CASE STUDY

Evaluating CO_2 Tracer Gas Ingress for Integrity Assessment of Flexible Film Packaging

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Introduction

Ensuring Container Closure Integrity (CCI) in parentals is critical for maintaining sterility and product stability in pharmaceuticals.[1] Flexible film packaging systems (e.g., bags and pouches) present specific challenges for Container Closure Integrity Testing (CCIT) due to material permeability, seal variability, and susceptibility to micro-defects.[2] [3] Several CCIT technologies are currently applied to such systems, including probabilistic methods such as dye ingress and bubble emission testing, as well as deterministic techniques like High Voltage Leak Detection (HVLD) and vacuum decay. While these methods are well established, their applicability to flexible film formats may be influenced by package compliance, material permeability, product conductivity, and test sensitivity limitations for very small defects.

In this study we present how laser-based Headspace Analysis (HSA), with carbon dioxide (CO_2) pretreatment can be applied to packages of flexible film. This concept with detection of ingress of CO_2 as a trace gas has been demonstrated for other packaging systems, like vials and is today presently used in the industry but has been limited to rigid containers. Tunable Diode Laser Absorption Spectroscopy (TDLAS) enables highly sensitive and specific detection of gases such as CO_2. When CO_2 is used as a tracer gas, CCIT can be evaluated by measuring controlled ingress into the package headspace through defects in the film or seals.

This study evaluates CO_2 tracer gas ingress combined with laser-based HSA as a deterministic approach for CCI assessment of flexible film packaging systems.

Honourable Mention

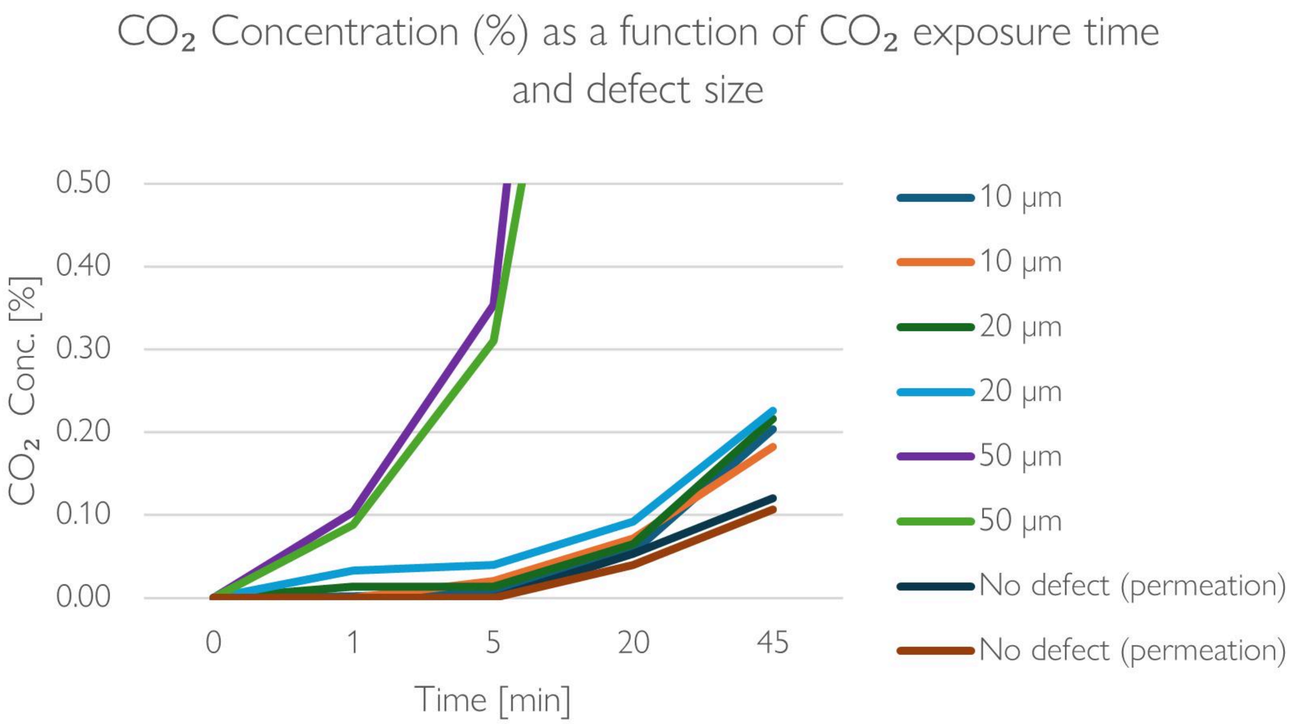
This research was conducted in collaboration with Oxford Lasers, whose expertise supported the evaluation of laser based CO_2 tracer gas analysis as a deterministic solution for flexible packaging integrity.

GPX1500 Film Pharma - Instrument used in study

Method

Flexible film bags were prepared with controlled artificial defects created by precision laser drilling and certified by Oxford Lasers. Three nominal defect sizes were introduced: 10 µm, 20 µm, and 50 µm.

- All bag samples were placed in a pressure vessel and exposed to 100% CO_2 at 1 bar overpressure for 45 minutes to allow tracer gas ingress through the defects.
- A subset of samples was additionally subjected to 24 hours of overpressurization to evaluate permeation behavior over time.
- Internal headspace CO_2 concentration was measured using the Gasporox GPX1500 Film Pharma instrument using laser-based HSA technology, with measurements performed at baseline (pre-exposure) and after 1, 5, 20, and 45 minutes of CO_2 exposure.
- CO_2 concentration was reported as percent headspace concentration and evaluated as a function of defect size and exposure time.



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Results

The CO_2 ingress study demonstrates a clear correlation between defect size and gas accumulation over time under 1 bar overpressure. Bags containing a 50 µm laser-drilled defect showed rapid and substantial CO_2 ingress, reaching 4.8% concentration at 45 minutes. In contrast, bags with a 20 µm defect resulted in significantly lower accumulation, reaching approximately 0.22% at 45 minutes. For bags with a 10 µm defect, CO_2 levels increased more gradually, approaching 0.19% at 45 minutes.

Extended pressure pretreatment time demonstrated measurable contributions from film permeation, reaching 5.48% at 24 hours; however, defect-driven ingress remained clearly distinguishable from background permeation. Notably, the permeation-driven CO_2 ingress exhibited a non-linear time dependence, consistent with diffusion-controlled transport rather than linear accumulation over time.

Defect size	0 min	1 min	5 min	20 min	45 min	24 h
10 µm	0.00	0.00	0.01 - 0.02	0.06 - 0.07	0.18 - 0.2	
20 µm	0.00	0.01- 0.03	0.01 - 0.04	0.07 - 0.09	0.22 - 0.23	
50 µm	0.00	0.09 - 0.10	0.31 - 0.35	1.25 - 1.87	4.31 - 5.29	
No defect (permeation	0.00	0.00	0.00	0.04 - 0.05	0.11 - 0.12	5.34 - 5.62

Conclusion

Measurement of CO_2 tracer gas ingress using laser-based HSA provides a sensitive, quantitative, and deterministic method for CCIT of flexible-film packaging systems. Under controlled overpressure conditions, CO_2 accumulation in the package headspace correlated directly with laser-drilled defect size, demonstrating clear distinction between 10 µm, 20 µm, and 50 µm defects. The method enabled objective detection of micro-defects while distinguishing ingress from material permeation, which is critical for polymeric and semi-permeable systems. These results demonstrate that laser-based CO_2 tracer gas analysis is a robust, deterministic, and scalable solution for CCI testing of flexible packaging systems, offering improved sensitivity and objective measurement.

References

[1]United States Pharmacopeia (2017). General Chapter, ⟨1207⟩ Package Integrity Evaluation—Sterile Products. USP-NF. Rockville, MD: United States Pharmacopeia. https://doi.org/10.31003/USPNF_M99926_01_0

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