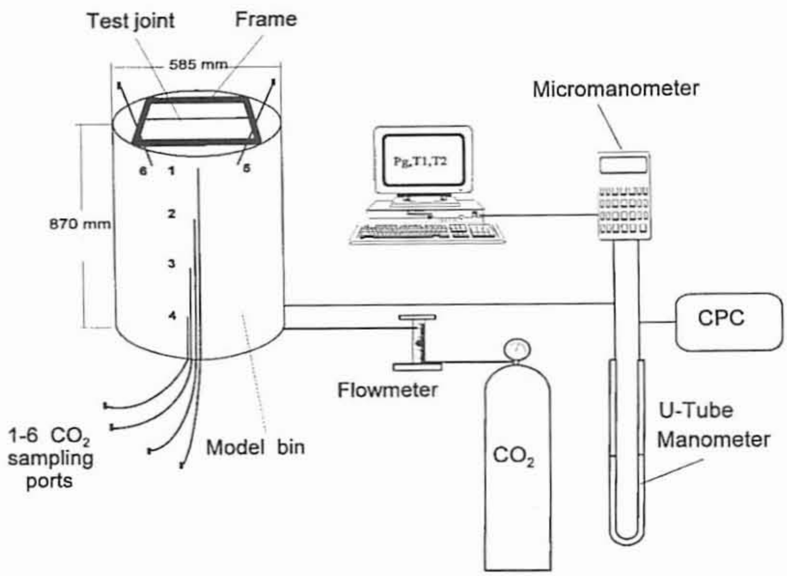

Fig. 1. Schematic diagram of the experimental setup. One of the sampling ports of the model bin was used for pressurization.

Mathematical models may be used for predicting the loss of CA gases from the bins (Peck 1994). There is a need for accurate experimental data on gas loss through the leaks in bolted metal bins, which would serve as input data for the mathematical models.

The objective of this study was to quantify the CO_2 leakage through the seams of bolted-metal bins by measuring the pressure decay times and CO_2 retention rates. The effects of sealing method, initial CO_2 concentrations in the container, and the presence of grain in the test container were also determined.

MATERIALS and METHODS

Experimental design

Experiments were conducted to determine the influence of the sealing methods of wall seams, the initial concentrations of CO_2, and the presence of grain in the bin on the time for the differential pressure to decay to one-half the initial value, and the CO_2 retention coefficient. The experimental design was a Completely Randomized Design (CRD). The treatment structure was a 3-way factorial (4x4x2) with the factors as follows: sealing method (3 levels + control); initial CO_2 concentration (4 levels) created by purging the container with CO_2 for 0, 15, 30, and 60 min; empty container and container filled with grain (2 levels). There were a total of 128 tests consisting of four replicates, four sealing methods (S0, S1, S2, S3), four initial CO_2 concentrations (C0, C15, C30, C60), and 2 levels of the grain presence in the bin (G-grain, E-empty).

Two additional trials were conducted: 1) the CO_2 concentration was monitored for 5 d after CO_2 purging for 60 min in the bin filled with wheat; and 2) the pressure and CO_2 concentration decay in 24 h were measured for the CO_2 purging time of 60 min in the empty bin after the joints were covered with a new (unnamed) protective coating from Niagara Protective Coatings (Niagara Falls, ON).

Experimental setup

The model bin was a steel barrel (oil drum) 0.585 m in diameter and 0.870 m tall (Fig. 1). The bin was filled with 160 kg of Canadian hard red spring wheat, cultivar 'Katepwa', graded No.1 by the Canadian Grain Commission. The wheat had 0.5% dockage by mass and 11.8% moisture content (m.c.) (on a wet mass basis). To create the desired initial CO_2 concentrations, the bin was connected through a flowmeter (Series 50, Advanced Specialty Gas Equipment Corp., South Plainfield, NJ) to the tank of compressed CO_2. The flowmeter was adjusted to give a constant flow rate of 2.4 L/min of CO_2.

Four types of seams and sealing methods were prepared for testing: sample 0 - control sample without any seam, sample 1 - a vertical seam (285 mm long) with a double row of butyl caulking, sample 2 - a vertical seam (285 mm long) with a single row of butyl caulking, and sample 3 - a horizontal seam (295 mm long) without caulking. Samples 1, 2, and 3 were similar to the seams used in the bolted metal bins manufactured by Westeel (Winnipeg, MB). For samples 1 and 2, butyl caulking was applied between the metal sheets that overlapped 60 mm and the joints were tightened with 5 bolts spaced 65 mm apart (Fig. 2; right). For sample 3, the metal sheets overlapped 33 mm and the joint was tightened with 2 bolts spaced 250 mm apart (Fig. 2; left). The torque applied to each bolt was 24 N·m (the torque recommended by the manufacturer). The metal samples were framed with wood fastened to the top of the test bin with 12 bolts (Fig. 2, right). Silicone was applied to the junction between the frame and the bin prior to tightening the bolts. Additional silicone was applied to the joints around the tightened frame.

A reference pressure chamber (RPC) provided a reference pressure equal to the atmospheric pressure at the start of each experiment. A constant volume of air at the barometric pressure at the beginning of each test was contained in the RPC for the duration of the whole test. The differential (between the pressure in the bin and the reference pressure in the RPC) was measured. Sharp variations in the atmospheric pressure did not affect the pressure difference between the bin and the RPC.

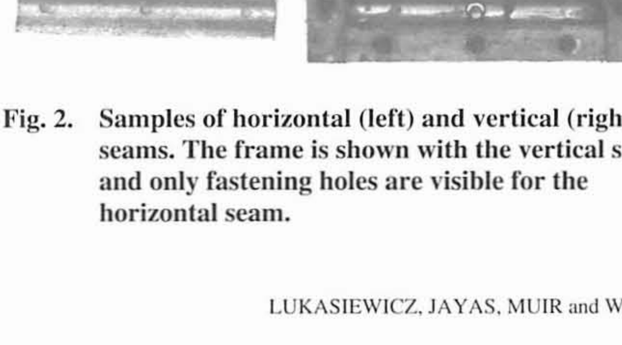

Fig. 2. Samples of horizontal (left) and vertical (right) seams. The frame is shown with the vertical seam and only fastening holes are visible for the horizontal seam.

Table I. Pressure decay times (min) to half level using bin filled with wheat (11.8% m.c.) and empty bin. The bin was purged with carbon dioxide and after an hour pressurized with air.

Sample Type	Replicate number	Filled						Empty						PC*
		Purging time (min)						Purging time (min)						
		0	15	30	60	Mean	SD	0	15	30	60	Mean	SD	
Sample 0	S01	540	405	405	405	439	68	645	840	870	510	716	170	
control	S02	450	285	315	285	334	79	510	435	465	450	465	32	
-no seam	S03	465	375	420	390	413	40	540	480	615	555	548	55	
	S04	480	390	360	345	394	60	435	435	435	420	431	8	
	Mean	484	364	375	356	395	60	533	548	596	484	540	46	
	SD	39	54	47	54	45		87	196	199	60	127		
Sample 1	S11	39	64	40	68	53	15	239	144	144	164	173	45	163
2 row caulking	S12	53	51	48	37	47	7	69	52	142	46	77	44	60
	S13	18	19	19	17	18	1	19	22	30	24	24	5	19
	S14	67	45	45	49	52	11	116	129	119	122	122	6	112
	Mean	44	45	38	43	42	3	111	87	109	89	99	13	89
	SD	21	19	13	21	16		94	59	54	65	63		63
Sample 2	S21	19	17	15	16	17	2	35	26	35	29	31	5	29
1 row caulking	S22	1.96	2.06	2.25	2.38	2	0.19	3.92	3.5	4.08	4.17	4	0.30	30
	S23	409	341	174	124	262	135	427	383	278	218	327	96	238
	S24	288	159	110	71	157	94	256	208	214	195	218	26	290
	Mean	179	130	75	53	109	57	180	155	133	112	145	30	147
	DS	201	158	81	56	123		199	177	134	111	154		137
Sample 3	S31	0.33	0.32	0.33	0.35	0.33	0.01	0.53	0.53	0.57	0.63	0.57	0.05	55
no caulking	S32	0.12	0.2	0.22	0.23	0.19	0.05	0.33	0.3	0.4	0.42	0.36	0.06	63
	S33	0.13	0.15	0.15	0.17	0.15	0.02	0.23	0.27	0.27	0.28	0.26	0.02	15
	S34	0.68	0.72	0.7	0.65	0.69	0.03	1.15	1.18	1.22	1.17	1.18	0.03	4
	Mean	0.32	0.35	0.35	0.35	0.34	0.02	0.56	0.57	0.62	0.63	0.59	0.03	34.3
	DS	0.26	0.26	0.24	0.21	0.24		0.41	0.42	0.42	0.39	0.41		29.1

*The wall's seam covered with the additional protective coating and a purging time of 60 min was used.

Differential pressures were measured with an electronic micromanometer (MP6KSR, Neotronics, Oxford, England) and checked with a U-tube manometer filled with water. The accuracy of the micromanometer was 2.5%. All tubing connections were sealed with silicone.

Temperatures were measured with copper-constantan thermocouples located inside the bin 100 mm below the sample and outside the bin. A 12-bit data acquisition system with output linearization and thermocouple cold junction compensation was used for monitoring the differential pressure and temperatures. The data were recorded by a computer.

Samples of intergranular air for determining CO_2 concentrations in the bin were withdrawn through semi-rigid nylon tubing, 3.2 mm outside diameter, installed at four levels, spaced 210 mm apart in the vertical direction along the central axis of the bin, and at two points 10 mm below the sample. The outer ends of the gas sampling tubes were fitted with rubber septa. Before collecting gas samples, the gas sampling tubes were flushed by drawing 10 mL of gas, which was discharged into the room air. After flushing, a further 10 mL of gas were withdrawn for analysis. Gas samples were collected using 10-mL syringes and analysed for CO_2 concentrations using a gas chromatograph (GC) (Model 5890A, Hewlett-Packard Company, Avondale, PA) with a thermal conductivity detector and steel columns packed with Porapak N and molecular sieve 5A in series; the GC was connected to an integrator (Model 3396A, Hewlett-Packard Company, Avondale, PA).

Experimental procedure

The seam samples for testing were chosen at random. For each replicate, first, the tests with grain were carried out for the four initial CO_2 concentrations, then, after unloading the bin, the empty bin tests were conducted. To obtain an initial uniform distribution of gas inside the bin, every test began 1 h after the CO_2 purging. Initial concentrations of CO_2 of approximately 0, 15, 30, and 60% were obtained by purging the container with CO_2 for 0, 15, 30, and 60 min, respectively. The CO_2 supply was shut off after purging and the container was pressurized with a hand pump to an initial differential pressure of 2.5 kPa, the pressure recommended by Banks and Annis (1977) for testing airtight bins. The decay of the differential pressure to

Table II. Analysis of variance of the pressure decay times (min) to half level as influenced by the method of sealing, initial CO_2 concentration, and presence of grain in the bin.

Source of variation ψ	Degrees of freedom (DF)	Sum of squares (SS)	Mean square (MS)	F value	Pr>F
S_i	3	4121865	1373955	148.47	0.0001% **
C_j	3	38929	12976	1.4	0.25% ns
G_k	1	112756	112756	12.18	0.007% **
S_i x C_j	9	41353	4595	0.5	0.87% ns
S_i x G_k	3	91678	30559	3.3	0.024% *
C_j x G_k	3	13729	4576	0.49	0.69% ns
S_i x C_j x G_k	9	25861	2873	0.31	0.97% ns
Error	96	888395	9254		
Total	127	5334567			
Contrast	DF	Contrast SS	Contrast MS	F value	Pr>F
S0 vs Others	1	3863787	3863787	417	0.0001% **
S1 vs S2	1	51264	51264	5.54	0.021% *
S2 vs S3	1	257098	257098	27.78	0.0001%**
C0 vs Others	1	26872	26872	2.9	0.092% ns
C15 vs C30	1	0.57	0.57	0	0.99% ns
C30 vs C60	1	8971	8971	0.97	0.33% ns

** = highly significant effect at 1% significance level.
* = significant effect at 5% significance level.
ns = not significant effect.
ψ S_i = the effect of the ith method of sealing; C_j=the effect of the jth initial CO_2 concentration; G_k=the effect of the kth level of the grain presence in the bin.

one-half the initial value was recorded by the data acquisition system, every 15 min for sample 0, every 1 min for sample 1 and sample 2, and every 1 s for sample 3.

The CO_2 concentrations were measured before the bin was pressurized and 1, 3, 6, and 24 h after the initial sampling. For the long-term tests, which lasted 5 d, CO_2 concentrations were measured every 24 h.

RESULTS and DISCUSSION

Pressure decay times

For the tests conducted in the bin filled with wheat, the times for the differential pressure to decay to one-half the initial pressure (2.5 to 1.25 kPa) varied among the samples, from 285 to 540 min for the control sample 0 (no seam), 17 to 68 min for sample 1 (2 row caulking), 1.96 to 409 min for sample 2 (1 row caulking), and from 0.15 to 0.72 min for sample 3 (no caulking). For the tests carried out using the empty bin, the pressure decay times were generally longer than for tests done using the bin filled with grain (Table I). The differences between the empty and filled bins may have been caused by sorption of CO_2 by grain and the fact that more CO_2 was present in the empty bin and thus may take longer to leave to cause a reduction in pressure. Unexpectedly, the decay times and variability among replicates for 1 row caulking (sample 2) were greater than for 2 row caulking (sample 1).

An analysis of variance was performed on the pressure decay times (Table I) using the General Linear Model (GLM) Procedure from SAS (SAS 1986) (Table II). The experiment was reliable because the variations in the results were reasonably taken into account by the model (83.4%) as is indicated by the coefficient of determination (r^2) value of 0.834. The effects of the method of sealing and presence of grain in the bin were highly significant at the 1% significance level. Interactions among the factors were not significant at the 1% level.

The differences between the control sample with no seam and the remaining samples, as well as between the horizontal seam and samples with the vertical seams were highly significant. The means of the pressure decay times between one and two rows of caulking were different at the 5% significance level. Initial CO_2 concentration did not have a significant effect on the pressure decay times.

Effects of the differences between internal and external temperatures of the bin and the barometric pressure on the repeatability of the pressure decay tests

During the experiments, sudden changes in the differential pressure in the bins were observed in some tests. With the assumption made at the beginning of the experiment that the temperatures inside and outside the bin would be constant (the experiment was conducted in a heated building) and the reference pressure was set at the barometric pressure at the beginning of each test in the RPC, sudden changes in differential pressures were not expected. An analysis of the recorded temperatures and barometric pressures revealed that, during some tests, sharp changes in the outside temperature had occurred. In the RPC, a constant volume of air was entrapped at the atmospheric pressure at the start of each experiment. A sudden decrease in the outside temperature may have caused a sudden drop in the temperature of the laboratory followed by a drop in the reference pressure in the RPC. The drop in the reference pressure in the RPC could be the reason for the increases in the differential pressure between the RPC and the test bin. The

Table III. The retention coefficients (%) after 24 h for the empty bin and filled with wheat (11.8% m.c.). The bin was purged with carbon dioxide and after an hour pressurized with air.

Sample Type	Replicate Number	Filled Purging Time (min)					Empty Purging Time (min)					PC*
		15	30	60	Mean	SD	15	30	60	Mean	SD	
Sample 0	S01	97.8	97.2	96.6	97.2	0.6	105.8	102.1	99.5	102.5	3.2	
control	S02	100.8	97.4	96.5	98.2	2.3	100.3	101.1	99.5	100.3	0.8	
-no seam	S03	99.7	97.5	98.2	98.5	1.1	99.6	99.2	98.5	99.1	0.6	
	S04	99.5	99.7	97.4	98.9	1.3	97.7	98.5	98.9	98.4	0.6	
	Mean	99.5	98.0	97.2	98.2	1.2	100.9	100.2	99.1	100.1	0.9	
	SD	1.2	1.2	0.8	0.7		3.5	1.7	0.5	1.8		
Sample 1	S11	97.3	99.0	92.8	96.4	3.2	100.3	91.5	87.5	93.1	6.5	95.7
2 row caulking	S12	95.4	90.8	92.2	92.8	2.4	99.7	101.3	94.0	98.3	3.8	94.0
	S13	97.3	94.3	97.2	96.3	1.7	105.6	93.5	90.9	96.7	7.8	98.7
	S14	96.1	94.2	83.9	91.4	6.6	97.2	99.8	97.4	98.1	1.4	93.3
	Mean	96.5	94.6	91.5	94.2	2.5	100.7	96.5	92.5	96.6	4.1	95.4
	SD	0.9	3.4	5.6	2.5		3.5	4.8	4.2	2.4		2.4
Sample 2	S21	95.0	94.0	90.8	93.3	2.2	96.6	95.4	93.3	95.1	1.7	95.3
1 row caulking	S22	94.6	93.3	91.8	93.2	1.4	97.4	93.2	91.8	94.1	2.9	96.1
	S23	98.8	98.3	94.1	97.1	2.6	101.7	98.3	97.4	99.1	2.3	99.7
	S24	98.8	96.8	95.6	97.1	1.6	101.2	104.5	100.3	102.0	2.2	96.3
	Mean	96.8	95.6	93.1	95.2	1.9	99.2	97.9	95.7	97.6	1.8	96.9
	SD	2.3	2.4	2.2	2.2		2.6	4.9	3.9	3.7		1.9
Sample 3	S31	95.3	84.5	90.9	90.2	5.4	93.1	90.2	88.1	90.5	2.5	91.3
no caulking	S32	94.0	91.9	94.0	93.3	1.2	104.8	96.3	94.8	98.6	5.4	95.8
	S33	96.3	86.4	91.7	91.5	5.0	91.2	87.7	94.9	91.3	3.6	102.3
	S34	95.8	83.9	87.5	89.1	6.1	96.3	95.2	94.5	95.3	0.9	95.3
	Mean	95.4	86.7	91.0	91.0	4.3	96.4	92.4	93.1	93.9	2.1	96.2
	SD	1.0	3.6	2.7	1.8		6.0	4.1	3.3	3.8		4.6

*The wall's seam covered with the additional protective coating and a purging time of 60 min was used.

RPC had a smaller volume than the test bin, therefore, was exposed to changes in ambient temperature to a greater extent than the test bin. This was probably a reason for the sharp increases in the differential pressure during the decay tests.

Although sudden variations in barometric pressure for the duration of the tests were not observed, the continuous decay or rise of the barometric pressure could contribute to the pattern of the differential pressure decay.

Carbon dioxide retention after 24 h

To estimate and compare the efficiency of CO_2 retention, the coefficient of retention (η_r), i.e. the ratio of the CO_2 concentration after 24 h to the initial concentration, was calculated as:

$$\eta_r = \frac{C_{24h}}{C_{0h}} * 100 \qquad (1)$$

where:

η_r = amount of CO_2 remaining in the bin after 24 h as a percentage of initial concentration (%),

C_{24h} = CO_2 concentration after 24 h (%), and

C_{0h} = initial CO_2 concentration (%).

For bins filled with wheat, coefficients of retention, η_r, varied from 96 to 101% (mean 98%) for no seam (control sample 0), 84 to 97% (mean 94%) for 2 row caulking (sample 1), 92 to 99% (mean 95%) for 1 row caulking (sample 2), and 84 to 96% (mean 91%) for horizontal seam with no caulking (sample 3) (Table III).

For the empty bin, η_r was greater than for the bin filled with wheat. The η_r ranged from 98 to 106% (mean 100%) for no seam (control sample), from 88 to 106% (mean 97%) for 2 row caulking (sample 1), from 93 to 105% (mean 98%) for 1 row caulking (sample 2), and from 88 to 105% (mean 94%) for horizontal seam with no caulking (sample 3) (Table III). The values of η_r greater than 100% probably are caused by the measurement errors in the gas concentrations using the gas chromatograph and nonuniform distributions of CO_2 in the bin at all times.

The means of η_r for sample 2 (1 row caulking) were only slightly higher than for sample 1 (2 row caulking). This confirmed the pressure decay test results in which one and two rows of caulking performed similarly. Retention coefficients decreased with increasing initial CO_2 concentrations, i.e. increasing purge times from 15 to 60 min.

Table IV. Analysis of variance of the coefficients of CO_2 retention (%) after 24 h as influenced by the method of sealing, initial CO_2 concentration, and the presence of grain in the bin.

Source of variation ψ	Degrees of freedom (DF)	Sum of squares (SS)	Mean square (MS)	F value	Pr>F
S_i	3	535.45	178.48	16.35	0.0001% **
C_j	2	280.55	140.27	12.85	0.0001% **
G_k	1	134.19	134.19	12.29	0.0008% **
S_i x C_j	6	130.87	21.81	2.00	0.077% ns
S_i x G_k	3	3.95	1.32	0.12	0.95% ns
C_j x G_k	2	6.22	3.11	0.28	0.75% ns
S_i x C_j x G_k	6	29.85	4.98	0.46	0.84% ns
Error	72	786.02	10.92		
Total	95	1907.10			
Contrast	DF	Contrast SS	Contrast MS	F value	Pr>F
S0 vs Others	1	337.78	337.78	30.94	0.0001% **
S1 vs S2	1	11.80	11.80	1.08	0.30% ns
S2 vs S3	1	182.91	182.91	16.75	0.0001% **
C15 vs C30	1	138.06	138.06	12.65	0.0007% **
C30 vs C60	1	19.91	19.91	1.82	0.18% ns
C15 vs C60	1	262.84	262.84	24.08	0.0001% **

** = highly significant effect at 1% significance level.
ns = not significant effect.
ψ S_i = the effect of the ith method of sealing; C_j=the effect of the jth initial CO_2 concentration; G_k= the effect of the kth level of the grain presence in the bin.

An analysis of variance was performed on the CO_2 retention coefficients (Table III) using the General Linear Model (GLM) Procedure (SAS 1986) (Table IV). The variations in the results were reasonably taken into account by the model as indicated by the coefficient of variation (CV) which had a very small value of 3.45, although r^2 was only 0.59.

The main effects of sealing method, initial CO_2 concentration, and grain in the bin were highly significant at the 1% significance level. The interactions between the factors were not significant at the 1% significance level.

Highly significant differences occurred between the control sample with no seam and the remaining samples, as well as between no caulking and 2 and 1 row caulking. Although differences were small due to the small specimens used, the differences in full size bins could be considerable. The difference between sealing with 1 and 2 row caulking was not significant. There were significant differences between initial CO_2 concentrations created in 15 and 30 min, and between 15 and 60 min, but not between 30 and 60 min.

Carbon dioxide retention after 120 h (5 d)

After 120 h, the retention coefficients with the wheat-filled bin were 84% for 2 row caulking, 73% for 1 row caulking, and 66% for the horizontal seam with no caulking. The coefficients of retention after 24 h (94, 95, and 91%, respectively) were closer to each other. The effect of sealing method was more evident after 120 h than after only 24 h. Sealing with two rows of caulking was better in retaining CO_2 than one row of caulking for vertical seam, and much better than when no caulking was applied to the horizontal seam.

Effect of an additional protective coating

For the CO_2 purging time of 60 min in the empty bins, the pressure decay (Table I) and the CO_2 concentration decay (Table III) were measured when the seams were covered with an additional protective coating (PC), i.e., covered with a flexible membrane resin. The pressure decay tests (Table I) revealed that effectiveness of the additional coating was not significant for samples which were previously sealed with double or single row caulking (samples 1 and 2), but was significant for the uncaulked horizontal seam (sample 3). The coefficients of retention of CO_2 for all samples increased by 1-3% when an additional coating was used (Table III). It was expected that performance of the additional protective coating would be better. A proper coating of the flexible membrane resin was difficult to obtain on the vertical seams because of the resin's high liquidity.

CONCLUSIONS

The following conclusions can be drawn from this study:

1. Both the pressure decay and the CO_2 concentration decay tests are useful for estimating the degree of sealing of bolted metal bins.
2. Sealing vertical seams of bin walls with single or double row butyl caulking were not significantly different, but both of them were significantly different from uncaulked horizontal seams based on 24-h tests. However, based on 120-h tests, double row caulking was better than single row caulking which was better than uncaulked horizontal seams.
3. The CO_2 retention coefficient decreased with increasing initial concentrations.
4. The presence of grain in the bin affected both pressure decay times as well as CO_2 retention coefficients. Pressure decay times were shorter in the bin filled with grain than in the empty bin. Also, CO_2 retention coefficients were lower when the bin was filled with grain than when the bin was empty.
5. Covering the seams with a flexible membrane resin was not significant when caulking was previously applied but was effective on the uncaulked horizontal seams.

ACKNOWLEDGEMENTS

We thank the Natural Sciences and Engineering Research Council of Canada for funding this study and Messrs. Jack Putnam, Matt McDonald, and Dale Bourns for their technical assistance.

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