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RESEARCH ARTICLE

Packaging Film Impact on Food Organoleptic Properties: An Experimental Study

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ABSTRACT

Most packaged food products deteriorate in quality due to mass transfer phenomena such as moisture absorption, oxygen invasion, flavor loss, undesirable odor absorption and packaging material migration. An approach to increase the shelf life of the packaged food, while exploiting the advantages of flexible packaging, is to use flexible multi-layer polyethylene (PE)-based films including a barrier layer.
This paper reviews different oxygen barrier layers used in Doy Pack flexible pouches for fruit puree and presents the impact of storage conditions on the organoleptic properties.
In this study, the performances of five types of PE film pouched filled with fruit puree have been studied and compared.

Regarding the organoleptic properties, this study focused on the 5 Hydroxy Methyl Furfural (HMF) increase, the L-Ascorbic Acid reduction, the brightness and the yellow color intensity decreases versus storage conditions. From those measurements, kinetic models of these organoleptic characteristics have been extracted and their relative behavior at longer shelf life have been calculated. Those results can be used as a reference by laminates process and development packaging engineers to support PE multi-layer film structure definition according to the targeted food application and shelf life conditions.

1.0 INTRODUCTION

Among all the requirements that food & beverage primary packaging have to meet, one of the most important is the product protection itself. To achieve preservation and safe delivery until consumption, primary packaging must be designed and developed to prevent or at least delay chemical, microbiological and physical deteriorations of the content. [1], [2],[3]

Over and above the attributes of the food itself, the quality of the packaged product is then directly linked to the used packaging materials. Actually, most food products deteriorate in quality due to mass transfer phenomena such as moisture absorption, oxygen invasion, flavour loss, undesirable odour absorption and packaging material migration. [4]

An approach to improve protection and increase the shelf life of the packaged food, while exploiting the advantages of flexible packaging, is to use flexible multi-layer polyethylene (PE)-based films including a high barrier layer. This paper reviews different film structures with oxygen barrier layers used in stand up flexible pouches for fruit puree and presents the impact of shelf life on the organoleptic properties.

2.0 FOOD PACKAGING FUNCTIONALITIES

2.1 The Last but not the Least: Food Protection

A non-exhaustive list of performances that food packaging must meet could be the following one:

- Be a sufficient containment for the final product
- Provide an easy transportation & storage
- Give clear information to consumers
- Have an attractive appearance
- Facilitate distribution, marketing & recycling
- Presents convenience features (such as easy opening, reclosability, portability, handling…)

However, the first and most fundamental requirements of packaging remains the protection and the safe delivery of food products from manufacturing until consumption. [1], [2], [3]

Packages must actually create an acceptable barrier for food products from biological, chemical, physical degradations. They must prevent or at least, delay the loss of nutrients, functional properties, colour, aroma, taste and preserve the general appearance expected by consumers.

By consequence, the barrier capability of a package, in particular with respect to water vapour, oxygen, and microorganisms will impact the shelf life, the length of time that product remains in an acceptable condition for use. [4]

2.2 Mass Transfer Phenomena

Permeation, absorption and diffusion are typical mass transfer phenomena occurring in packaging systems. Equations behind these phenomena have been widely reviewed. [3], [5], [6]

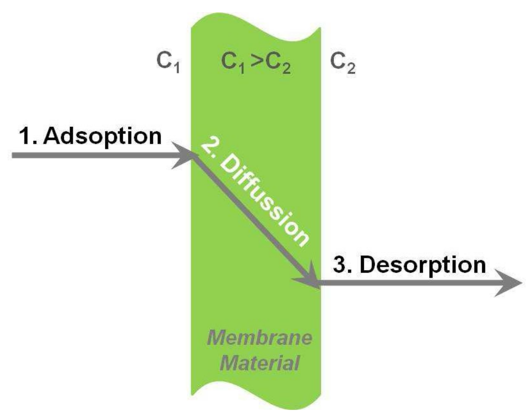


Fig. 1. Permeation Phenomenon Scheme

The measurement of the Permeability – also called Transmission Rate (TR) measures the flow of permeate without taking into account the thickness of the sample. Ensuing properties such as oxygen transmission rate (OTR) and water vapour permeability (WVTR) are expressed in [$g.m^{-2}.s^{-1}$] or more practically in [$g.m^{-2}.day^{-1}$].

Permeation mechanisms are specific to given polymers. It depends on the chemical nature of the polymer. It also depends on the polymers grafting, blending with other polymers or the addition of a plasticizing agent. Finally, it depends on the permeate characteristics such as solubility (as seen above), the size and shape of the molecules, polarity and its interaction with the polymer, explaining why barrier properties are classified according to the type of permeate. [7]

2.3 Oxygen Barrier Materials

Under ambient temperature and humidity, oxygen is most responsible for both oxidative and non-oxidative deterioration (appearance, mouth feel, change of flavour, microbial growth) of the packaged foods. [8] This explains why a large segment of commercial manufacturing deals with the production of packaging that extends shelf life of food by controlling oxygen transport.

Plastic material rarely answered to all properties as polymers usually have an intrinsic permeability either to water or oxygen. To combine both water and oxygen barrier properties in packaging, multi-layer and polymer mixtures are developed, as an alternative.

2.4 Oxygen Transmission Rate Measurement Method

The oxygen transmission of a film is generally measured using a Mocon Ox-Tran (Modern Controls Inc., Minneapolis, USA) in accordance with ASTM method D 3985-95 "Standard test method for oxygen transmission rate through plastic film and sheeting using a colorimetric sensor". [9]

The sample is placed as to separate the measuring cell in two. On one side, the air is continuously, purged with nitrogen whereas controls flow of oxygen is injected on the other side. The oxygen crossing over the sample is detected with a colorimetric sensor. A different instrument configuration, though based on the same principle, is used to run measurements on pouches (Fig.3 b). For the measurement of a finished pouch the method ASTM F1307 – 14 is used.

3.0 STUDIED PE-BASED MULTILAYER FILM PACKAGINGS

A typical packaging material for spouted pouches is a laminated multilayer film. In order to accomplish to the main feature of a primary packaging, every layer of the film composition has specific characteristics. A typical product filled into spouted pouches is fruit puree, a product shelf stable and with a usual shelf life of 12 months.

For this study, different multilayer film structures have been taken in consideration for the testing.

3.1 PET/ALU/PE

The reference film structure is the most common material used for the application and is composed as follows (from external to internal material):

- External PET is the printing support.
- Aluminium foil is the functional barrier for light and oxygen permeation
- PE layer is the welding layer and in direct contact with the product

The analytical study has been carried out taking in consideration this structure as reference and all the values are normalized on this structure.

3.2 PET barrier/OPA/PE

As an innovative family of film, the coated polyester, constitute an alternative to the aluminium foil in regards to the barrier for oxygen, though gaining transparency as plastic materials.

The coating of the polyester can be realized either with Aluminium Oxide, either with Silicium Oxide. The both have characteristics of barrier to oxygen varying according to the grade and quality of the coating. Aluminium Oxide has the advantage of better mechanical performances.

As valid alternatives to the reference structure, with different characteristics, two film structure of this family have been taken in consideration for the study: PET barrier high performance/OPA/PE and a PET barrier average performance/OPA/PE. The qualification of the performance is related to the barrier level based on technical datasheet

3.3 PET/PET/PE EVOH PE

Another technical solution to achieve oxygen barrier is the use of EVOH within the film structure. Ethylene vinyl alcohol has well known barrier properties when coextruded in a polymeric sandwich. The following film structure has been used for the study.

3.4 PET/PET/PE

In order to fully understand the interaction of oxygen with the product, also a film structure without any barrier property has been taken in consideration for the study.

3.5 Summary able of studied film structures

All the pouches have a rigid HDPE fitment for product consumption, therefor the name spouted pouches.

Table 1.

Sampled ID	Film Structure
Pouch 1	PET/ALU/PE
Pouch 2	PET barrier High Performance/OPA/PE
Pouch 3	PET barrier Average Performance/OPA/PE
Pouch 4	PET/PET/PE EVOH PE
Pouch 5	PET/PET/PE

4.0 EXPERIMENTAL DETAILS ON HIGH BARRIER FILM PERMEABILITY MEASUREMENTS

4.1 Experimental Techniques

Measurements were performed by CIS (Centro di Certificazione e Analisi comportamentale) in Bollate (Mi) [12], according to ASTM F1307 - 14 on the different pouches spouted listed above, in the following conditions:

Temperature: 23°C +/- 1°C
Relative Humidity: <10% O2, 90% +/- 3% N2
Oxygen pressure: 1 bar
Barometric pressure: 746 +/- 1 mmHg
Carrier flow: 10 cc/min
Sampling conditioning: >10 hours

4.2 Results Analysis

The following table reports the analytical results of the OTR test.

Table 2.

Sample ID	OTR cc/pkg x 24h x atm
Pouch 1	0,042 – 0,047
Pouch 2	0,033 – 0,038
Pouch 3	0,043 – 0,048
Pouch 4	0,043 – 0,047
Pouch 5	0,76 – 0,77

Range of values of sampling tested. Values referring to OTR for single packaging in 24h. The highly technological coated PET barrier high performance gives the best result even on the aluminum foil. The aluminum foil is subject to the presence of micro pin holes that can affect permeability.

Other transparent barrier materials are comparable with aluminum foil. The total absence of barrier material reveal a 20-time worst performance for Pouch 5.

5.0 EXPERIMENTAL DETAILS ON THE MEASUREMENTS OF FRUIT PUREE ORGANOLEPTIC PROPERTIES

5.1 Sample Description

Pouches in PE-based multilayer film listed in paragraph 2 have been filled with fruit puree and impact of storage conditions on organoleptic proprieties have been analyzed to correlate it with OTR properties of the different multilayer film composition.

5.2 Experimental Techniques

An accelerated aging has been simulated. Studied storage conditions temperature depending were the following ones: 4°C, 20°C, 30°C and 37°C for the temperature range. Samples checking and measurements were done at time interval as follow: 0, 1, 2, 3, and 4 months of aging. In collaboration with SSICA (Stazione Sperimentale per l'Industria delle Conserve Alimentari) in Parma [12] it has been defined typical indicators for the characterization of the product (fruit puree) in terms of organoleptic properties. Through the measurement and the variation of these indicators, it has been possible to evaluate the impact of the different film structure on the product during time.

The indicators chosen for this study are the 5 Hydroxy Methyl Furfural (HMF) increase, the L-Ascorbic Acid reduction, the Color Spectrum variation. Measurements were performed by SSICA in Parma [12]. From those measurements, kinetic models of these organoleptic characteristics have been extracted and their relative behavior at longer shelf life have been calculated.

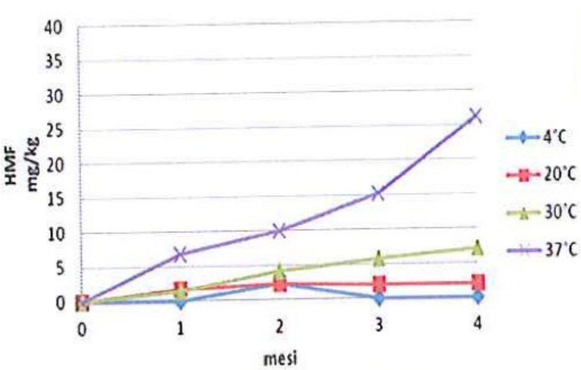
5.3 Five Hydroxy Methyl Furfural (HMF) Measurements Analysis

HMF is a chemical component that is generated by the dehydration and oxidation of certain type of sugars present in the product. It is an indicator, for example, of how the thermic treatment of the food has affected it. It is basically generated by the Maillard reaction and therefore is a good indicator of the oxygen permeation into the packaging. The higher quantity in the food, the worst is, as it means there has been availability of oxygen to feed the chemical reaction. The effect on the food is a color change of the product (browning) and taste deterioration.

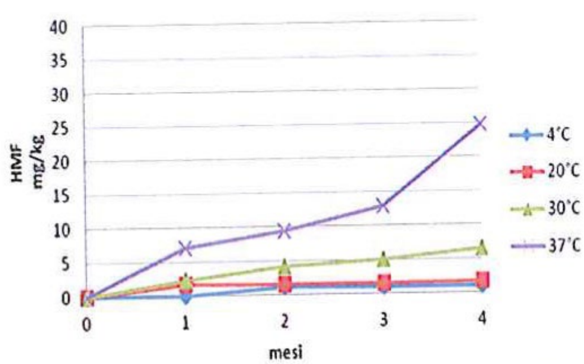
It is recognized that in a typical fruit puree the limit of acceptance of HMF is set at 20 mg/kg.

As method for measurement it has been used the liquid chromatography (HPLC), giving as result the concentration in mg/kg.

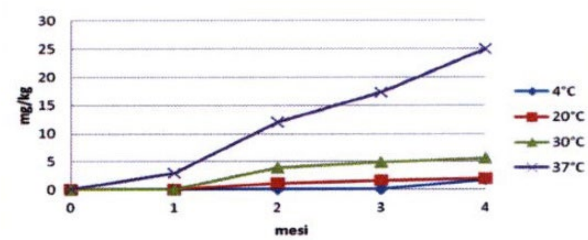
The following graphs represent the analytical results for the HMF:



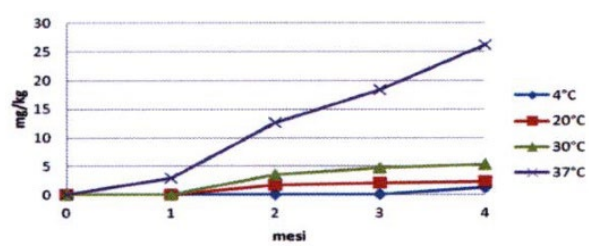
Pouch 1



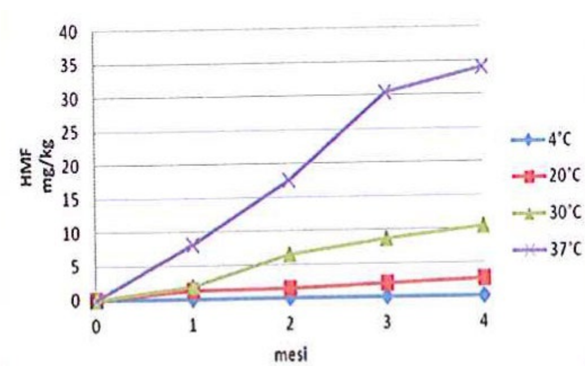
Pouch 2



Pouch 3



Pouch 4



Pouch 5

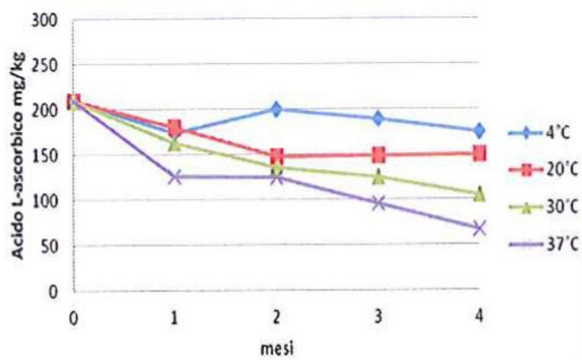
Table 3. The elaboration of the analytical data using extrapolation bring to the following evaluations. Concentration values are normalized on the reference value of Pouch 1

Sample ID	Reached threshold 20 [mg/kg] at 25 °C after	HMF concentration [mg/kg] after 12 months at 25 °C
Pouch 1	38 months	100
Pouch 2	32 months	102
Pouch 3	31 months	110
Pouch 4	26 months	110
Pouch 5	20 months	150

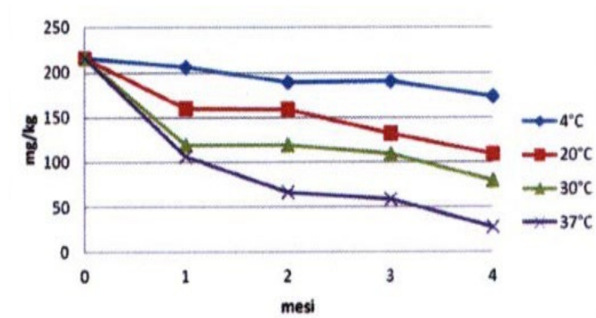
5.4 L-Ascorbic Acid Measurements Analysis

Ascorbic Acid is a naturally present vitamin in fruits, commonly referred as Vitamin C. In industrial manufacturing of certain categories of food can be added on purpose to increase the initial amount. It represents the indicator for the organoleptic and nutritional characteristic of a product. Degradation of this vitamin is related to heat, light and oxygen interaction. As method for measurement it has been used the liquid chromatography (HPLC), giving as result the concentration in mg/kg.

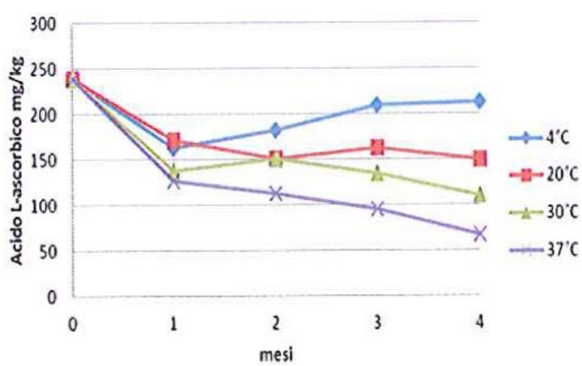
The following graphs represent the analytical results for the Vitamin C:



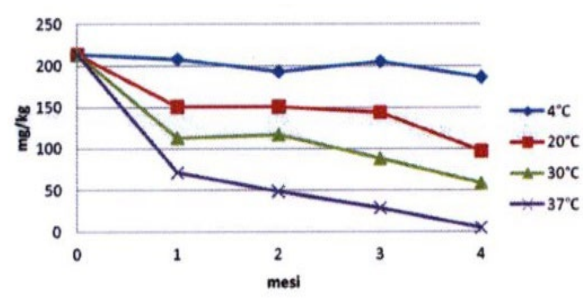
Pouch 1



Pouch 3



Pouch 2



Pouch 4

No data available for Pouch 5 as the amount of the indicator was not significant since the beginning

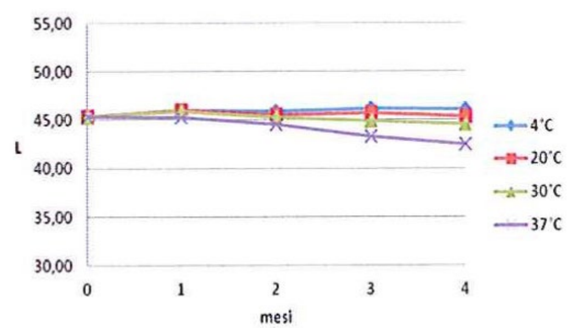
Table 4. The elaboration of the analytical data using extrapolation bring to the following evaluations. Concentration values are normalized on the reference value of Pouch 1

Sample ID	Vitamin C after 12 months at 4°C [mg/kg]	Vitamin C after 12 months at 25°C [mg/kg] (simulation)
Pouch 1	100	100
Pouch 2	83	73
Pouch 3	72	20
Pouch 4	81	5
Pouch 5	<1	<1

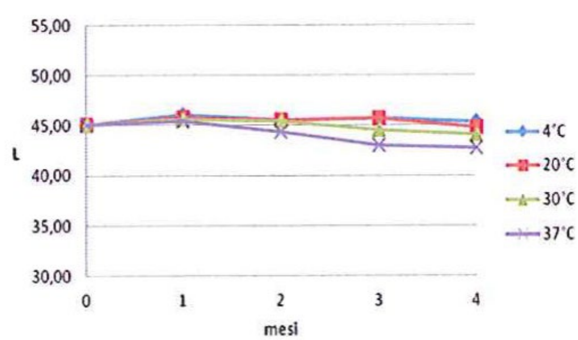
5.5 Colour Spectrum Measurements Analysis

The color variation of a food product is a clear indication of the degradation. Commonly a fruit puree, when degrading deviates its color towards browning. It has been measured with a spectrophotometer. The indicator is the L value that represent the variation of brightness and the colors.

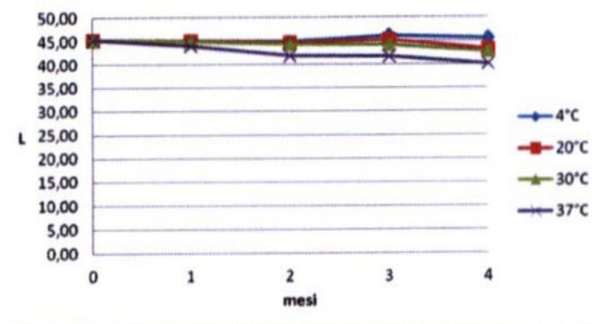
The following graphs represent the analytical results for the L value color variation:



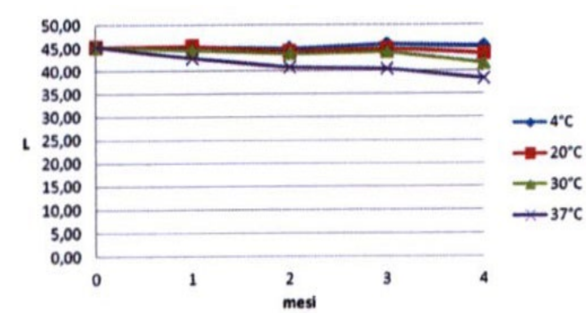
Pouch 1



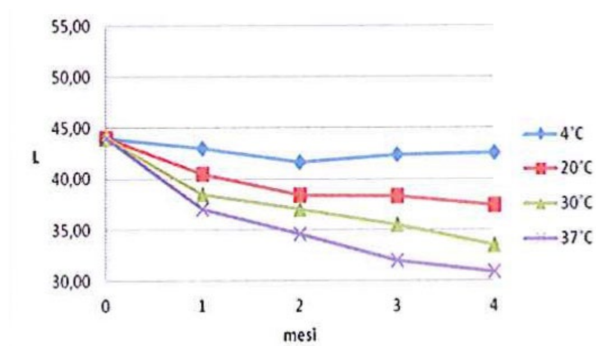
Pouch 2



Pouch 3



Pouch 4



Pouch 5

The elaboration of the analytical data using extrapolation bring to the following evaluations.

Table 5. Concentration values are normalized on the reference value of Pouch 1

Sample ID	L value after 12 months at 25°C (simulation)
Pouch 1	100
Pouch 2	98
Pouch 3	92
Pouch 4	92
Pouch 5	72

5.6 Synthesis: Impact of Storgae Conditions on Organoleptic Properties of Fruit Puree

Apple puree remaining at higher temperatures presents major degradation (high level of HMF & low level of C Vitamin) than the ones remain at lower temperatures whatever the packaging chosen.

Concerning HMF, every pouch with barrier film material has similar performances, allowing to target the standard shelf life of the product. In terms of C vitamin concentration, only the High performance coated PET (Pouch 2) has similar behaviour in regards to the reference. In all the other pouches the degradation of this value is significant.

Regarding Colour variation, no sensible differences among the tested film material have been measured. As a general consideration all the alternative film structures, in comparison with the reference aluminium foil structure, do not bring sensible differences in terms of aesthetic and visual characteristic to the product. On the other hand the organoleptic properties are affected.

The best alternative to the reference film composition is the High performance PET coated material.

CONCLUSION

Regarding HMF increase in fruit puree, results demonstrated that it is strongly correlated to storage temperature value whatever the type of package structures chosen.

This study shows as well that C vitamin decrease is proportional to storage conditions increase (temperature & duration) for all kind of packages.

Finally, the two last organoleptic characteristics (the brightness and the yellow color) remain stable in the measured storage conditions range whatever the type of oxygen barrier layer. This experimental study point out that the advances of material technology have paved the way for the use of barrier layers to enhance food safety and shelf life in flexible pouch packaging. Also it adds the possibility to give the packaging a whole new functionality for the final user like see-through pouch and new graphics.

Those results can be used as a reference by laminates process and development packaging engineers to support PE multi-layer film structure definition according to the targeted food application and shelf life conditions.

REFERENCES

[1] Akashi Kadoya, 1990. "*Food Packaging*", Takashi Kadoya, Academic Pres, Inc.

[2] ASTM 1995, *"Standard test method for oxygen transmission rate through plastic film and sheeting using a coulometric sensor designation D 3985-9"*. In Annual Book of ASTM Standards, American Society for Testing and Materials.

[3] Bras, J. 2004, *"Etude de Propriétes Barri*ères de D*é*rivé*s Cellulosiques. Application au gel de cellulose du papier sulfurisé"*, PhD thesis INP Toulouse, France.

[4] Brody, A. L.; Strupinsky, E. R.; Kline, L. R., 2001. *"Active packaging for food applications".* CRC Press: New York.

[5] CIS. www.cis-spa.com

[6] Crank, J., 1975. *"The mathematics of diffusion".* Second Edition; Clarendon Press: Oxford.

[7] Desobry, S.; Hardy, J., 1997. "*The increase of CO2 permeability of paper packaging with increasing hydration. "*, International Journal of Food Science & Technology 32, (5), 407-410.

[8] Di Luciano Piergiovanni,Sara Limbo, 2010, *"Food packaging: Materiali, tecnologie e soluzioni",* Springer Science & Business Media, 562 pages

[9] Han, J. H.; Scanlon, M. G., 2005. *"Innovation in Food Packaging"*, Han, J. H., Edition Elsevier.

[10] Jacques, C. H. M.; Hopfenberg, H. B.; Stannett, V. T., 1974. *"The permeability of plastic films and coatings to gas, vapors, and liquids".* Plenum Publishing Corp: New York and London, Vol. 6, p.73

[11] Robertson, G. L., 1993. *"Permeability of Thermoplastic Polymers. In Food Packaging: Principles and Practice"*, Dekker, M., Edition; p 73-110.

[12] Sina Ebnesajjad. 2013, *"Plastic Films in Food Packaging: Materials, Technology and Applications"* Sina Ebnesajjad., Edition Elsevier

[13] SSICA. www.ssica.it