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EVALUATION OF THE EFFICIENCY OF OXYGEN ABSORBERS DURING STORAGE

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Abstract: Oxygen absorbers allow for the effective extension of the shelf life of food products. A necessary condition is the correct selection of the absorber for the size of the packaging and its correct operation. Oxygen absorbers have a factory-declared minimum sorption capacity, but manufacturers do not declare the period during which this parameter will be maintained. The lack of information on the period for maintaining the original parameters of oxygen absorbers means that users do not know how long from the date of production they can still use the absorbers so that they provide optimal protection for the products packed with them. The aim of the work was to assess the sorption properties of absorbers after a specified period of storage in the original bulk. Sorption capacity was determined using a non-invasive, fluorescent method of measuring oxygen. The obtained results were compared with the results obtained for brand new oxygen absorbers of the same type. Additionally, the properties of packaging materials (i.e. thickness, composition, oxygen transmission rate) from which the factory packaging of the tested absorbers was made were determined, as well as the effect of the type of packaging on maintaining sorption capacity.

Keywords: oxygen absorber, quality, absorption efficiency, shelf life.

1. INTRODUCTION

Many food products are sensitive to the presence of oxygen, which can decrease their quality due to the autoxidation of fats, L-ascorbic acid, vitamin E, dyes, some amino acids, enzymatic browning reactions, and the growth of aerobic bacteria. These processes can affect the taste, smell, and colour of products, which can influence the consumers' purchasing decisions [Böhner et al. 2014; Cichello 2014; Zardin et al. 2016].

The presence of oxygen in food packaging often results from errors in the packaging process or the improper selection of packaging materials. It may also result from processes occurring in the product or through penetration through the packaging material [Kozak 2016; Kaiser, Schmid and Schlummer 2018].

One method to prevent the effects of oxygen is to completely remove it from the packaging, e.g., by using oxygen absorbers. Oxygen absorbers, often called oxygen adsorbents, allow the removal of oxygen introduced during packaging and penetrating through the packaging material [Cierpiszewski 2016].

Oxygen absorbers are a form of active packaging used to maintain or improve the quality of food products. This group of solutions also includes moisture absorbers, ethylene absorbers, antimicrobial packaging, etc. [Realini and Marcos 2014; Fuertes et al. 2016].

Research on oxygen absorbers began in the early years of the 20th century. The first commercial products appeared in the 1970s [Charles, Sanchez and Gontard 2006]. In the following years, oxygen absorbers developed mainly in Japan, the USA, and Australia [Solovyov 2009].

Oxygen scavengers are becoming increasingly attractive to food producers and traders. The Global Oxygen Scavenger Market was valued at USD 1.7 billion in 2024 and is expected to reach USD 2.36 billion by 2033, at a CAGR (Compound Annual Growth Rate) of 3.7% [The Business Research Company 2025]. Oxygen scavengers are often produced in sachets (Fig. 1), but they are also found in labels, films, cards, and bottle caps [Dey and Neogi 2019; Gupta 2023].

The most popular group of oxygen absorbers is solutions based on iron oxidation, which are introduced into the absorbing element as a powder. Due to the high sorption capacity of iron compounds, oxygen adsorbents based on them are the most effective absorbers available on the market. They can adsorb from 20 to 2000 cm^3 of oxygen and reduce its concentration in the package to below 0.01%. A disadvantage of this type of absorber is the need to maintain a relatively high humidity in the package [Cruz, Camilloto and dos Santos Pires 2012; Soltani Firouz, Mohi-Alden and Omid 2021]. The equations present the mechanism of iron oxidation in such absorbers are:

$$4\,Fe \rightarrow 4\,Fe^{2+} + 8e^-$$

$$2\,O_2 + 4\,H_2O + 8\,e^- \rightarrow 8\,OH^-$$

$$4\,Fe^{2+} + 8\,OH^- \rightarrow 4\,Fe(OH)_2$$

$$4\,Fe(OH)_2 + O_2 + 2\,H_2O \rightarrow 4\,Fe(OH)_3$$

When the reaction rate of the product with oxygen and the rate of oxygen transport through the packaging material are known, the above equations allow for the calculation of the required amount of the absorber to maintain the desired oxygen concentration in a given time [Cruz, Camilloto and dos Santos Pires 2012]. The reaction rate of iron contained in absorbers with oxygen has been studied in many works [Miltz and Perry 2005; Polyakov and Miltz 2010; Kombaya-Touckia-Linina et al. 2019]. The adsorption rate should be high enough for the oxygen in the package to react with the absorber and not the product contained in the package [Lai and Wong 2022; Deshmukh, Hakim and Gaikwad 2023]. The effect of temperature and relative humidity (RH) on the absorption kinetics has also been studied [Polyakov and Miltz 2010; Feng et al. 2013]. Data on the actual absorption capacity of oxygen scavengers and the absorption rate are required to design an optimal and economical package using the scavenger. Information provided by the manufacturers of oxygen scavengers does not always contain all the information required to design an appropriate package. Much more information is needed to provide insight into the performance of oxygen scavengers in various environments. This lack of information is particularly evident in the case of oxygen-scavenging films, sheets, labels, trays, and containers [Miltz and Perry 2005].

Another serious problem when selecting an adsorbent is its appropriate capacity, i.e., the amount of oxygen the absorber can adsorb. The absorber capacity is important because it must be adapted to the volume of the packaging and the amount of oxygen that can penetrate the packaging material. It will also depend on the type of packaged product, its dimensions, mass, water activity, the rate of oxygen penetration into the product, or the required shelf life [Vermeiren et al. 2003]. Other important features that absorbers must have are their durability and the lack of change in properties over time [Rooney 1995].

Manufacturers indicate many of their properties, such as capacity, in the information about absorbers, but none of the absorbers known to us provide a shelf life for such a product. This study aimed to investigate the effect of long-term storage on the absorption capacity of commercial oxygen absorbers. To date, such data has been lacking in the literature. This information will enable more effective use of oxygen absorbers in commercial practice.

2. MATERIALS AND METHODS

2.1. Materials

The study was conducted on commercial oxygen absorbers from three manufacturers: Mitsubishi Gas Chemical (Japan), IMPAK/Sorbentsystems (USA), and CHUNWANG (China) with different nominal sorption capacities of 10 cm^3, 750 cm^3, and 200 cm^3, respectively (Fig. 1). The nominal sorption wasn't investigated by the authors. This information was given by manufacturers of investigated oxygen scavengers on dedicated product websites. The absorbers from Mitsubishi Gas Chemical, called PharmaKeep™, had the form of small containers and were factory-packed in double bags made of metalized foil. Each bulk bag contained 1000 PharmaKeep™ oxygen scavengers. In contrast, the products from the other two companies came in sachets and were packed in single bags made of non-metalized foil. Absorbers made by IMPAK are called StayFresh and were packed 25 sachets per bulk bag, while absorbers from CHUNWANG, known as just oxygen absorbers, were packed 100 sachets per bulk bag. All absorbers were vacuum-packed. In order to determine the effect of storage time on the sorption efficiency of the absorbers, the study was conducted on two series of absorbers, i.e., factory new absorbers and absorbers of the same type, but previously stored at room conditions for 8 (PharmaKeep™ absorbers) and 10 years (StayFresh and CHUNWANG absorbers), respectively.

A B C

Fig. 1. Oxygen absorbers used for testing:
A – PharmaKeep™, B – StayFresh, C – CHUNWANG

Source: own study.

2.2. Statistical analysis

Statistical analyses included factorial one-way ANOVA, followed by Tukey's honest significant difference (HSD) test at $\alpha = 0.05$. All statistical analyses were performed using the Statistica software, version 13.3 (TIBCO Software Inc., USA).

2.3. Measurement of oxygen absorption capacity and rate

In these studies, the sorption properties of oxygen absorbers were determined using the OxySense® 325i measuring system (Industrial Physics, USA). This system measures oxygen concentration optically by using the phenomenon of selective fluorescence quenching, with the sensor placed inside the tested package under the influence of oxygen. The study was conducted in accordance with the methodology included in the ASTM F2714-08 standard (ASTM - American Society for Testing and Materials). Twist-off glass jars of various capacities were used to determine the sorption properties of the tested absorbers.

The choice of this type of packaging resulted from the fact that the glass is transparent (enabling optical measurement of oxygen concentration) and additionally does not allow oxygen to pass through, which is a necessary condition for obtaining objective results during measurements of the sorption capacity of the oxygen absorbers. Two Schrader-type twist valves were additionally installed in the jar lids, enabling easy and quick changing of the internal atmosphere, which was necessary during calibration and proper measurements. On the inner wall of the jar, OxyDot oxygen indicators were additionally glued (using transparent double-sided tape), allowing for the measurement of oxygen concentration using the OxySense system.

Nitrogen 5.0 and compressed air were used as gases for testing and calibration. The study began with placing the originally packed absorbers in a glove-box type chamber, type 810 (PLAS-LABS Inc., USA). Then, the absorbers were placed in the aforementioned measuring packages. Carrying out this operation in an anaerobic chamber was necessary since even short-term contact of the absorbers with oxygen contained in the air would change their original properties. The chamber used in the study had a transfer chamber through which it was possible to place the necessary equipment and materials inside. After placing the absorbers in the measuring jars, they were pulled out and then filled with air. The moment of filling the jars was determined based on parallel oxygen measurements. The volume of the measuring jars was adjusted so that the oxygen volume in the jar, after being filled with air, exceeded the declared nominal capacity of the tested absorbers. It allowed the determination of the completion of the measurement process at the saturation point of a given absorber with oxygen. The sorption capacity of a given oxygen absorber was calculated based on the difference between the initial and final oxygen concentration in the jar.

Additionally, the time after which the minimum oxygen concentration was measured in the jar was recorded. On this basis, the average rate of oxygen absorption by a given absorber was calculated. The capacity of the jars was determined with an accuracy of 1 cm^3 by measuring the amount of water in a closed jar. After filling the jars with water, the jar lids were mounted with open valves (with Schrader inserts removed), and then the jars were refilled with water through them to ensure the volume of the valves was included in the measurements. After filling the jars, the valves were closed by installing the Schrader inserts (Fig. 2).

Fig. 2. Measuring jar, Schrader valve and insert

Source: own study.

The amount of water corresponding to the volume was determined by weighing the difference between a jar filled with water and an empty jar using a technical scale with an accuracy of 1 g. Before weighing, the jars filled with water were wiped dry, paying particular attention to the valves. Tests were performed in jars with volumes of 317, 2639, and 4263 cm^3, which provided the optimum volume for the sorption capacity of the tested absorbers. Jars with the lids were previously tested for tightness. The actual sorption tests were performed on 10 pieces of each type of absorber, with five being brand new and the other five stored for the period mentioned previously.

2.4. Measurement of oxygen transmission rate (OTR)

Oxygen transmission rate (OTR) was determined according to the ASTM F3136-15 standard. The measurement system consisted of the OxySense 325 oxygen analyser and a dedicated permeation chamber with a built-in optical oxygen sensor (Industrial Physics, USA). During the measurement, changes in oxygen concentration within the permeation chamber were recorded. The oxygen concentration measurement was made in accordance with the ASTM F2714-08 standard. The test samples of the bulk packaging were cut into squares with sides of 6.5 cm and placed between two halves of a permeation chamber (Fig. 3). The point of contact between the sample and the chamber was sealed using special silicon grease. Nitrogen of 5.0 purity was used as the deoxidizing gas to achieve an oxygen-free atmosphere in the measuring chamber. At the same time, atmospheric air was the oxygen carrier during the actual permeation process. The permeation chambers were stored in a climatic chamber with the temperature set to 23°C and the relative humidity set to 65%. The oxygen measurements were performed periodically until the determination coefficient between the individual measurements was greater than 0.95. Three replicates were made for each packaging material. The OTR results were given in $cm^3\ O_2/1m^2 x24$ h.

Fig. 3. OTR permeation chamber, samples of tested bulk packaging films

Source: own study.

2.5. Measurement of the oxygen absorbers packaging bag thickness

The thickness of the bags in which the absorbers were factory packed was measured using a Mitutoyo ID-C112B (Mitutoyo, Japan) thickness gauge with an accuracy of 0.001 mm. The measurements were taken at five locations using the same samples as for the oxygen transmission rate measurement. The thickness results were given in micrometres.

2.6. Infrared spectroscopy FT-IR

Film identification was performed using a Jasco FT/IR-4X FT-IR spectrometer equipped with an ATR apparatus and a monolithic diamond. Spectra were recorded from 4000 cm-1 to 400 cm-1, with a resolution of 0.4 cm-1 and 16 scans per sample. Initial spectral processing, including normalization and baseline correction, was performed using Spectra Manager Suite for FT/IR-X series software. The materials of the films were identified using the Advanced Spectra Search software.

3. RESULTS AND DISCUSSION

Figure 4 shows an example graph of the sorption capacity measurement of an oxygen absorber. The difference between the initial and final oxygen concentration in a container of known capacity allows the calculation of the absorber's adsorption capacity. However, care should be taken to select measuring containers so that the final concentration does not reach values close to zero. In such a case, the calculated sorption capacities may be burdened with an underestimation, the size of which cannot be predicted. The average adsorption rate can also be calculated from the curve of the change in concentration versus time.

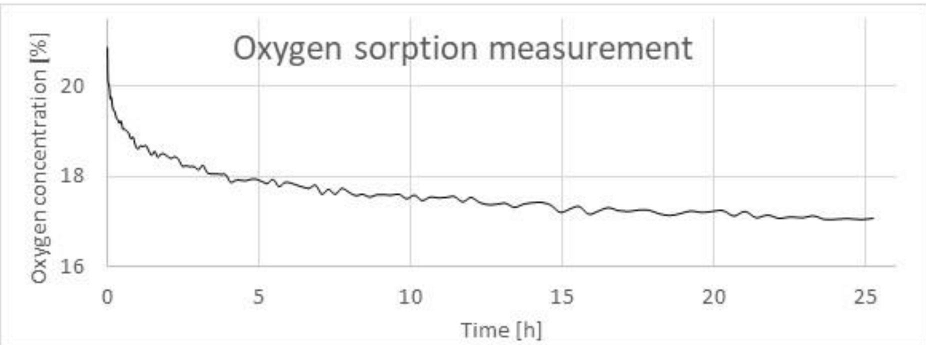

Fig. 4. Example of a measurement chart of the sorption capacity of an oxygen absorber (StayFresh SF750, jar volume – 4263 cm^3)

Source: own study.

Table 1 presents the measured capacities of oxygen absorbers and their sorption rates. The results show that newly purchased absorbers have significantly higher capacities than those declared by the manufacturers. The STAYFRESH absorbers had the closest capacities to the declared capacities, differing on average by about 11%. In contrast, the CHUNWANG absorbers differed on average the most, by as much as 332%, which was surprising. PharmaKeep™ absorbers showed an average capacity that was one and a half times greater than the declared capacity. A completely different situation occurs when comparing the sorption capacities of absorbers stored for extended periods, i.e., 8 and 10 years. In such cases, all capacities are lower than those measured initially for brand-new absorbers. The relatively lowest reduction in sorption capacity was observed for PharmaKeep™ absorbers, where the measured value still exceeded the declared value. On the other hand, the remaining products retained about 15% (StayFresh absorber) and 5% (CHUNWANG) of their original capacity, respectively.

As the studies have shown, the absorbents tested also differed significantly in terms of absorption rate. In this case, the higher absorption rate was characteristic of oxygen absorbers with a larger nominal capacity. However, absorbers stored for a long time showed a decrease in absorption speed. This decrease can be explained by partial oxidation of the active ingredients of the absorbers during storage, which makes it more difficult for oxygen to access them.

Table 1. Results of determination of oxygen absorber capacity and sorption rate depending on storage time

Absorber name, Manufacturer, Country of origin	Type	Nominal sorption capacity [cm^3]	Factory new		Stored	
			Measured sorption capacity [cm^3]	Measured average sorption rate [cm^3/h]	Measured sorption capacity [cm^3]	Measured average sorption rate [cm^3/h]
PharmaKeep™ Mitsubishi Gas Chemical, Japan	CD-1G	10	16.8 ±1.4^a	0.35 ±0.03^a	12.7 ±1.1^{*b}	0.21 ±0.02^b
StayFresh, IMPAK Corporation, USA	SF750	750	831.3 ±48^a	42.4 ±2.3^a	124.4 ±21^{**b}	5.7 ±0.9^b
CHUNWANG, CHUNWANG, China	200cc	200	864.7 ±54^a	22.6 ±2.4^a	45.2 ±14^{**b}	2.5 ±0.7^b

* stored 8 years
** stored 10 years
^{a,b} Values marked with different letters in a data group are statistically significantly different at a confidence level of 0.95, according to Tukey's test.

Source: own study.

The results obtained for absorbers stored for an extended period correspond to the properties of the packaging materials in which they were stored. A necessary condition for maintaining the sorption properties of absorbers is the use of packaging material with the best possible barrier properties against oxygen. As the conducted tests showed, these materials differed in both composition and thickness, which directly translated into their oxygen transmission rate (OTR). The best protection was provided by the material used by Mitsubishi, i.e., metalized PE/PET laminate. At the same time, it was the thickest material.

Additionally, to enhance the protection of the packed absorbers, double packaging was used at the factory. The packaging of StayFresh absorbers provided poorer protection, with a non-metalized PE/PET laminate being used. The packaging of CHUNWANG absorbers provided the worst protection, with a non-metalized PE/PP laminate being used. It was also the thinnest material.

Details of the aforementioned packaging materials determined during the tests are presented in Table 2.

Table 2. Properties of packaging materials of the tested oxygen absorbers

Absorber Name, Manufacturer, Country of origin	**Packaging material thickness [μm]**	**Packaging material composition inner/outer layer**	**Packaging material OTR [cm^3 O_2/m^2x24h]**
PharmaKeep™ Mitsubishi Gas Chemical, Japan	2x124 ±1*	PE/PET*	3.4 ±0.04*
StayFresh, IMPAK Corporation, USA	120 ±1	PE/PET	14 ±1
CHUNWANG, CHUNWANG, China	110 ±1	PE/PP	20.6 ±0.3

* double packaging, metallized laminate

Source: own study.

4. CONCLUSIONS

The conducted studies indicate that all tested, brand-new oxygen absorbers have a sorption capacity significantly greater than that declared by the manufacturers. This is evident in the case of CHUNWANG absorbers. It speaks positively about the manufacturers because the actual sorption capacity of the absorbers is greater than the declared capacity, allowing all operations related to the application of the absorbers in the packaging without excessive loss of the ability to absorb oxygen from the packaging atmosphere during the real product's shelf life. During the placement of absorbers during the packaging process, there is practically always a short-term exposure of the absorbers to atmospheric air containing about 21% oxygen, causing a partial loss of their original sorption capacity. Sorption capacity greater than the declared one eliminates this problem. Higher than the declared capacity also allows for the optimization of oxygen absorbers, enabling the use of smaller and cheaper ones.

However, this is possible only if there is prior experimental verification of real sorption capacity before using the absorber.

It is also worth emphasizing that oxygen absorbers stored under room conditions for 8 or 10 years, packed in factory collective packaging, still retain the ability to absorb oxygen. Of course, there is a noticeable decrease in this ability, which is related to, among other things, the barrier properties and the type and thickness of the materials used in the collective packaging of oxygen absorbers, which was the subject of the presented research.

However, considering such a long storage period, the results are a positive surprise and offer practical tips for potential users of oxygen absorbers, suggesting their later use is possible, not necessarily immediately after purchase. The oxygen absorbers tested differed in terms of the oxygen absorption rate and, as the tests showed, the long storage period of the absorbers also had an impact on this parameter – the average oxygen absorption rate decreased, which can be explained by the partial oxidation of the active substances contained in the absorbers. As a result, access of oxygen to the remaining oxygen absorbing substances was made difficult.

The research also showed how important are the type and properties of the material used in the factory's collective packaging of the oxygen absorbers.

In this case, using double metalized laminate provides the best effect in maintaining the sorption properties of the absorbers. The use of a single package made of non-metalized laminates does not provide such good protection for the oxygen absorbers.

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