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## **Implementing CO<sub>2</sub>-Based Controlled Atmosphere Treatments in Big Bags with Inexpensive Liners**

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### **ABSTRACT**

The production of specialty grains such as quinoa, amaranth, teff, popcorn, peanuts, and different organic crops involves meeting specific safety requirements, including the necessity to be free of insecticide residues. Consequently, in recent years there has been a growing interest in alternative controlled atmosphere (CA) treatments. On the other hand, market opportunities have required the adaptation of CA application to big bags and raffia containers typically holding 1 m<sup>3</sup> of product. Normally, the CA treatment system consists of a gas application system (CO<sub>2</sub> or N<sub>2</sub>), an internal polyethylene bag with specific design and gas barrier properties, and a closure via heat sealing. However, these systems can prove costly, limiting their usability for numerous products. The purpose of this experiment was to assess the feasibility of implementing a CA treatment (with CO<sub>2</sub>) through the design of simpler and more cost-effective technologies, aiming to expand the user base of CA treatments. The experiment involved analyzing the effectiveness of a simple and economical polyethylene bag (70 microns, without a gas barrier) in combination with two closure systems: a simple one (twisting-folding-knot) versus the control system (heat-sealing). The control condition was established when a concentration × time product (Ct product) of 12,000 %h was reached (minimum allowed concentration of 40%). The initial injection created an internal atmosphere of 90% CO<sub>2</sub>. Overall, it was observed that treatments with a heat-sealed closure achieved satisfactory control conditions with a single initial injection, reaching the target Ct product while consistently maintaining the CO<sub>2</sub> concentration above 40%. In contrast, treatments with the knot-closure method did not ensure adequate sealing, requiring gas reinjections in some cases to achieve the control condition. In conclusion, this study demonstrates the feasibility of implementing a successful CA treatment in raffia big bags using low-cost polyethylene liners. However, it is crucial to employ the heat-sealed closure system to ensure the efficacy of the treatment.

**Keywords:** Specialty grains, Controlled atmosphere, Carbon dioxide, Big bag, Liners

## INTRODUCTION

Argentina boasts a large expanse of organic agricultural land (Research Institute of Organic Agriculture FiBL, 2021), encompassing approximately 100,000 ha dedicated to crops. Among these hectares, 33% are specifically dedicated to the cultivation of cereals and oilseeds (SENASA, 2022). This cultivation encompasses a variety of crops, including traditional cereals such as wheat, corn, barley, and oats, as well as oilseeds like sunflower and soybean. Additionally, Argentina cultivates various other organic crops, such as white sorghum, amaranth, and teff.

Despite their differential price premiums, organic crops typically yield lower outputs compared with conventional crops (Seufert et al., 2012), require strict protocols for segregation, and have severe restrictions on the use of chemical inputs (Research Institute of Organic Agriculture FiBL, 2021). Organic crops encounter significant challenges in controlling insects during storage, primarily due to the restricted availability of alternatives to conventional insecticides. Among the few options utilized are diatomaceous earth and essential oils (IICA, 2009). In this context, the use of modified atmospheres (MAs) emerges as an alternative to traditional insecticides (Pons et al., 2010). Modified atmospheres function by altering the storage environment to create conditions for insect control without relying on insecticide application, thereby minimizing pesticide residues (Banks et al., 1990). Such atmospheric modifications require airtight storage conditions and can either be self-generated through biological processes within the stored bulk (auto-modified atmosphere) or externally induced by enriching the intergranular atmosphere with nitrogen (N<sub>2</sub>) or carbon dioxide (CO<sub>2</sub>) (controlled atmospheres) (Navarro and Donahaye, 1990).

The investigation into MAs with flexible liners has extended from small-scale airtight storage technologies (usually 30–100 kg capacity), which are designed for low-resource users (Baributsa and Ignacio, 2020), to notably large-capacity polypropylene containers known as big bags, boasting a volumetric capacity of 1 m<sup>3</sup> (700–800 kg). These containers (plain raffia bags, without airtight conditions) have garnered extensive use in Argentina for the storage and/or transportation of organic grains. One key advantage they offer is the optimization of logistics and operational efficiency, particularly in managing intermediate grain volumes ranging from 5 to 30 t. In contrast to smaller 30–100 kg bags, which necessitate manual handling, big bags can be efficiently managed with forklifts, thus reducing labor requirements.

Research in this field has employed internal polyethylene bags engineered with specific design and gas barrier properties (e.g., ethylene-vinyl alcohol copolymer (EVOH) or polyamide), sealed through heat-sealing techniques, thereby demonstrating effective insect control (Pons et al., 2010). In recent years, commercial products have been developed with a focus on providing packaging with high barrier properties and a high degree of automation in sealing and gas application. However, the widespread adoption of such advanced technologies remains hampered by associated costs, thereby confining its utilization to a minority of organic crop producers and/or marketers in Argentina and much of South America.

The utilization of liners lacking (cost-effective) gas barrier properties poses the challenge of establishing and sustaining an effective atmosphere. Previously, our research group investigated the feasibility of implementing MA systems with raffia big bags and cost-effective polyethylene liners using soaked grains as O<sub>2</sub> scavengers to maintain a hypoxic atmosphere (Bartosik et al., 2021; Taher and Bartosik, 2018). While our study demonstrated the conceptual feasibility of this approach, the practical utility was limited by the development of an unpleasant moldy grain odor, thus constraining its application in real-world scenarios.

The objective of this study was to evaluate the viability of implementing a controlled atmosphere (CA) treatment for organic grains utilizing CO<sub>2</sub> by developing simplified and economically viable technologies. The aim was to broaden the accessibility of this technology to a wider user base.

## MATERIAL AND METHODS

The experiment was conducted at an organic grain storage facility located in Balcarce city, Argentina, utilizing eleven big bags containing various organic grains, including teff (6 bags), amaranth (2 bags), white sorghum (2 bags), and wheat (1 bag). Prior to the implementation of the CA treatment, the grain was transferred from the original containers to the raffia big bags, which were internally lined with polyethylene bags.

The polyethylene liners had a dimension of 2.4 m in length and 1 m in diameter, with the lower end bellows folded and heat sealed. The upper end remained completely open without any additional features for facilitating closure. The material composition of the bag comprised a standard 70 micron thick monolayer polyethylene liner lacking any barrier properties, commonly employed for holding construction material (such as sand) and available at a market price of about USD 3.

After filling, nine of the big bags were sealed using the twisting-folding-knot system (KS), with consistent adherence to a standardized procedure (Fig. 1). Conversely, the remaining two big bags were sealed using a heat-sealing system (HS), employing a portable heat sealer (La Pipiola, Argentina), which was connected to a 12 V power source. Subsequently, gas injection into the big bags was carried out using compressed CO<sub>2</sub> from 120 kg cylinders. Each cylinder, equipped with a pressure regulator, was connected to a flexible hose. This hose was inserted into the grain mass through a 5 cm incision in the upper side of the liner, aiming to reach the bottom of the bag with the assistance of a metal rod. This incision also served as a gas purge point during injection. The CO<sub>2</sub> concentration during injection was continuously monitored at the purging point using a portable gas analyzer (Motomco, USA), with measurements taken at 30 s intervals until a concentration of 90% in the exhausted gas was achieved. Finally, the incision utilized for CO<sub>2</sub> injection and gas purging was sealed with a special rubber-based tape (Rivamar, Argentina).



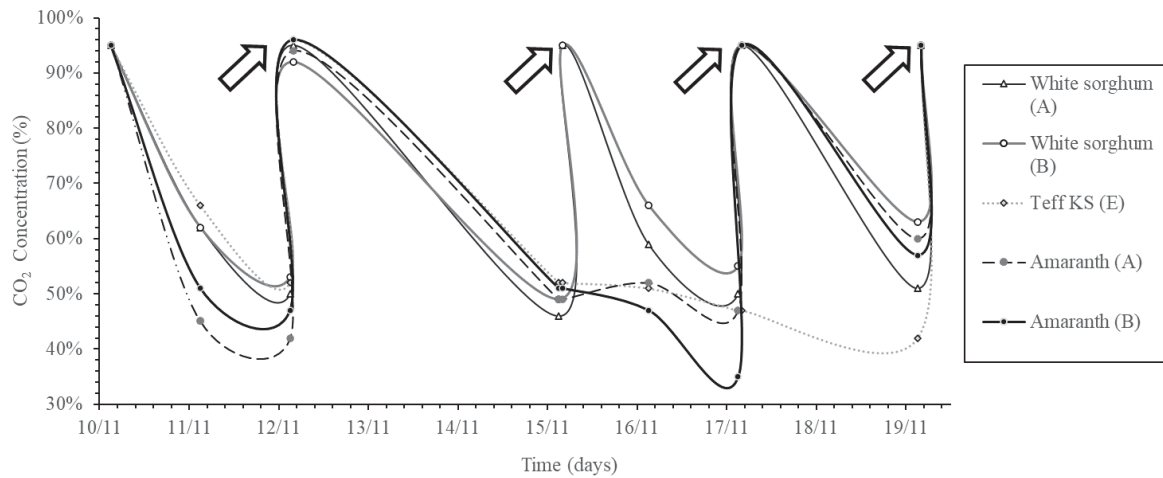
**Fig. 1.** Left: Big bag closed using the twisting-folding-knot system. Right: Big bag closed using the heat-sealing method (indicated by arrow).

Following gas application, CO<sub>2</sub> concentration inside the big bags was monitored every 24 h throughout the treatment duration. Gas samples were extracted by inserting a histological needle through a septum placed in the liner of the big bag. The goal was to maintain a minimum CO<sub>2</sub> concentration of 40%, with a reinjection procedure implemented whenever the CO<sub>2</sub> concentration dropped below this threshold, aiming to restore the internal concentration to 90%. The criterion employed to determine the duration of treatment entailed calculating the cumulative sum of the products derived from the concentrations attained (%) and the duration of exposure (h), yielding the cumulative concentration × time (Ct product) metric. Treatment completion was defined as achieving a Ct product value equal to or greater than 12,600 %h (Alagusundaram et al., 1995).

## RESULTS AND DISCUSSION

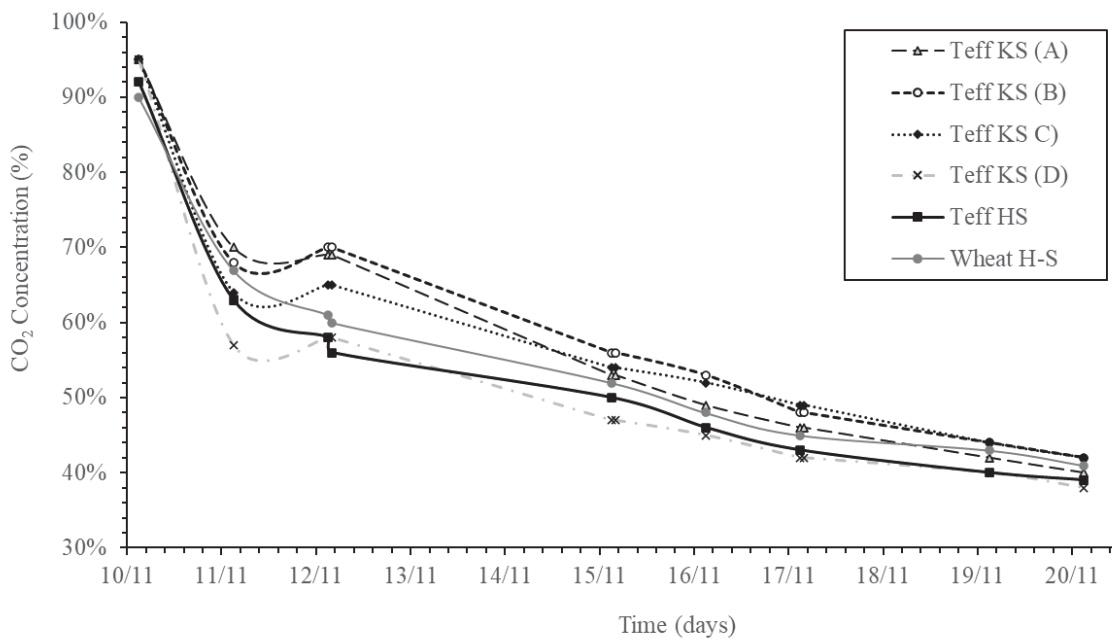
The injection time required to attain the target CO<sub>2</sub> concentration of 90% was less than 5 min per big bag. The injection procedure adhered to the guidelines outlined by Garcia (2020), which advocates for placing the purging point as far as possible from the injection point. This positioning facilitates an effective sweeping or piston effect within the intergranular air due to the injected gas, optimizing the distribution and dispersion of CO<sub>2</sub> throughout the grain mass.

The two big bags with the HS system achieved the control criteria with only one injection, indicating satisfactory airtightness. In contrast, only four out of the nine big bags utilizing the KS mechanism attained the same condition (Table 1). The remaining five big bags employing the KS system necessitated reapplications, as the CO<sub>2</sub> concentration fell below 40% within 2–3 d post injection (Fig. 2). Moreover, discrepancies were noted in the number of reinjection cycles required, ranging from 1 to 4.



**Fig. 2.** Carbon dioxide concentration (%) over time (d) for big bags requiring gas supplementation (arrows indicate the moment of gas reapplication).

In the big bags where a single application was sufficient, it is noteworthy that the CO<sub>2</sub> concentration experienced a pronounced decline during the initial 24 h period, dropping from approximately 90–95% down to 60–70% (Fig. 3).



**Fig. 3.** Carbon dioxide concentration (%) over time (d) for big bags requiring a single gas application.

This phenomenon can be attributed to the sorption process, the extent of which varies depending on factors such as the grain type, moisture content, and temperature, and may exhibit partial reversibility (Yamamoto and Mitsuda, 1980). Subsequent maintenance or marginal augmentation

of gas concentration is indicative of the partial reversibility of this sorption phenomenon. Following the first 24 h, the CO<sub>2</sub> concentration exhibited a near-linear decline until reaching a level of 38–40% after 10–11 d post treatment. This gradual reduction in concentration is attributed to potential leakage through the sealing system or natural permeation through the plastic liner. It is pertinent to note that the sustainment of an effective intergranular CO<sub>2</sub> concentration is determined not only by gas leakage but also by CO<sub>2</sub> sorption by the grains themselves (Cofie-Agblor et al., 1998). This aspect assumes particular significance when considering the application of CA treatments in grains characterized by high sorption rates.

The target Ct product (12,600 %h) was reached earlier in the big bags that had undergone more reapplications (white sorghum, 8 d) (Table 1). The required exposure time gradually increased to a maximum (10–11 d) in the big bags with a single application; this is because the CO<sub>2</sub> values were frequently restored to levels above 90%. The concentration of CO<sub>2</sub> remained elevated in the hours following the application, thereby increasing the average concentration compared with the big bags with a single application.

Although the control criterion was also achieved in the big bags requiring multiple injections, the practical implementation of CA treatments under this condition could pose challenges. Continuous monitoring is essential to detect when reinjection is necessary. Additionally, the process of reinjection demands additional labor and consumes more CO<sub>2</sub>. For instance, treatments requiring a single application were estimated to consume 1.3 kg of CO<sub>2</sub>, while those necessitating four additional reinjections consumed a total of 4.2 kg (Table 1).

**Table 1.** Number of gas injection cycles required, total CO<sub>2</sub> consumed and time (h) to reach the required Concentration time (Ct) -product (12600 %h) for the different big bags with twisting-folding-knot and heat-sealing closure systems.

Big Bag	Closure system	Number of CO <sub>2</sub> applications	Total CO <sub>2</sub> consumption (kg)*	Time to reach target Ct (h)
White sorghum (A)	Twisting-folding-knot (KS)	5	4.2	200
White sorghum (B)				
Amaranth (A)		4	3.5	215
Amaranth (B)				
Teff (E)		3	2.8	220
Teff (A)				
Teff (B)		1	1.3	240-250
Teff (C)				
Teff (D)				
Teff				
Wheat	Heat-sealing (HS)			

\*Estimate made based on initial and final CO<sub>2</sub> concentrations, an interstitial air volume of 0.4 m<sup>3</sup>, and a purging efficiency of 50%.

Therefore, while multiple injections may achieve the desired outcome, they also entail increased resource consumption and labor, highlighting the importance of optimizing the sealing procedure to minimize these drawbacks.

## CONCLUSIONS

Our results suggest that a single-shot treatment suffices when the big bag is adequately sealed, even with a non-barrier liner. Consistent with this, Navarro (2013) noted the efficacy of a solitary CO<sub>2</sub> treatment when an initial concentration exceeding 70% is established, maintaining a concentration above 35% for at least 10 d. However, the use of the KS did not consistently achieve the desired airtightness. Similar observations were made in a previous study evaluating the KS through a pressure decay test (Bartosik et al., 2021). Challenges in achieving consistent airtightness with the KS may stem from the larger size of the big bag's opening (2 m wide), which exceeds that of smaller bags (30–100 kg capacity with 0.5 m wide opening) employing the same closure mechanism (Arthur et al., 2022). Exploring alternative technological solutions, such as bags with smaller openings that can be easily sealed after filling, may mitigate these challenges despite potential increases in packaging costs. Conversely, the HS technology offers greater predictability in closure system outcomes.

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