SEALING EFFICIENCY ASSESSMENT IN MODIFIED ATMOSPHERE STORAGES

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ABSTRACT

The leak area that would permit air infiltration into an experimental 665.7-L silo was assessed with constant pressure tests using 23–1,006-mm² orifice cross-sections. The influence of both the area of the orifice cross-section and its depth (wall thickness) on variations of the empirical constants that describe the constant pressure test was also demonstrated. Based on a series of tests, an empirical equation to estimate leak area was proposed.

The experimental silo was filled to 92% of its capacity with ca. $500 \, kg$ wheat and tested, using variously sized orifices, for carbon dioxide (CO₂) loss. The measured CO₂ concentrations were compared with the calculated values based on equations that took into consideration initial CO₂ adsorption by the wheat, diffusion of CO₂ through the leak, and variations in temperature and barometric pressure. Under the experimental conditions, close agreement between the measured and calculated values was obtained. The influence of temperature and barometric pressure variation on infiltration of air into the silo was also analyzed. The information obtained from the constant pressure tests and from analysis of the weather conditions provided guidelines concerning how long a certain concentration could be maintained in a CO₂-treated structure. Leak rates for any structure may be assessed by the method developed in these experiments.

INTRODUCTION

A well-sealed storage structure is fundamental for the application of modified atmospheres (MA's). A MA created by the introduction of carbon dioxide (CO₂) can be maintained in such a structure at an efficient level because air ingress is restricted; otherwise it would eventually lead to dilution of the CO_2 concentration. To control stored-product insects, Jay (1971) recommended a concentration of 60% CO_2 in air for 4 d at a temperature of $27^{\circ}C$ or above. Gastightness has been a problem for years in the application of fumigants in storage. The consequences of poorly sealed storages under fumigation have been discussed by Banks and Desmarchelier (1979) and Banks (1981), though the requirement that storages be gastight appears to be more critical in using MA's than in applying fumigants (Monro, 1969).

The degree of gastightness that is satisfactory, as well as what gas concentrations can be maintained under given environmental and structural conditions (Hunt, 1980), should be decided prior to the application of a MA. These decisions should be weighed against the investment involved in sealing a leaky structure to prevent excessive loss of the MA.

The present work assesses gas-leak rates from grain bulks treated with CO₂ at different temperatures and atmospheric pressures.

MATERIALS AND METHODS

Equipment

Pressure measurements were carried out using a Pace Wianko pressure transducer kit with a model CD25 indicator and a model KP15 transducer having interchangeable diaphragms. The transducer source was calibrated against a Dwyer model 40023 manometer.

Temperatures were determined with thermistor probes connected to a Yellow Springs Instrument Co. Model 47.1 scanning tele-thermometer. Two Yellow Springs model 80A continuous recorders were connected to the equipment, one to the pressure indicator and the other to the scanning tele-thermometer. These readings were recorded on a 25.4 cm scale, using a Hewlett-Packard model 7128A recorder with a 25.4 cm/h scale.

Barometric pressure changes were recorded on a Belfort Instrument Co. No. 5-800A microbarograph with a weekly chart record. This microbarograph was calibrated and periodically checked against a Fortin-type Fisher Scientific Co. Model 02-380 mercury barometer.

Air flow was measured by using either Gilmont K-3200-00 float-type variable-area flowmeters having several ranges or a Sierra Instruments, Inc. model 715 mass flowmeter. Flowmeters were calibrated against a bubble-type flowmeter. The mass flowmeter was used for air volumes up to 50 L/min, while higher air volumes were measured with an Alnor Thermoanemometer Model 8500.

Atmospheric gas composition was analysed with a Fisher-Hamilton Model 29 dual column gas-chromatograph equipped with a thermal conductivity detector. Peak areas were measured with a Hewlett-Packard integrator model 3390A.

A 665.7-L silo, 259.5 cm high with an internal diameter of 57.15 cm, was used for the experiments (Fig. 1). The silo was constructed by welding together three commercially available metal drums, the wall thickness of which was 0.825 mm.

Variously sized orifices, simulating leaks in the experiments, were prepared by using hypodermic needles having measured diameters for orifices less than 3 mm wide or by drilling holes on removable flanges mounted on the experimental silo.

Constant pressure test for determination of leak area

In two series of tests both small and large leaks were studied. In the series with small leaks, needles with openings of 0.57, 1.14 and 1.42 mm² in area, and orifice lengths (representing simulated wall thickness) of 5, 20 and 40 mm, were employed. To minimise

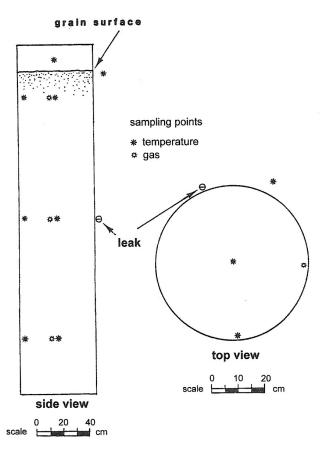


Fig. 1. Experimental silo designed for assessment of gastightness in modified atmosphere studies.

the influence of variations (in both the ambient temperature and barometric pressure) that would affect the results obtained when the small leaks were located on the silo wall, it was necessary to reduce the volume of the silo. Therefore a parallel series of experiments was carried out with needles mounted on a 0.95-L glass container.

Large leaks were tested on a 3.15-mm thick removable sliding side plate. Orifices were 23 to 1,006 mm². During a test, air from a pressurized cylinder was introduced into the silo and the ratio of volumetric flow to final equilibrium pressure recorded. The general equation (Anon. 1972; Hill and Kusuda, 1975):

$$Q = KA \cdot \Delta P^n \tag{1}$$

was used to obtain the value for the proportionality constant (KA). Since the airflow into the silo at equilibrium equals the total air leakage out of the silo, the pressure difference (ΔP) could be correlated to K (the empirical constant) and A (the effective leak area). Sharp (1982) and Dickson (1981) found values of n close to 0.5 for sharp-edged orifices and 1 for cracks.

CO₂ decay tests

CO₂ decay tests were carried out with the silo containing 499.4 kg of soft red winter wheat at 12.5% moisture content (m.c.). The wheat bulk occupied 92% of the silo volume.

 ${
m CO_2}$ gas was supplied from pressurized cylinders. After a certain ${
m CO_2}$ concentration was reached, 2–3 d were allowed to elapse to enable the gas to attain a uniform concentration. Gas samples were taken periodically using a 10-ml Hamilton model 1010 gastight syringe fitted with a 5-cm size hole model 6-90224 needle. Gas sampling ports were located at 42, 131 and 218 cm from the bottom of the silo (Fig. 1). ${
m CO_2}$ concentrations were analyzed by gas chromatography.

The silo was equipped with eight temperature measuring points. Gas pressure within the silo was measured from the center of the silo through a 5.7-mm diameter tygon tube connected to the transducer. All tests were carried out in a room maintained at 25 ± 1 °C.

Calculation of CO₂ loss

The calculation of CO₂ loss was based several assumptions. The effect of wind was assumed to be negligible since the leak site was protected from any direct air circulation which could have been caused by the air-conditioning system in the room where the silos were held. The CO₂ produced by grain respiration was also assumed to be negligible and the grain was assumed to be free from insect infestation. The volume occupied by the grain, with a calculated void space of 288.7 L (including the head space) was assumed to remain constant. The corrected diffusion rate through the leak was assumed to be 16.1 mm²/sec at 25°C and 101.325 kPa (Roberts, 1963), and the concentration of CO₂ in the silo was assumed to be uniform, while the CO₂ concentration outside the silo was assumed to equal the atmospheric CO₂ concentration. Finally, the representative silo temperature was measured in the center of the empty silo and in the silo containing wheat, and the external temperature was assumed to affect only the headspace volume.

Variations in temperature and barometric pressure and diffusion through the leak all caused air to infiltrate into the void space in the silo. The air infiltrating into the silo caused a change in CO_2 concentration due to a uniform diluting effect. The CO_2 concentration at time t (C_t) was uniform throughout the silo. Appropriate equations were developed to determine the volume of air entering the silo and thus causing changes in the CO_2 concentration.

Based on these assumptions, the change in concentration (dC) is equal to the concentration (C) times the fraction of the volume withdrawn dV (Nelson, 1971):

$$dC = -\frac{C \, dV}{V} \tag{2}$$

This integrates to give a resultant concentration C_i :

$$C_t = C_0 \exp\left(-V_a / V_e\right) \tag{3}$$

where V_a is the volume of air infiltrating the silo and V_e the void volume of the silo and C_0 is the initial concentration.

The volume of air/CO₂ mixture diffusing out of the silo is equal to the volume of air entering the silo from the diffusion rate equation:

$$V_{\rm d} = \frac{AD \cdot \Delta C \cdot 3.6t}{L10^8} \tag{4}$$

where V_d is the amount of CO_2 that diffused through the leak in m^3 ; L is length of the leak in mm; A is the cross-sectional area of the leak in mm^2 ; t is the time during which a given amount of CO_2 diffused in h; ΔC is the CO_2 concentration difference between the silo atmosphere and ambient atmosphere in %; and D is the diffusion coefficient in mm^2/s .

Fluctuations in gas temperature result from solar radiation, ambient air temperature, wind and precipitation, all of which depend on the thermal properties of the silo's construction material. The total number of temperature changes is directly proportional to the frequency and amplitude of the temperature fluctuations in the silo's void space. Therefore, changes in the silo gas temperature were approximated by the linear functions used by Meiring (1982):

$$T_{t} = T_{0} + R_{T} \cdot t \tag{5}$$

where T_t is the silo gas temperature in °K at time t; T_0 is the initial silo gas temperature in °K; R_T is the temperature rate of change in °C/h and t is the time in h.

Since air infiltrates into the silo void space as a result of a decrease in gas temperature, the ratio of silo gas temperature to the initial silo gas temperature can be used as a measure of the influence of temperature on the volume of air which infiltrates the silo. In practice, the drop in nighttime temperature affects both the headspace temperature of the silo and that of the wheat to a depth of approximately 0.15 m from the outside wall (Oxley, 1948; Muir, *et al.*, 1980). If the void space portion of the silo under the influence of ambient temperature is $V_{\rm h}$, the volume of air entering the silo due to temperature change is:

$$V_{\rm t} = \frac{V_{\rm h} \cdot R_{\rm T} \cdot t}{T_0} \tag{6}$$

Similarly, an increase in barometric pressure causes air to infiltrate the silo. Changes in barometric pressure may be approximated by the linear function:

$$P_{t} = P_{0} + R_{P} \cdot t \tag{7}$$

where $P_{\rm t}$ is the silo gas pressure in Pa at time t; $P_{\rm 0}$ is the initial atmospheric pressure; and $R_{\rm P}$ is the rate of atmospheric pressure change in Pa/h. Since the atmospheric pressure would affect the effective total void volume ($V_{\rm e}$) of the silo, the volume of air entering the silo due to atmospheric pressure change is:

$$V_{\rm p} = \frac{V_{\rm e} R_{\rm P} \cdot t}{P_{\rm o}} \tag{8}$$

The basic assumption in calculating V_t and V_p is that the volume infiltrating the silo is not restricted by leaks. Obviously in a gastight structure not equipped with a pressure relief valve air infiltration into the silo during sudden changes in temperature or barometric pressure will be independent of the leak characteristic. When this happens, the pressure differential required to cause air infiltration is approximately 50% of the pressure differential value. The time required for this process is important. For a given time t_p :

$$\Delta P = \frac{V_{\rm t} + V_{\rm p}}{2t_{\rm p}V_{\rm e}} \tag{9}$$

by substituting (9) into (1)

$$Q = KA \left[\frac{V_{\rm t} + V_{\rm p}}{2t_{\rm p}V_{\rm e}} \right]^n \tag{10}$$

so that

$$\log Q = \log KA + n \log \left[\frac{V_{\rm t} + V_{\rm p}}{2t_{\rm p}V_{\rm e}} \right]$$
(11)

If A and t_p are sufficiently small values, then Q will be:

$$Q < \frac{V_{\rm t} + V_{\rm p}}{t_{\rm p}} \tag{12}$$

A quick volume change would result in a pressure change in the system and the value Q should be combined into (3) as:

$$C_{t} = C_{0} \exp \left(-\left(V_{d} + Q\right) / V_{e}\right) \tag{13}$$

But if A and t_p are large values, then Q is:

$$Q > \frac{V_{\rm t} + V_{\rm p}}{t_{\rm p}} \tag{14}$$

and equation (3) can be transformed to:

$$C_{\rm t} = C_0 \exp\left(-V_{\rm a}/V_{\rm e}\right) \tag{15}$$

where $V_a = V_d + V_t + V_p$.

The validity of the above assumptions was investigated using the 665.7-L experimental silo containing wheat. When a silo is purged with CO_2 , the initial change in concentration due to sorption also needs to be considered. If the sorption rate is known, the expected drop in CO_2 concentration can be estimated. The volume of CO_2 sorbed by wheat will

cause negative pressure in the system, and air infiltrating the silo will further lower the CO_2 concentration. This will cause a different rate of sorption than that obtained at a constant CO_2 concentration. Since the proportion of grain volume to the total capacity of the bin may vary, the resulting sorption rate would also vary in relation to the changing CO_2 concentration.

RESULTS AND DISCUSSION

Constant pressure tests

Volumetric flow in small leaks or cracks is strongly dependent on orifice length (L). In a grain bin or silo, this orifice length may constitute the thickness of the wall. This relationship was measured for a 1.42-mm² cross-section with various orifice lengths. The longer the orifice was, the higher the value of the empirical parameter n was. Kreith and Eisenstadt (1957) found that the expression of length-to-diameter (L/D) was a function of n when determining flow characteristics of short capillary tubes. Using a similar approach, the results obtained for different n values were plotted against orifice length/diameter (L/D) ratios for leaks of various lengths (Fig. 2).

This relation would clearly indicate that where cracks exist in the structure, the flow characteristics necessary to determine KA (Equation 1) will be strongly dependent on the orifice length which affects the pressure-drop expressed by the exponent n.

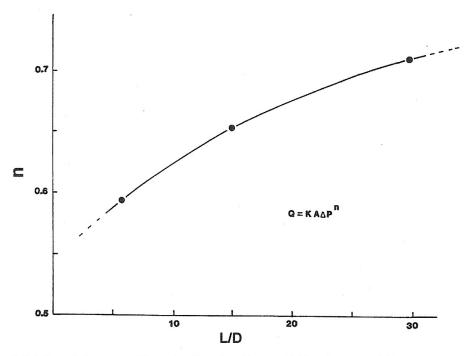


Fig. 2. Relationship between orifice ratio of length to diameter (L/D) and pressure difference exponent (n).

These results indicated that unless the orifice length is maintained constant, KA can not be generalized in relation to exponent n. Additional research is needed to quantify the relationship of these constants.

Various orifice sizes 3.15 mm long were studied to determine the relationship of KA and n. From a practical view, determination of the leak area of a given silo where exponent n = 0.55105 is applicable, would require only a single constant pressure test. The empirical values obtained for different areas in cross section were tested for values up to 60 Pa (Fig. 3). Since the current values were studied on round orifices having a fixed length, more experimental values are needed to determine the relation of KA and n for different leak configurations.

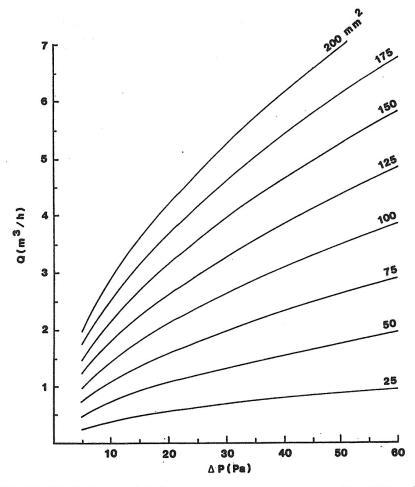


Fig. 3. Calculated family of curves for leak cross-section areas varying between 25 and 200 mm², based on constant pressure tests using air at 25°C, where the empirical parameters were $K = 4.0516 \times 10^{-3}$ and n = 0.55105.

Influence of temperature

Although the experimental silos were maintained in a controlled temperature room with a thermostat setting of 25°C, the changes in temperature outside the room influenced the frequency of thermostat activation due to heat loss from the room to the outside air. This loss caused the thermostat to activate the heating unit for intermittent periods, especially when the ambient temperature outside the room was in the 0–10°C range. The periodic operation of the heater to maintain the room at 25°C was accompanied by an increase in pressure in the silo, and these periodic changes in internal silo pressure caused a pumping effect which resulted in either blowing out the CO₂-air mixture or sucking ambient air into the silo.

Temperature changes which affected the silo's internal pressure could best be detected when the silo had a leak with a cross-sectional area of less than 1.478 mm². Typical silo pressure changes recorded during the observations with leak cross-section areas of 0.586 and 0.068 mm² were recorded (Fig. 4). The smaller the leak was, the greater the influence of temperature on the build-up of internal pressure was also. This may be especially important in horizontal silos having a large head space volume. To prevent damage to tightly sealed silos during critical pressure increases, use of a pressure relief valve is recommended by Banks and Annis (1977).

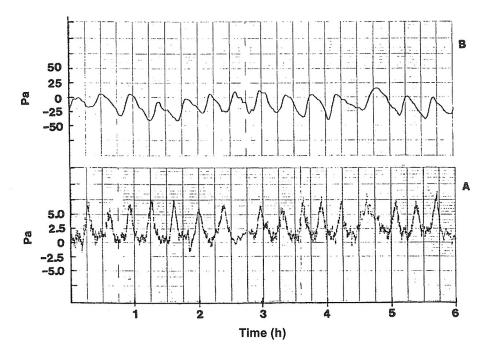


Fig. 4. Typical silo pressure changes recorded under the influence of temperature fluctuations in the experimental silo containing ca. 500 kg of wheat and with 0.586 mm^2 (A), and 0.068 mm^2 (B) leak cross-section areas.

The typical ambient temperature changes recorded in these observations do not represent field conditions where temperature changes may be much greater and last for extended periods of time. However, R_T values (Equation 5) calculated in these experiments ranged from +0.13°C/h to +3.56°C/h. These values may be compared with temperature changes under maritime weather conditions, which may vary between +3°C/h and +10°C/h (Meiering and Wenner, 1970).

Meiering (1982), working with gastight silos, found that when temperature rises 3°C/h, there is sufficient time for gas flow to create pressure equilibrium with the atmosphere at a specific permeability of 0.2 mm²/m³ (ratio of leak cross-section area to void volume of silo). Our experiments were carried out with specific permeabilities of 0.23, 2.03, 5.12 and 76.67 mm²/m³ and, accordingly, sufficient time existed for gas flow to create pressure equilibrium with the atmosphere.

Influence of barometric pressure

Changes in barometric pressure are important in infiltration of air into silos which are under MA treatment. The dominant cause of air infiltration was the change in barometric pressure when control of room temperature was discontinued. Variations in barometric pressure ranged between 11 and 17 Pa/h in the test room. Under maritime weather conditions, Meiering and Wenner (1970) observed typical atmospheric pressure changes of +25 Pa/h. Their data and results in Fig. 5 demonstrate the importance of local weather observations, when a MA is to be applied, in order to enable the CO₂ loss from the storage structure to be assessed.

Air infiltration into the experimental silo

To control stored-product insects Jay (1971) recommended a MA mixture of 60% CO_2 in air for 4 d at a temperature at or above 27°C. An initial concentration of 60% CO_2 will gradually decrease due to sorption of the gas by the grain (Banks *et al.*, 1980). In our experiments the CO_2 purge was carried out from a single port at the bottom of the silo; this created an initial CO_2 gradient between the different sampling ports. This gradient progressively changed (due to diffusion) until a uniform CO_2 concentration was obtained. The initial sorption of CO_2 by the grain has a significant effect on reducing the initial CO_2 concentration attained. Therefore, in the application of CO_2 to maintain a concentration effective to control insects, addition of CO_2 in the maintenance phase should be balanced against the expected mass of CO_2 to be adsorbed by the commodity.

Four experiments with leak areas of 0.068, 0.586, 1.478 and 22.134 mm² were carried out while temperature, barometric pressure and gas composition of the experimental silo were monitored. Results of CO₂ loss and calculated CO₂ concentrations based on Equation 15 are shown in Fig. 6.

Characteristic changes in barometric pressure and ambient temperature were considered in calculating CO₂ loss, and they were plotted against each period of time (20 h, 10 h or less).

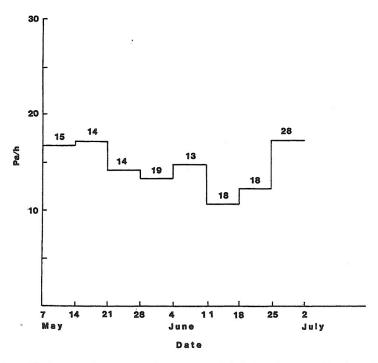


Fig. 5. Typical weekly barometric pressure changes recorded during the tests. Number of changes in barometric pressure are given in figures shown for each weekly period.

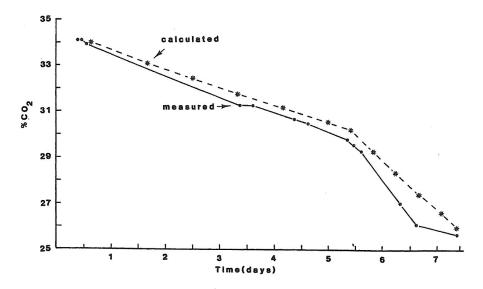


Fig. 6. Measured and calculated CO_2 concentrations obtained in the experimental silo containing ca. 500 kg of wheat and equipped with a leak cross-section area of 22.134 mm².

Other parameters affecting CO₂ loss, such as wind and chimney effects, were not considered in these studies. Under extreme weather conditions, the effects of temperature gradients and gas density differences would clearly have additional effects on the CO₂ loss from treated structures (Banks *et al.*, 1975). These weather parameters would require complicated computations using simulation models. Empirical models to measure air infiltration into structures have been proposed by Sherman *et al.* (1980), but application of this information to grain storage facilities requires additional research. If the leak-rate estimates given in the present study indicate that a silo is unsatisfactory for the application of CO₂ (or other MA's), efforts should be made to render the storage structure more gastight. The expected CO₂ loss from a silo containing grain can be assessed by estimating the total leak area and gathering information on expected changes in local temperature and atmospheric pressure. This assessment is essential for evaluating the cost of maintaining an adequate CO₂ concentration during the purge and maintenance phases of treatment. It is also very important in enabling comparison of added treatment costs with the capital investment necessary to render the storage structure gastight.

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LIST OF SYMBOLS USED

 $A = \text{orifice cross-section area, mm}^2$.

 $D = \text{diffusion coefficient, mm}^2/\text{s.}$

L = initial length, mm.

 C_0 = initial CO₂ concentration, %.

 $C_t = CO_2$ concentration at time t.

 $\Delta C = CO_2$ concentration difference between the silo and ambient.

M = mass of grain, kg.

t = time, h.

 t_p = time to cause pressure differential, h.

 P_a = atmospheric pressure, Pa (Pascals).

 P_t = silo gas pressure, Pa (Pascals), at time t.

 P_0 = initial atmospheric pressure, Pa.

 ΔP = pressure difference, Pa.

 $R_{\rm P}$ = change of atmospheric pressure, Pa/h.

 $Q = \text{volumetric flow rate, m}^3/\text{sec or m}^3/\text{h}.$

- $T_{\rm t}$ = silo gas temperature, °K, at time t.
- T_0 = initial silo gas temperature, °K.
- $R_{\rm T}$ = change of temperature, °C/h.
- $V_{\rm e}$ = silo gas volume, m³.
- $V_{\rm h}$ = silo headspace gas volume, m³.
- V_a = volume of air infiltrating the silo, m³.
- $V_{\rm d}$ = volume of air infiltrating the silo due to diffusion through the orifice, m³.
- V_t = volume of air infiltrating the silo, due to temperature change, m³.
- V_p = volume of air-infiltrating the silo, due to atmospheric pressure change, m³.
- n = characteristic pressure difference exponent, empirical parameter varying between 0.5 and 1.0.
- K = empirical parameter, multiplier of the orifice cross-section area (A) in constant pressure test.

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