Journal of Stored Products Research 69 (2016) 143–151

Contents lists available at ScienceDirect

Journal of Stored Products Research

journal homepage: www.elsevier.com/locate/jspr

Pest control treatments with phosphine and controlled atmospheres in silo bags with different airtightness conditions

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ARTICLE INFO

Article history:
Received 12 December 2015
Received in revised form
14 June 2016
Accepted 27 July 2016

Keywords:
Grains
Oilseeds
Insects
Hermetic storage
Pressure decay test
Carbon dioxide

ABSTRACT

The silo bag technology has been extensively used in Argentina for storing grains (e.g. wheat, corn, barley, sunflower and soybean among others) since the mid-1990s. Silo bag are widely considered a hermetic storage system in which PH_3 fumigation is frequently implemented for pest control. However, there is insufficient information regarding the potential airtightness of silo bags and how it could affect the performance of fumigation and controlled atmosphere treatments. In this study, a pressure decay test (PDT) was implemented to characterize airtightness level of silo bags set up following various procedures. PH_3 fumigation treatments with different dosages and hermeticity levels were conducted, and fumigant concentration was monitored. Controlled atmosphere treatments with carbon dioxide were also implemented in silo bags with different hermeticity levels. Results showed that less than half of the bags tested in the field had a PDT indicated for fumigation (90 s), and that when a bag without thermo sealing was used for fumigation, this treatment failed. However, it was demonstrated that with simple and inexpensive practices silo bags can achieve high enough airtightness conditions to implement successful PH_3 fumigation (5 days above 200 ppm with a dosage of 1 g of PH_3/m^3) and even controlled atmosphere treatments (more than 18 days with CO_2 concentration above 70%). This study shows that silo bags could be used as a cost competitive hermetic storage technology for performing controlled atmosphere treatments.

1. Introduction

The silo bag technology has been extensively implemented in Argentina for storing grains (e.g. wheat, corn, barley, sunflower and soybean among others) since the mid-1990s (Bartosik, 2012). During the last 5 years a total of 40–45 million tons of different grains have been stored every year in this flexible hermetic system. Today, the silo bag technology is being implemented in more than 50 countries improving postharvest logistics and reducing grain postharvest losses.

Silo bags are considered hermetic storage systems. However, when dry grain is stored in silo bags the resulting biological activity is low, and hence, the degree of modification of the internal atmosphere is not sufficient to generate a lethal environment for insects (CO_2 concentration less than 10% and O_2 concentration above 10%) (Abalone et al., 2011; Arias Barreto et al., 2013; Bartosik et al., 2008; Cardoso et al., 2008; Rodríguez et al., 2008).

Nevertheless, insect development could be affected in this semi-restricted environment (Subramanyam et al., 2012). In many countries there is a legal restriction for trading grains with live insects (i.e. nil tolerance), implying that a pest control treatment has to be implemented in most of the grain at some point of the commercialization chain. When silo bags are used for storing grains, the preferred pest control treatment is phosphine (PH_3) due to the potential airtightness of the bag. For the same reason, controlled atmospheres (CA) treatment could be a suitable control method.

In Argentina, the fumigation of silo bags with PH_3 could be done right after the harvest when the bag is loaded, or after 3–8 months of storage in the field (in this case the hermeticity of the bag could be compromised (Darby and Caddick, 2007). The tablets of aluminum phosphide are inserted every 5–10 m along one side of the bag, with a dosage from 1 to 2 g of PH_3 per t, regardless the type

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http://dx.doi.org/10.1016/j.jspr.2016.07.007
0022-474X/

of grain (equivalent to 0.75–1.5 g/m^3 for wheat) and the hermeticity level of the bag. No particular care is taken regarding the sealing of the end of the bag, and most of the time the bag is closed by folding the plastic cover on itself and adding some weight on top. Hermeticity of silo bags is not tested and it is difficult to predict beforehand. As a result, fumigant leakage is not considered for prescribing the initial dosage. PH_3 concentration is not measured during treatment and fumigators just assume that the procedure accomplishes the Argentine standard for effective PH_3 fumigation, which is 200 ppm for 5 days, although sometimes it might not be accomplished.

On the other hand there is an increasing demand for grains free of pesticides residues. The silo bag technology has the potential to be used for CA treatments by injecting CO_2 or N_2 and maintaining a critical concentration during a certain period of time.

Research has been done regarding the effect of hermetic storage in silo bag on grain quality, mold and micotoxin development, early detection of spoilage, economics and logistics, and modeling of the internal environment, which were summarized by Bartosik (2012). In addition, research has been done on control of pests with PH_3 in silo bags (Cardoso et al., 2009; Darby and Caddick, 2007; Ridley et al., 2011), but the documented experiences did not explore in detail the relationship between a successful PH_3 treatment and the airtightness level of the silo bag. Additionally, the airtightness level required for CA treatment is higher than that required for PH_3 fumigation (Navarro, 1998), and there is insufficient information on whether the silo bags can achieve enough hermeticity to implement a successful CA treatment (Darby and Caddick (2007) performed PDT in few silo bags in the field with, in general, unsatisfactory results). One of the main limitations for implementing CA treatments at a large scale is the cost of airtight bins. If the silo bag can be airtight enough for CA treatment, they could be successfully used as an inexpensive technology for CA treatments.

The objectives of this study were 1) to characterize the airtightness of silo bags placed in the field; 2) to evaluate the efficacy of a PH_3 fumigation treatment in silo bags with different dosages and hermeticity levels; and 3) to evaluate the feasibility of implementing a successful controlled atmosphere treatment in silo bags.

2. Materials and methods

2.1. Silo bags hermeticity assessment

A pressure decay test (PDT) adapted from Darby and Caddick (2007) was carried out in 21 silo bags placed in different farms and grain storage facilities in Southeast of Buenos Aires province, Argentina. Silo bags are made of three layers liner (230 μm thickness with UV protection) with a diameter of 2.74 m and from 20 to 60 m long, holding approximately from 70 to 200 t of wheat, corn and soybean, and up to 120 t of sunflower. The PDT consists of generating a negative pressure inside the bag (i.e. −1200 Pa) with a fan, and measuring the time it takes to drop to half of the initial negative pressure (i.e. −600 Pa). Approximately at the center of the silo bag, a pipe of 40 mm diameter was inserted 0.8 m inside the grain mass. The edge of the plastic cover, in the perforation made to insert the pipe, was sealed to the pipe with silicon sealant and tapes, so hermeticity was not affected by the inserted pipe. The outside end of the pipe had a valve that can hermetically close the connection between the inside atmosphere of the bag and the outside. The valve was connected to a centrifugal fan (Chicago Blower, 0.33 HP, USA) through a flexible plastic hose of 40 mm diameter. The pressure was monitored with a digital pressure gauge (Sper Scientific, China) connected to the bag by a plastic tube of 5 mm diameter, and the time from −1200 Pa to −600 Pa was recorded with a chronometer (Fig. 1).

2.2. PH_3 fumigation treatments

Five standard silo bags of 2.7 m diameter and from 20 to 60 m long holding wheat (T1, T2 and T5) and sunflower (T3 and T4) were used to perform the fumigation trials. The wheat silo bags were placed in farms close to Balcarce, Buenos Aires province, Argentina, and the sunflower silo bags were placed at the Cargill oil crushing facility near Quequen port, Buenos Aires province, Argentina.

The silo bags corresponding to T1 to T4 were made without any particular care to preserve the hermeticity of the system, representing the most common conditions in which fumigation is performed in silo bags. It means that no soil preparation was done to limit the risk of perforation by stones or other hard objects, and the end of the bag was folded over and some weight was added on top. On the other hand, the bag used for T5 was thermo sealed at the ends directly in the field with a portable sealing equipment (La Pipiola, Argentina), enhancing the potential hermeticity of the bag. Thermo sealing is a simple and affordable practice to be implemented by farmers and local elevators.

Grain samples were collected before the fumigation trials from 5 locations along the bag with a standard probe and moisture content (m.c.) was measured with a moisture meter (Dickey John, Gac 2100, USA). The wheat silo bags (T1, T2 and T5) were filled with recently harvested grain, with m.c. between 12.8 and 13.4%. The sunflower silo bags (T3 and T4) were filled with recently harvested oilseeds with m.c. around 11%. Grain temperature was measured before and after the fumigation trials by inserting in the bag a temperature probe in 5 locations (Cereal Tools, Temperature Probe, Argentina). In all treatments the temperature was between 20 and 25 °C.

The fumigation product was aluminum phosphide tablets of 3 g (Phostoxin, Degesch, Germany) that produces 1 g of PH_3. The dosage was from 0.75 to 1.72 g PH_3/m^3 (Table 1), and the expected maximum theoretical concentration (MTC) ranged from 1100 to 2500 ppm, approximately (see Section 2.2.1 for MTC computation).

The aluminum phosphide tablets were inserted in the grain mass every 5 m along the bag, thus the PH_3 gas should move in the horizontal direction 2.5 m to each side of the application point at the most. For instance, for T1 the total length of the bag was 60 m, so 12 application points were considered in the total length of the bag. The first application point was located at 2.5 m from the beginning of the bag. In each application point the amount of tablets inserted were related to the volume of grain contained in between two applications points. Considering that a 60 m long bag holds 260 m^3 of wheat, a section of 5 m contains 21.5 m^3, implying that for T1 a total of 16 tablets were inserted in the grain mass in each application point (16 g PH_3/21.5 m^3 = 0.75 g/m^3).

A plastic tube of 40 mm in diameter was introduced diagonally toward the center and bottom of the grain mass and the tablets were placed inside, with the precaution of spreading the tablets throughout the profile of the grain mass in each application point. This was achieved by lifting of the plastic tube a few centimeters after dropping some tablets. The contact between the aluminum phosphide tablets and the plastic cover was avoided by leaving a layer of grain in between the tablets and the plastic cover to prevent an eventual damage of the plastic cover during the exothermic reaction of the tablets when releasing the gas.

The measurement of gas concentration was made with a hand pump and colorimetric tubes (Drager, Lubeck, Germany − accuracy ±10% *SD*). The measurement was performed every 1–2 days from application at the closing end of the silo bag, in between application points and at the application point.

2.2.1. Maximum theoretical concentration

In order to quantify the evolution of PH_3 concentration in relationship to the initial dosage, the maximum theoretical

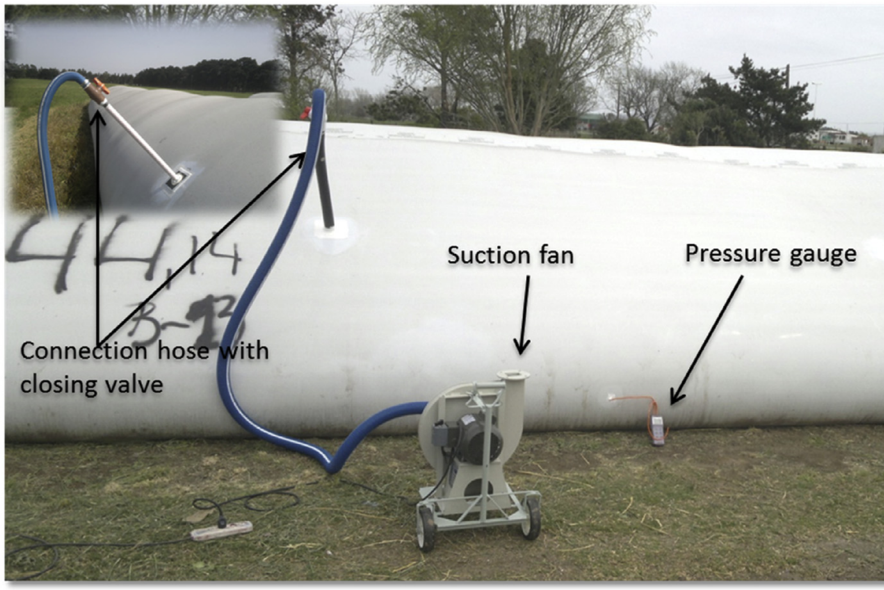

Fig. 1. Set up for conducting the pressure decay test, showing the fan, the hose connected to the silo bag and the pressure gauge.

Table 1
Silo bag sealing method, soil preparation, crop, silo bag capacity, pressure decay test (PDT), phosphine dosage, maximum theoretical concentration (MTC), CO_2 dosage, CO_2 average initial concentration and purging efficiency for the fumigation (F) and controlled atmosphere (CA) treatments.

Treatment	Thermo sealing	Soil preparation	Crop	Bag capacity (t–[m^3])	PDT (sec)	Phosphine dosage (gr/m^3)	MTC (ppm)
T1 – F	No	No	Wheat	200 [260]	<25[a]	0.79	1087
T2 – F	No	No	Wheat	200 [260]	<25[a]	1.50	2175
T3 – F	No	No	Sunflower	56 [175]	<25[a]	1.72	2494
T4 – F	No	No	Sunflower	60 [188]	<25[a]	1.64	2368
T5 – F	Yes	No	Wheat	67 [87]	48	1.00	1468

Treatment	Thermo sealing	Soil preparation	Crop	Bag capacity (t –[m^3])	PDT (sec)	CO_2 dosage (m^3/m^3)	Average concentration after purging (%)	Purging efficiency (%)
T6 – CA	No	No	Wheat	180 [233]	<5[a]	0.58	86	59
T7 – CA	Yes	Yes	Sunflower	50 [156]	2700	0.71	90	51

[a] Estimated based upon visual inspection of the bag.

concentration was calculated for each treatment as follows (Table 1):

$$MTC = \frac{D*725*(1-S)}{P} \quad (1)$$

where:

MTC is the maximum expected theoretical concentration of PH_3 (ppm); *D* is the dosage of PH_3 (g/m^3); 725 is the expected concentration of PH_3 with a dosage of 1 g/m^3 (ppm); *S* is the amount of PH_3 chemically sorbed by the product (dec); and *P* is the porosity of the grain bulk or void space (dec). The values considered for *S* was 0.2 (Reddy et al., 2007) and for *P* was 0.4 (ASAE, 2013).

2.3. Controlled atmosphere treatments

Two standard silo bags of 2.74 m diameter and 55 and 20 m long holding wheat and sunflower, respectively, were used to perform the controlled atmosphere trials (T6 and T7, respectively, Table 1). The silo bag used for T6 was set up in a farm, and the one used for T7 at an elevator. Both locations were close to Balcarce, Buenos Aires province, Argentina. The grain m.c. was around 14% for T6 and 11% for T7, and the grain temperature was in between 20 and 25 °C for both treatments.

The silo bag corresponding to T6 was set up directly in the field, without any particular care to preserve the hermeticity of the system (similar procedure to that described for T1 to T4), while the silo bag used for T7 was set up on cleaned and leveled soil to prevent damage or perforations in the bottom, and the ends were thermo sealed. The soil preparation was made with a tractor with a grader blade. No hermeticity test was performed in T6, but the PDT was estimated in less than 25 s due to the poor sealing condition (this is a typical PDT for silo bags not properly sealed). On the other hand, the PDT achieved in T7 was of 2700 s (Table 1), meaning that the bag had a very good airtightness level, more than 15 times higher than that recommended for CA treatments (180 s, according to Navarro, 1998). After the CA trial (25 days), the PDT decreased to 1200 s, but still remained well above the prescribed limit.

The CO_2 injection was made from a cylinder containing 120 kg of liquefied CO_2 which was connected to a gasifier (heat exchanger) with a hose, and then to a galvanized steel pipe that was inserted into the grain mass at the center of the silo bag through the plastic cover. The last 50 cm of the pipe were perforated to allow the flow of the CO_2 into the grain mass, and the edge of the plastic cover, in the perforation made to insert the pipe, was sealed to the pipe with silicon sealant and tapes, so hermeticity was not affected by the inserted pipe. At both ends of the bags two purging plastic tubes of 50 mm diameter were inserted through the plastic cover, so the CO_2

was flowing from the center of the bag toward the ends (Fig. 2). The goal was to achieve an initial CO_2 concentration above 80% average in the bags. After the desired initial concentration was achieved, the injection and purging pipes were removed and the perforations sealed with a duct tape.

The measurement of the CO_2 concentration was made with a portable gas meter (PBI Dansensor, Checkpoint, Denmark), perforating the plastic cover with a needle at different distances from the center of the bag to both ends. During the purging phase the measurements were made each 3–10 min, while during the treatment the measurement was made at least once a day.

2.3.1. Purging efficiency computation

The purging efficiency was defined as the amount of CO_2 that remained in the silo bag after the injection phase was concluded (without considering gas sorption by the grain), and its equation was derived from the retention efficiency equations presented by Alagusundaram et al. (1995) and Mann et al. (1999) as follows:

$$Pe = \frac{fc{*}\mathrm{d}v}{gv}{*}100 \qquad (2)$$

$$\mathrm{d}v = \frac{gm}{\delta}{*}v \qquad (3)$$

where: *Pe* is the purging efficiency (%); *fc* is the average final purging CO_2 concentration (dec.); *dv* is the domain volume (volume of the gaseous space of the bin, m^3); *gv* is the total CO_2 injected into the silo bag (m^3); *gm* is the total grain mass in the silo bag (t), δ is grain density (t/m^3); and *v* is the grain void space (dec.). The value of δ and *v* considered in this study were 0.75 and 0.4 for wheat and 0.32 and 0.4 for sunflower, respectively (ASAE, 2013).

3. Results

3.1. Silo bags hermeticity assessment

Fig. 3 shows that the PDT of 21 silo bags performed in the field resulted with a wide range of hermeticity levels (from almost 0 s to more than 1150 s). Based on these results, the typical setup of silo bags in the field cannot guarantee that the bag will be sufficiently gas tight for performing a successful fumigation or CA treatment. Thus, a protocol was developed for improving the hermeticity in a silo bag, which consisted in leveling and cleaning the soil with a tractor grader blade, thermo seal the ends of the bag (according to the description of the bag used for T7), and seal any visible perforation. The bag made according to this protocol was tested for hermeticity right after the bag was filled resulting with a PDT of 2700 s.

3.2. PH_3 fumigation treatments

3.2.1. Fumigation in unsealed silo bags

Fig. 4 shows the concentration of PH_3 in unsealed silo bags of wheat (T1: 0.75 g/m^3 of PH_3; T2: 1.5 g/m^3 of PH_3) and sunflower (T3: 1.72 g/m^3 of PH_3; T4: 1.64 g/m^3 of PH_3). No hermeticity test was performed in these bags however, based on hermeticity tests performed in other not sealed silo bags, the PDT was estimated in less than 25 s. The wheat fumigation treatments (T1 and T2) in unsealed silo bags failed because the concentration at the closing end of the bag did not pass the 200 ppm due to the leakage caused by the poor sealing implemented (folding the plastic cover on itself and adding some weight on top). In T1 (0.75 g/m^3 of PH_3), at the application point the highest PH_3 concentration was 430 ppm (40% of the MTC) at day 3, and then dropped to 180 ppm by day 8, with an average decay rate of about 55 ppm/day. In between application points the maximum PH_3 concentration was 250 ppm (180 ppm lower than at the application point) and dropped below 200 ppm by day 7. In T2 (1.5 g/m^3 of PH_3), even though PH_3 concentration reached 200 ppm by day 1 at the closing end, it quickly dropped below the target concentration in day 2. The maximum PH_3 concentration was 450 ppm at the application point at day 2 (21% of the MTC) and dropped to 30 ppm by day 7, with an average decay rate of 84 ppm/day. In between application points the maximum PH_3 concentration was 410 ppm. The concentration observed in the treatment with higher dosage (T2) resulted with a little higher maximum concentration, but only achieved the 21% of the MTC, and the concentration decreased more rapidly than in the bag with lower dosage (T1). An explanation for the lower % of MTC achieved and the greater concentration decay rate observed in T2 in comparison with T1 could be that the bag used for T1 had higher hermeticity than the bag used for T2, resulting this last treatment in a greater fumigant leakage that could not be compensated with the higher dosage.

The dosage for the sunflower fumigation treatments was increased to 1.72 and 1.64 g/m^3 for T3 and T4, respectively in order to compensate the PH_3 losses of unsealed silo bags observed in T1 and T2, and a possible larger sorption effect of sunflower (Reddy et al., 2007). Fig. 4 shows similar PH_3 concentration patterns in sunflower (T3 and T4) than that observed for wheat, but overall the maximum concentration achieved was substantially higher. However, at the closing end of the bag the concentration was between 200 and 300 ppm from days 3–7, showing that this is the weakest point of the bag and it might cause the failure of the fumigation treatment. In T3, the dosage was of 1.72 g/m^3 PH_3 with a peak of concentration of 1964 ppm (79% of the MTC) which occurred after three days from application at the application point, and then dropped to 120 ppm by day 8, with an average decay rate of about 370 ppm/day. In between application points the maximum PH_3 concentration was 764 ppm (1200 ppm lower than at the application point) and dropped to 120 ppm by day 8. Similar results were

Fig. 2. Images of the CA trial (T7) showing the CO_2 cylinders and the gasifier (left), the setup of the injection pipe at the center of the bag (center), and the purging pipes at the end of the bag (right).

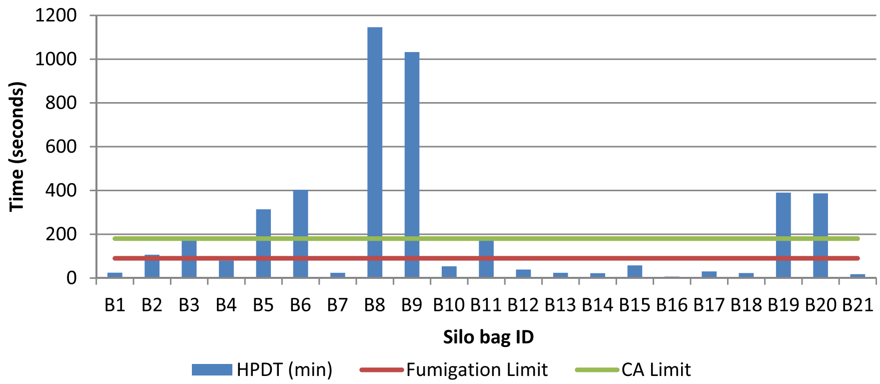

Fig. 3. Pressure decay test (seconds) for different silo bags and prescribed limits for fumigation and controlled atmosphere treatments.

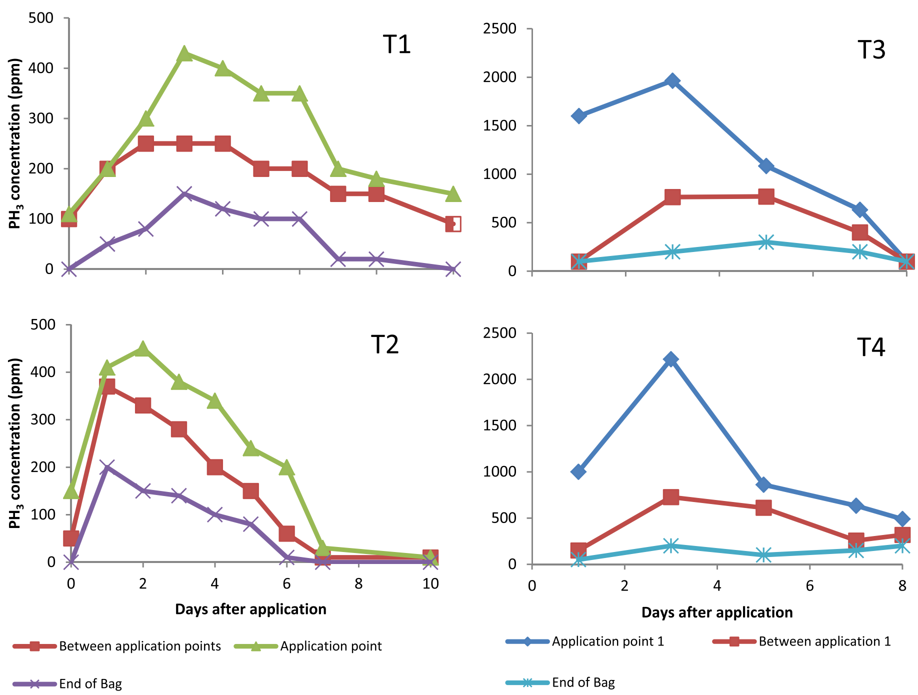

Fig. 4. Phosphine concentration in different measuring points of two unsealed silo bags of wheat with different dosages (T1: 0.75 gr/m$_3$ of PH_3; and T2: 1.5 gr/m$_3$ of PH_3) and two unsealed silo bags of sunflower with different dosages (T3: 1.72 gr/m$_3$ of PH_3; and T4: 1.64 gr/m$_3$ of PH_3).

observed in T4, but with a higher MTC (93%) and an average decay rate of about 345 ppm/day.

3.2.2. Fumigation in sealed silo bag

Fig. 5 shows the PH_3 concentration in a wheat silo bag in which both, the start and close ends, where thermo-sealed (with a PDT of 48 s) and the fumigation dosage was of 1 g/m^3 PH_3 (T5). The maximum PH_3 concentration at day 6 was 1000 ppm at one application point, at day 9 was the peak with 1150 ppm (78% of the MTC), and after, the concentration remained without significant change until day 13 (1100 ppm), in which the treatment was concluded. In the other measuring points the PH_3 concentration was always above 570 ppm (and increasing) from day 6–13, including at the closing end of the bag.

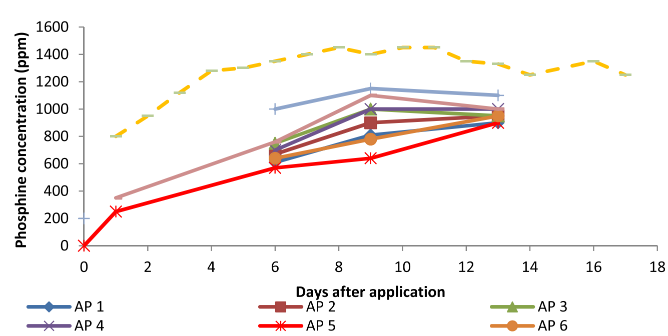

Fig. 5. Phosphine concentration in different measuring points (AP 1-7: application points; End: end of the bag) of a sealed silo bags of wheat with aluminum phosphorus tablets added after bagging, with a dosage of 1 gr/m^3 PH_3, and fumigation data extracted from Ridley et al. (2011) with a dosage of 1.6 gr/m^3.

3.3. Controlled atmospheres treatments

3.3.1. Controlled atmosphere in unsealed silo bags

A total of 255 kg of CO_2 were injected during the purging phase

to reach an initial concentration above 80% in average. The CO_2 flowed from the center of the bags (injection point) towards the ends, where the purging openings were set. Fig. 6 A and B show the CO_2 concentration at different sampling points from the center to the left and right ends, and it shows that about 290 min were required to reach 80% at the end of the bag (left side). The CO_2 concentration in all the sampling points was in between 80% and 100% (average 86%) when the purging phase was concluded. During the purging phase the injection had to be stopped four times (60, 90, 140 and 210 min) to defrost the heat exchange system (the evaporation rate was too high), and after that the injection was continued. This can be appreciated in the falls of the lines showing CO_2 concentration at 1, 3 and 10 m from injection at 60–70, 120–170 and 190–230 min, approximately. The purging efficiency was of 59%.

The treatment phase started with a CO_2 concentration of 80–100%, and rapidly dropped in all the measuring points (Fig. 6 C and D), especially at 1 and 3 m of the right side of the bag, where in a couple of hours the concentration was of 50–60%. After the first hours in which the CO_2 concentration had a large variation in some measuring points, the decrease rate stabilized from 9 to 4.1%/day. After 7 days of treatments the concentration was from 60% (Fig. 6 C, 20 m) to as low as 22% (Fig. 6 D, 3 m), indicating that the silo bag leaked significant amount of CO_2, and that the leakage was not uniform in the different sections of the silo bag.

3.3.2. Controlled atmosphere in sealed silo bags

A total of 203 kg of CO_2 were injected during the purging phase. Fig. 7 A and B shows the CO_2 concentration at different sampling points from the center to the left and right ends. The injection was stopped at 150 min, since the entire bag was at 90–100% (average 90%), but the sampling point at 11 m (the right end of the bag) which was at 45%, assuming the CO_2 will move by diffusion and increase the concentration in that sampling point as well. The purging efficiency was of 51%.

The treatment phase started with a CO_2 concentration of 50–100%, and after a few hours, the concentration equilibrated between 70 and 80% in the entire bag (Fig. 7 C and D), remained above 70% even after 18 days, and 24 days after injection the CO_2 concentration was still above 60% in all measuring points.

4. Discussion

4.1. Silo bags hermeticity assessment

According to Navarro (1998), a structure of less than 500 t, 95% full, could be suitable for fumigant treatment if the PDT is greater than 90 s, while for CA treatment the PDT should be greater than 180 s. Results shown in Fig. 3 indicated that about 40% of the bags in the field were airtight enough for a fumigation treatment and that only 30% could be used for CA treatment, pointing out that silo bags have the potential of being hermetic, but that condition should not be given for granted. This remarks the importance of performing a PDT in silo bags before deciding whether to conduct a fumigation of a CA treatment. Darby and Caddick (2007) found similar results and arrived to the same conclusion.

The implementation of a simple protocol (cleaning soil, thermo-sealing the ends of the bag and patching visible perforations) allowed to achieve a PDT of 2700 s, which exceeded by 15 folds the recommendation of Navarro (1998). After 20 days in the field, the PDT dropped about 50% (1200 s), but the airtightness level was still well above the required for any fumigation or CA treatment. Additionally, to improve hermeticity in silo bag, do not excessively probe the bags (reduce probability of losing the patches of the perforations), install a fence around the silo bag to keep animals away, keep the areas close to the bag clean, implement a rodent control protocol, and use repellents. Other practical recommendation is to conducts the fumigation or CA treatment right after filling

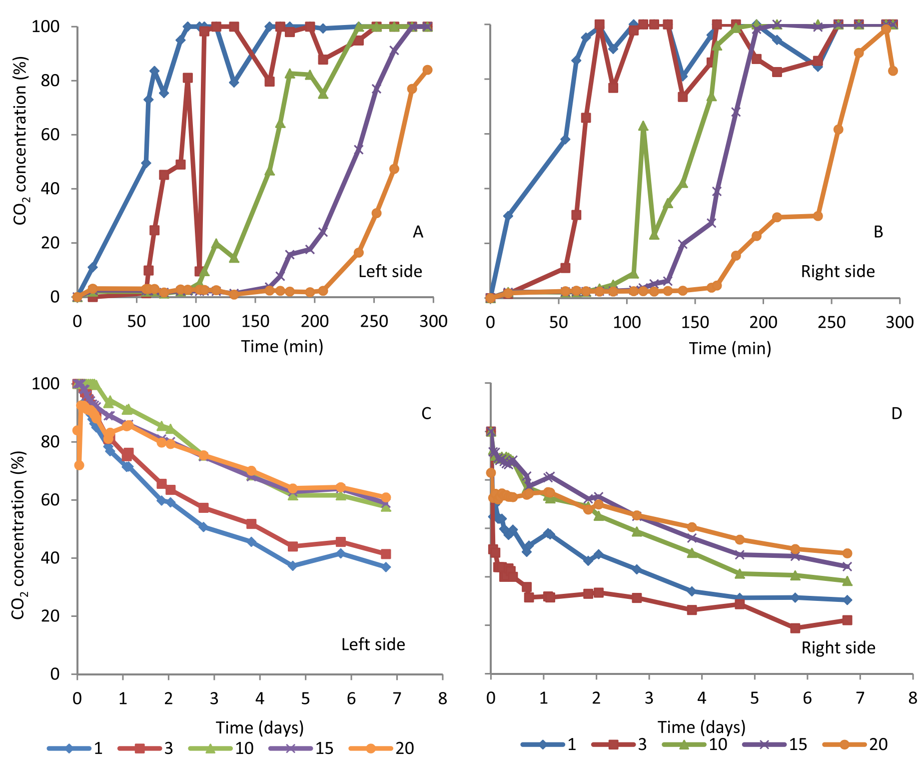

Fig. 6. Carbon dioxide concentration during the purging phase (A and B) and concentration during treatment (C and D) at different locations from the injection point (1–20 m) for the left and right side of an unsealed silo bag full of wheat (T6).

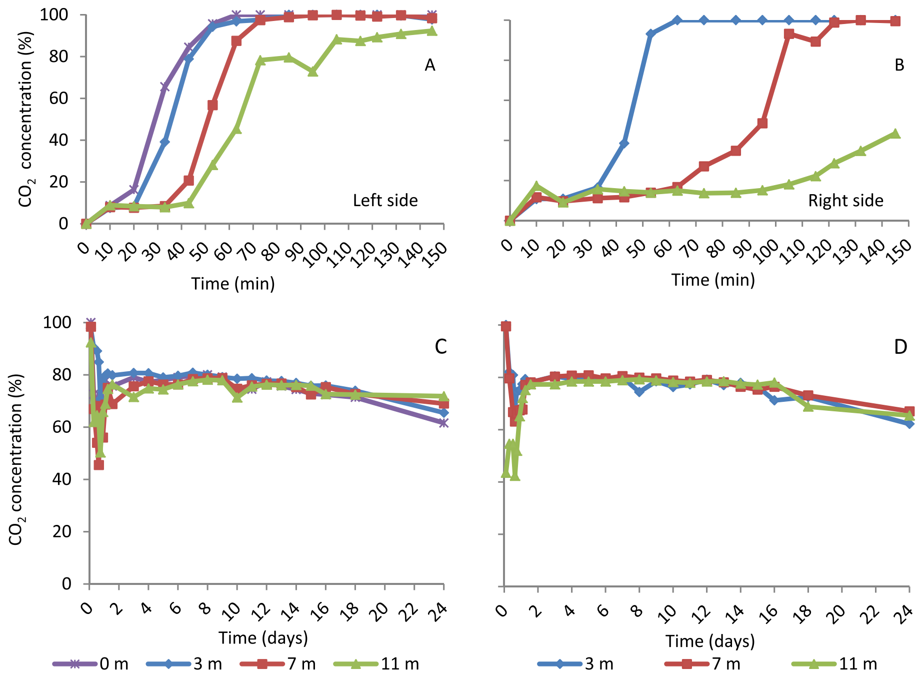

Fig. 7. Carbon dioxide concentration during the purging phase (A and B) and concentration during treatment (C and D) at different locations from the injection point (0–11 m) for the left and right side of a sealed silo bag full of sunflower (T7).

the silo bag, when the hermeticity is the highest, since the hermeticity of the bags most likely will be compromised after some months of storage in the field (Darby and Caddick, 2007). None of the recommendations described above are difficult to implement or require unreasonable expenses.

4.2. PH_3 fumigation treatments

Airtightness greatly affected the success of fumigation in silo bags, mainly due to fumigant leakage at the closing end which prevented to achieve the Argentine fumigation standard (200 ppm for 5 days) (Fig. 4). If fumigation must be implemented in a poorly sealed silo bag, in addition to making efforts for improving airtightness, increasing the dosage at the closing end of the bag and/or re-applying fumigants after 3–4 days from the initial application of aluminum phosphide tablets at the closing end section of the bag could help to compensate the fumigant leakage. On the other hand, when the fumigation treatment was performed in sealed silo bag the concentration at the closing end was similar to the concentration in other locations of the bags, resulting in a successful fumigation, even when the same PH_3 dosage was applied at the closing end than at the other locations (Fig. 5).

Estimating fumigant losses during treatment is critical to target an initial dosage that would allow for a successful fumigation. Fumigant losses affect the maximum concentration, the time at which the maximum concentration is reached and the decrease rate of concentration after the maximum. In unsealed silo bags the peak of gas concentration was achieved about 2–3 days after application, and after the peak, the concentration quickly dropped with a decay rate from 55 to 370 ppm/day. According to the data reported by Navarro and Zettler (2000), the daily losses of PH_3 in poorly sealed containers should be around 220–250 ppm, which are in between the range observed in this study. On the other hand, in the fumigation treatment (T5) performed in a sealed silo bag the maximum concentration was observed 9 days from application and, after the peak was reached, the concentration remained stable during 4 days with minimal losses. Thus, fumigation in sealed silo bag resulted in higher and more uniform concentrations, including at the closing end (PH_3 concentrations above 500 ppm from day 5–13).

In the sealed silo bag a discrepancy was noted with the data reported in the literature and the derived recommendations. Fig. 5 shows that after the PH_3 concentration peak was reached, it remained almost unchanged during at least 4 days, indicating that the losses were much lower than that predicted by Navarro and Zettler (2000) (220 ppm/day for a PDT of 60 s). As a result, fumigation in T5 succeeded even though the PDT was of 48 s, significantly lower than the threshold of 90 s suggested by Navarro (1998). Two possible hypotheses could be offered to explain why a silo bag with insufficient PDT resulted with a successful fumigation with the prescribed dosage of 1 gr/m^3. The first one would imply that the PDT conducted was not representative of the true level of airtightness of the bag, which is not likely since the PDT is a simple procedure and when the test is replicated the results are highly similar. The second would imply that the threshold set by Navarro (1998) might not be appropriated for flexible grain silo bags, since in this kind of storage system there is no head space and, theoretically, no leakage of gas occurs as result of pressure release. In Fig. 5 is plotted the PH_3 concentration measured at the application point in a wheat silo bag treated with a dosage of 1.6 g/m^3 (data extracted from Ridley et al., 2011). The peak concentration for this fumigation occurred 9 days after application, and after reaching the peak, the concentration remained stable until day 17. These authors did not conduct a PDT on the silo bags used for fumigation, so comparison of gas tightness with our treatments is not possible. The concentration profiles from Ridley et al. (2011) are, however, similar with that of T5 and signifficantly different than those of T1–T4. This implies that the hermeticity achieved in the silo bags used by Ridley et al. (2011) is roughly equivalent to that achieved in T5 and would confirm the speculations that in sealed silo bags the fumigant losses are lower than those expectd for storage containers with head space. These observations might indicate that additional

fumigation test should be carried out to better estimate the fumigant losses based on the PDT for silo bags.

Other indicator for fumigant losses is to relate the measured fumigant concentration with the TMC. A measured concentration close to 100% of the TMC indicates that the silo bag had small fumigant losses, and otherwise. Measured fumigant concentration for unsealed silo bags of wheat were from 10 to 40% of TMC (for the end of the bag and the application point, respectively) and from 8 to 93% of MTC for the unsealed silo bag of sunflower, while for sealed silo bag of wheat were from 71 to 78% of TMC, indicating that in sealed silo bags a greater proportion of the initial dosage of PH_3 remained inside the silo bag. The difference between the measured concentration and the TMC could be caused by losses through opening, permeability of PH_3 through the plastic cover (Ridley et al. (2011) measured PH_3 concentration of 32 ppm in external containers in fumigated silo bags), and PH_3 sorption effect different than that considered in this analysis (20%).

The distribution frequency of aluminum phosphide tablets in the silo bag (distance between application points) is critical. If distance between application points is too close, a large number of perforations in the bag have to be made (increasing risk of hermeticity failure and more labor involved). In the opposite situation, fumigant concentration in between application points could result too low and cause fumigation failure. According to Figs. 4 and 5, inserting aluminum phosphide tablets every 5 m along the bag allows for a good distribution of PH_3, being able to reach the 200 ppm in between application points. Similar results were obtained by (Ridley et al., 2011) in a wheat silo bag where the application points were every 7 m interval.

4.3. Controlled atmospheres treatments

According to Annis (1986), in order to assure complete control of all insect species except Trogoderma granarium, an initial concentration of 70% declining to 35% in 15 days is needed. Based on this criterion, CA treatment in unsealed silo bags (T6) would not assure insect control, since there were sections in the bag in which the concentration dropped below 35% at the first day, and by day 7, 3 measuring points were already at 35% or below (Fig. 6). If the treatment were extended beyond 7 days, based on the concentration decay rate estimated (9–4.1%/day), most of the section of this silo bag would result with concentrations below 35% before 15 days. The results of T6 confirm that CA treatment cannot be successfully implemented in silo bags with poor hermeticity (unsealed and with PDT of less than 25 s).

CA treatment in sealed silo bag (T7) largely accomplished Annis (1986) recommendation regarding effective concentration, implying that this silo bag had a high level of hermeticity (PDT for this bag was of 2700 s before the trial, and 1200 s 24 days later) and gas leakage was very small (Fig. 7). It can be noticed that during the first hours of the treatment phase, the CO_2 concentration showed a reduction in all the measuring locations of approximately 10% points, which was subsequently recovered a few hours later (Fig. 7, C and D). This reduction in concentration was related to the CO_2 sorption effect of the grain (Cofie-Agblor et al., 1998), which was produced when the sunflower seeds were exposed to increasing CO_2 concentration during the purging phase (which reached almost 100% in most of the bag), and later, when the CO_2 concentration stabilized inside the bag, the sorbed CO_2, and the CO_2 concentration in the interstitial air, reached an equilibrium.

Minimizing the amount of CO_2 required for a successful fumigation would reduce the CA treatment cost. This could be achieved by increasing the purging efficiency, reducing gas leakage and refining initial concentration. The purging efficiency of 50% obtained in this trial could be improved by considering more injection points, increasing the injection rate, or concluding the injection phase at a CO_2 concentration lower than 90%. It was demonstrated that a silo bag with high airtightness level (PDT of 2700 s) resulted in minimum CO_2 leakage. Additionally, as a results of the small leakage, the initial concentration was maintained during 18 days, suggesting that a lower target concentration (i.e. 70%) could be also suitable for achieving complete insect control.

This trial showed that CA treatments with CO_2 to control insects could be successfully implemented in large scale silo bags. One of the main limitations for implementing CA treatments is the cost and labor involved in achieving a suitable airtight condition in traditional storage systems (bins, warehouses, etc). Taking into consideration that silo bags are extensively used for storing different grains because they are a low cost system, and that making an airtight silo bag for CA treatment does not require significant extra cost or labor, silo bags could help in the implementation of CA treatments in grains and oilseeds.

5. Summary and conclusions

The most common silo bag setup in the field implies no soil preparation, so it is likely that residues of previous crops, weeds with strong stems, stones, etc. perforate the bottom of the bag. The beginning of the bag is closed with a knot, and the finish end of the bag is careless closed by folding the plastic cover on itself and adding some weight on top. During storage, no major maintenance of the plastic cover of the bag is implemented, so perforations caused by rodents, dogs and other wild and domestic animals are not sealed. As a result, the hermeticity of a standard silo bag is rather poor, which was evidenced with a PDT of only a few seconds. Under these conditions, fumigation with an initial dosage of about 0.75–1.7 g PH_3/m^3 could fail to achieve the Argentine standard of PH_3 fumigation of 200 ppm during 5 days in the entire bag. It was observed that the most critical section of the bag was the closing end, thus increasing the dosage in this section, or making a reapplication of fumigant after 3–4 days of the first application, could help to maintain the effective concentration of 200 ppm during 5 days.

Thermo sealing the ends of the silo bag, and simple care of the plastic cover (patching perforations) increased the PDT to close to 60 s, and this assured a successful fumigation treatment in the entire silo bag with a dosage of 1 g/m^3.

The CA treatment in a non thermo-sealed silo bag (with an estimated PDT of less than 25 s) was not able to maintain a concentration above 35% of CO_2 more than 8 days (even less than 1 day in some measuring points) when the initial concentration was of 80–100%. Cleaning the soil where the silo bag was placed, thermo-sealing the ends of the bags and patching eventual perforations allowed achieving a PDT as high as 2700 s. Under this condition, a successful CA treatment with CO_2 was implemented; starting with an initial concentration of 70–80%, and 18 days after, the concentration was still above 70% in all the measuring points, assuring complete control of major grain stored pests.

This study proved that with simple and inexpensive practices (soil preparation, patching perforations and thermo-sealing the ends), successful PH_3 and even CA treatments can be implemented in silo bags. Silo bags could be used as a cost competitive hermetic storage technology for performing CA treatments.

Acknowledgements

The authors want to thank to Fugran Comercial e Industrial S.A. through Guillermo Romero; Air Liquide Argentina S.A. through Gabriel Alfonzo; Cargill S.A.C.I.; and Scorziello y Galella S.A. for their cooperation in this study. Special thanks to Leandro Cambareri for

his help and support in the field work. This work was supported by the National Institute of Agricultural Technology (PNAIyAV 1130023 and PNCyO 1127022), and a grant from the silo bag consortium companies

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