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SLiM for young 2015
SHELF LIFE INTERNATIONAL MEETING

SLIM 2015

Shelf-life International Meeting

Vimercate (MB), 21 – 23 October 2015

Edited by

G. Buonocore, F. Licciardello and L. Piergiovanni

Special Issue

ITALIAN JOURNAL OF FOOD SCIENCE
Rivista italiana di scienza degli alimenti

CHIRIOTTI EDITORI

This Special Issue of the Italian Journal of Food Science collects the presentations given at the "SLIM 2015, Shelf Life International Meeting" organized by the Italian Scientific Group of Food Packaging (GSICA) and Italian Institute of Plastics (IIP), held at Vimercate (MB), Italy on October 21-23, 2012.
These papers were reviewed by the Scientific Committee of SLIM 2015 before their presentation but they did not undergo the conventional reviewing system of the Italian Journal of Food Science.

Chiriotti Editori S.A.S. - Pinerolo - Italy

ISSN 1120-1770

ITALIAN JOURNAL OF FOOD SCIENCE
(RIVISTA ITALIANA DI SCIENZA DEGLI ALIMENTI) 2nd series

Founded By Paolo Fantozzi under the aeges of the University of Perugia
Official Journal of the Italian Society of Food Science and Technology
Società Italiana di Scienze e Tecnologie Alimentari (S.I.S.T.Al)
Initially supported in part by the Italian Research Council (CNR) - Rome - Italy
Recognised as a "Journal of High Cultural Level"
by the Ministry of Cultural Heritage - Rome - Italy

Publisher:
Alberto Chiriotti - Chiriotti Editori srl, Viale Rimembranza 60, I-10064 Pinerolo, Italy - Tel. +39 0121 393127 - Fax +39 0121 794480 e-mail: alberto@chiriottieditori.it - URL: www.chiriottieditori.it

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These papers were reviewed by the Scientific Committee of SLIM 2015 before their presentation but they did not undergo the conventional reviewing system of the Italian Journal of Food Science. Therefore they will not be sent to JCR-ISI for IJFS Impact Factor evaluation.

Frequency:

Quarterly - One volume in four issues. Guide for Authors is published in each number and annual indices are published in number 4 of each volume.

Impact Factor: 0.504 published in 2016 Journal of Citation Reports, Institute for Scientific Information; Index Copernicus Journal Master List 2009 (ICV): 13.19
IJFS is abstracted/indexed in: Chemical Abstracts Service (USA); Foods Adlibra Publ. (USA); Gialine - Ensia (F); Institut Information Sci. Acad. Sciences (Russia); Institute for Scientific Information; CurrentContents®/AB&ES; SciSearch® (USA-GB); Int. Food Information Service - IFIS (D); Int. Food Information Service - IFIS (UK); EBSCO Publishing; Index Copernicus Journal Master List (PL).

IJFS has a publication charge of € 350.00 each article.
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INTRODUCTION

The present volume collects the contributions to the 7th Shelf Life International Meeting (SLIM 2015), which was held in Vimercate (MB), close to Milan, on October 21-23, 2015. The conference has been organized and hosted by the Italian Scientific Group of Food Packaging (GSICA), in collaboration with the Italian Institute of Plastics (IIP).

The International Meeting took place during the Expo Milano 2015, the Universal Exposition around the theme of "Feeding the Planet, Energy for Life". SLIM 2015 received the patronage of EXPO 2015, being recognized as a scientific event whose topics perfectly suited the aim of the Universal Exposition.

The Italian Scientific Group of Food Packaging (GSICA) has always paid particular attention to the career and work in the field of young scientists, to whom this special edition of SLIM was dedicated, being titled "SLIM for YOUNG 2015". Young scientists (below 40 years old) were strongly invited to contribute and to attend the meeting and they were involved as Chairmen/women of the sessions and main lecturerers.

The aim of SLIM 2012 has been always to provide an international forum for presenting current development works and future directions of research and applications on the topic of shelf life of packaged food products. Since prediction, testing and extension of shelf life require multi-disciplinary approaches, scientists and technologists from different areas were invited to attend the meeting contributing to the in-depth scientific discussion in an open and free atmosphere, following the SLIM tradition.

The Conference Proceedings, published once again with the support of Chiriotti Publisher as a special issue of the Italian Journal of Food Science, contain research reports presented as oral and poster papers during SLIM2015.
25 oral presentations and 51 posters were equally distributed among the 4 sessions: **New nano Materials**, **New Technologies**, **New Shelf Life Testing** and **New Shelf Life modelling**. Eight plenary presentations were provided by the young scientists invited as session's chairpersons.
The conference structure, the diversity of the attendees and the selected contributions from both industry and academia, contributed significantly to identifying the problems and promoting scientific discussions and further collaborations among researchers and stakeholders concerned with shelf life.

As it was officially announced within the final remarks of the conference, the eighth edition of SLIM will be held in Thailand, very likely in October 2017: the "Food Science and Technology" department of "Kasetsart University of Bangkok", in collaboration with the "Packaging and Materials Technology" department confirmed its commitment to organize the next edition. The local organizing committee (leaded by prof. Sasitorn Tongchitpakdee) is already working at the event, which promises to be one of the not-to-be-missed forthcoming scientific events. Further useful information will be disclosed soon.
Looking forward to meeting at SLIM17!

G. Buonocore, F. Licciardello and L. Piergiovanni

SESSION I

"Long Life, High Sustainability through New (Nano) Materials"

PACKAGING REDUCTION TO IMPROVE THE SUSTAINABILITY OF CARBONATED SOFT DRINKS

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ABSTRACT

The popularity of carbonated soft drinks is mainly due to the refreshing taste which, in turn, depends on their flavour and on the carbon dioxide content. The shelf life of carbonated soft drinks is primarily correlated with the retention of carbon dioxide inside the PET bottle, hence with its barrier properties, however, other quality parameters, such as the volatiles content, should be taken into account and monitored in order to guarantee the consumers with the highest quality at every stage of the product commercial life. Considering the high incidence of packaging material on the final product, both in terms of cost and of environmental impact, the reduction of the bottle thickness could play a significant role in the overall improvement of the sustainability in the industry of soft drinks. Sibat Tomarchio s.r.l. is committed with the improvement of sustainability of productions and has decided to evaluate lighter preforms for bottling two of its core-products: Aranciata, containing 12% juice from Sicilian oranges, and Verdello, with 17% juice from Sicilian lemons. Tests on the CO_2 retention performances are performed at every change (design, volume, material, etc.) occurring in the bottle, however this parameter has been considered as the only representative of the overall quality loss of carbonated soft drinks, while the aroma composition has not received sufficient attention. The research aimed at assessing alternative preforms to the one actually in use. One standard preform (clear, 34 g) was compared with two alternative ones (clear, 32 g and coloured, 32 g). During 6 months the samples were subjected to CO2 retention test, analysis of aroma profiles by HS-SPME-GC, and sensory analysis. Results demonstrate that it is possible to improve the sustainability of carbonated soft drinks by selecting lighter preforms, through shelf life studies based on the main quality parameters, supported by sensory analysis.

Keywords: PET, carbonation, aroma, shelf life, packaging reduction

1. INTRODUCTION

PET bottles are characterized by some permeability to CO_2, which affects the shelf life of the packaged carbonated soft drinks. Indeed, the bottle material performances, with special regards for barrier to CO_2, play a major role in the shelf-life extension. In order to guarantee the consumers with the original characteristics and quality of PET-packaged beverages, companies select appropriate preforms through the verification of the bottle CO_2 retention performances (CORIOLANI *et al*., 2006; LICCIARDELLO *et al*., 2011). This determination is what companies usually do routinely and each time they consider alternative preforms. Together with the CO_2 concentration in the beverage, the aroma composition is an important quality attribute that should be taken into account in order to guarantee the highest global quality during storage. The aim of the study was to evaluate the possibility to reduce the preform weight for 1.5-litre PET bottles without compromising the shelf life standards. Since packaging is the main hotspot for most environmental impacts in the carbonated soft drinks sector (AMIENYO *et al*., 2013; MANFREDI and VIGNALI, 2015), the reduction of PET weight would represent a significative improvement for the sustainability of such production.

2. MATERIALS AND METHODS

A comparative shelf life test was carried out during 6 months for two products, namely Aranciata (orange-based soft drink) and Verdello (lemon-based soft drink). Each product was bottled using three different preforms: a 34-gram clear preform, considered as control, a 32-gram coloured preform and a 32-gram uncoloured one. All PET preforms were supplied by Plasco s.p.a. (Anagni, FR, Italy) and formed into 1.5-litre bottles following a consolidated industrial process. Bottle forming and filling was carried out at Sibat Tomarchio s.r.l (Acireale, CT, Italy). The gas level at bottling was 6.7 g/l for Aranciata and 6.5 g/l for Verdello, and gas measurements were carried out at definite time intervals using an aphrometer (Alca Impianti s.r.l., Bolgare, BG, Italy) on three replicate bottles for each product and preform. Volatiles profiles by HS-SPME-GC-MS, using a CAR/PDMS fibre, and sensory parameters were determined on two replicate bottles for each product and preform.

3. RESULTS AND DISCUSSIONS

The CO_2 level (Fig. 1) revealed a constant decrease during storage time, this change being independent from the packaging type. Since the CO_2 level is regarded as the main quality parameter in carbonated beverages, it can be inferred that different preforms guaranteed the same shelf life standards, irrespective of weight. Two typical chromatograms, one for each of the two soft drinks object of the study, are reported (Figs. 2 and 3). Concerning the orange-based beverage, the volatile composition is characterized by the presence of limonene, which alone represents around 70% of the total compounds, followed by β-ocimene, terpinolene, α-terpineol, neryl acetate, geranyl acetate, β-caryophyllene, α-bergamotene and bisabolene. Minor amounts of aldehydes, esters, terpenes and sesquiterpenes also contribute to the formation of the overall aroma. Concerning the lemon-based beverage, limonene, g-terpinene and decanal prevail, followed by terpinolene, linalool, myrcene and minor amounts of other volatiles. Overall, the volatile composition of the two soft drinks did not change significantly during storage time.

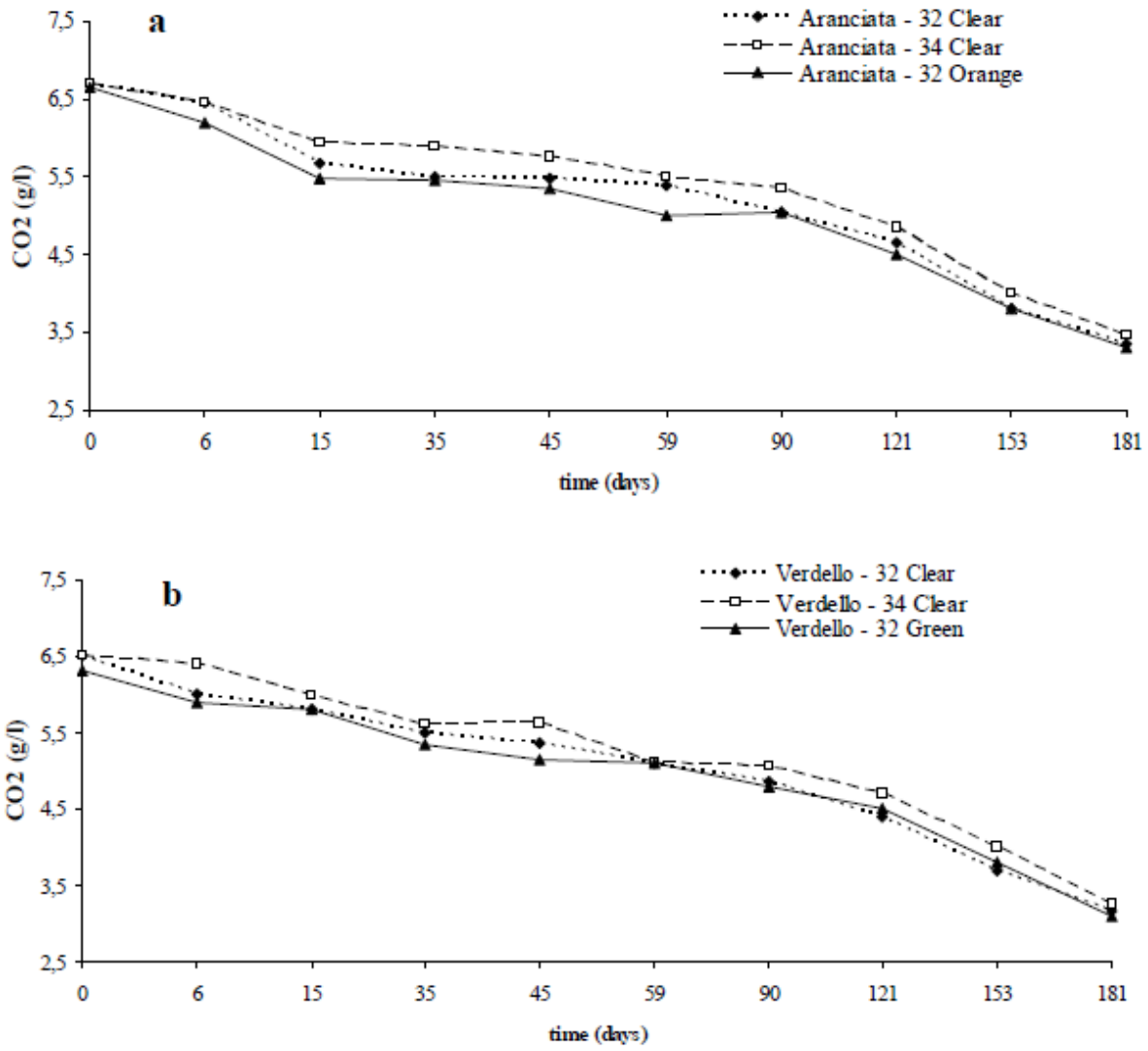

Figure 1: Variation of the CO_2 concentration in orange (a) and lemon-based (b) soft-drinks, as affected by preform and storage time.

Moreover, the volatile profiles among the three packaging types did not show significant differences for neither of the two beverages.

Sensory analysis demonstrated that the descriptors subjected to variation were those related with the CO_2 content: amount of bubbles and fizzy for the orange-based product, fizzy and off-flavour for the lemon-based one. Indeed, a reduction in the CO_2 level was registered, as expected, and this change was perceived by the judges.

Results for the CO_2 level, supported by data on the aroma composition and by sensory evaluation, demonstrate that it is possible to reduce the PET bottle weight by 2 grams without compromising the bottle performances. Being that packaging is among the most relevant environmental burdens for carbonated soft drinks (AMIENYO *et al.*, 2013; MANFREDI and VIGNALI, 2015), it can be concluded that the adoption of a lighter bottle

allows to reduce the environmental impact of packaging on the finished product, still guaranteeing the shelf life standards of the produce.

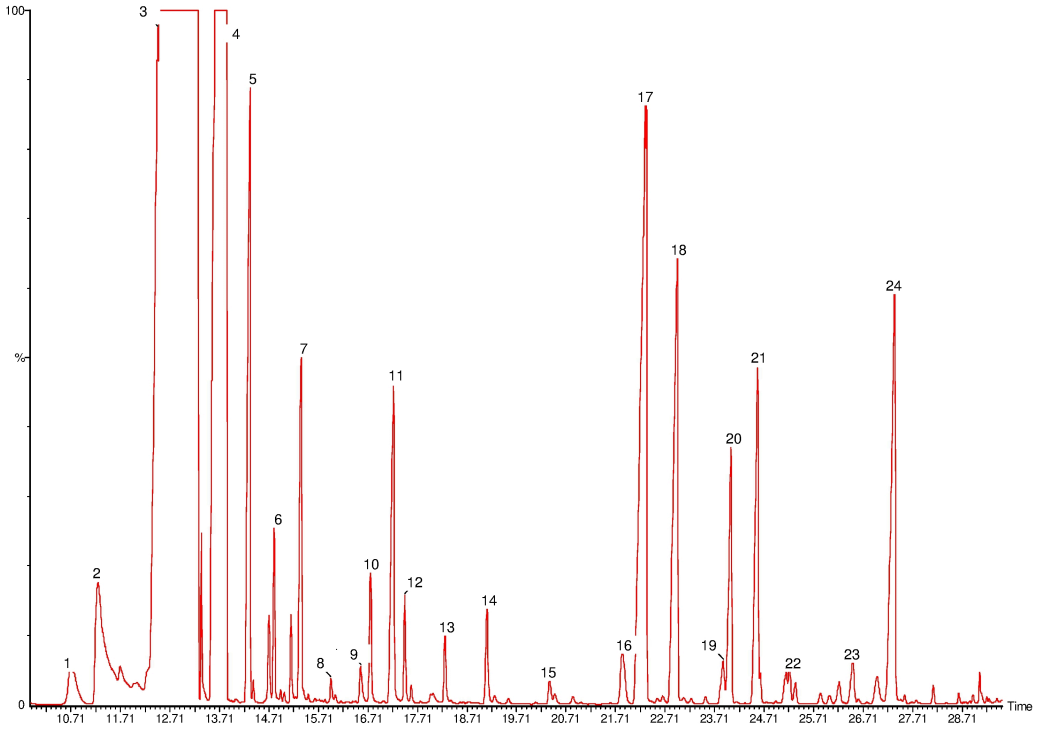

Figure 2: Typical chromatogram for an orange-based carbonated soft drink. 1: β-pinene; 2: myrcene; 3: limonene; 4: β-ocimene; 5: terpinolene; 6: nonanal; 7: methyl octanoate (internal std); 8: β-terpineol; 9: verbenol; 10: terpinen-4-ol; 11: α-terpineol; 12: decanal; 13: neral; 14: geranial; 15: undecanal; 16: citronellyl acetate; 17: neryl acetate; 18: geranyl acetate; 19: dodecanal; 20: β-caryophyllene; 21: α-bergamotene; 22: trans-β-farnesene; 23: valencene; 24: bisabolene.

Table 1: Mean scores of the significant sensory attributes for the orange-based beverage. Values marked with different letters in the same row are significantly different ($p \leq 0.05$) according to the LSD multiple comparison test.

		Days of storage					
Attributes	**Samples**	**1**	**7**	**15**	**30**	**90**	**180**
Amount of bubbles	32 Green	7.0^{b}	4.3^{a}	5.4^{ab}	4.5^{a}	5.1^{a}	4.7^{a}
Fizzy		5.5^{b}	2.9^{a}	3.8^{a}	2.5^{a}	3.2^{a}	2.3^{a}
Amount of bubbles	32 Clear	7.0^{b}	4.6^{a}	5.1^{a}	4.9^{a}	5.6^{ab}	4.5^{a}
Fizzy		5.5^{b}	4.0^{ab}	3.6^{a}	3.0^{a}	2.5^{a}	2.5^{a}
Amount of bubbles	Control	7.0^{b}	4.3^{a}	5.5^{ab}	5.0^{a}	5.9^{ab}	5.0^{a}
Fizzy		5.5^{b}	3.2^{a}	3.5^{a}	2.5^{a}	2.4^{a}	3.1^{a}

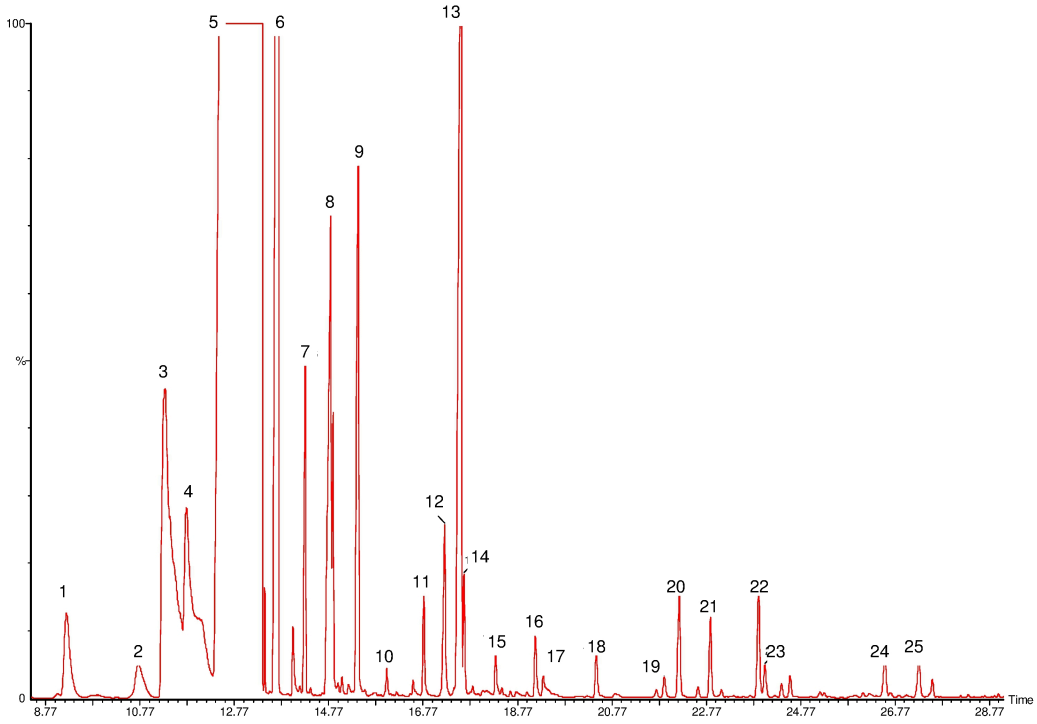

Figure 3: Typical chromatogram for a lemon-based carbonated soft drink. 1: phellandrene; 2: β-Pinene; 3: myrcene; 4: octanal; 5: limonene; 6: γ-terpinene; 7: terpinolene; 8: linalool; 9: methyl octanoate (internal std); 10: β-terpineol; 11: terpinen-4-ol; 12: *α*-terpineol; 13: decanal; 14: octyl acetate; 15: neral; 16: geranial; 17: peryllaldehyde; 18: undecanal; 19: citronellyl acetate; 20: neryl acetate; 21: geranyl acetate; 22: dodecanal; 23: β-caryophyllene; 24: valencene; 24: bisabolene.

Table 2: Mean scores of the significant sensory attributes for the lemon-based beverage. Values marked with different letters in the same row are significantly different ($p \leq 0.05$) according to the LSD multiple comparison test.

		Days of storage					
Attributes	**Samples**	**1**	**7**	**15**	**30**	**90**	**180**
Fizzy	32 Green	5.8^{c}	4.0^{ab}	5.1^{bc}	3.6^{ab}	4.0^{ab}	2.5^{a}
Off-flavour		3.4^{bc}	2.8^{abc}	1.7^{a}	2.2^{ab}	1.4^{a}	4.0^{c}
Fizzy	32 Clear	5.8^{c}	4.2^{b}	4.3^{bc}	3.5^{ab}	3.8^{b}	2.2^{a}
Off-flavour		3.4^{b}	2.7^{ab}	1.7^{a}	2.0^{ab}	2.3^{ab}	3.5^{b}
Fizzy	Control	5.8^{b}	4.4^{ab}	4.5^{ab}	3.5^{a}	3.8^{a}	3.0^{a}
Off-flavour		3.4^{b}	2.5^{ab}	1.9^{a}	2.1^{ab}	1.9^{a}	3.2^{ab}

4. CONCLUSIONS

A comparative shelf life test on two carbonated beverages performed with different preforms demonstrated that the PET bottle weight can be reduced by 2 grams without affecting the shelf life standards of the produce, neither in terms of CO_2 retention, nor volatile composition. Studies aimed at assessing alternative bottles are crucial for the improvement of sustainability of carbonated beverages, since packaging represents the main factor affecting environmental impact in this specific industrial sector.

ACKNOWLEDGEMENTS

This research was supported by Ministero dell'Istruzione, dell'Università e della Ricerca (Prot. 957/ric , 28/12/2012), through the Project 2012ZN3KJL "Long Life, High Sustainability".

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SHELF LIFE EVALUATION OF FRESH-CUT GLOBE ARTICHOKE PACKAGED IN A COMPOSTABLE BIOBASED FILM

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ABSTRACT

Globe artichoke is an important component in the Mediterranean diet, but its complexity of preparation and susceptibility to browning degree limit its consumption on a wider scale. Therefore, the aim of the present study was to propose on the one hand, a suitable processing to obtain fresh-cut globe artichoke heads using active compounds to delay browning and, on the other hand, the best packaging solution to reduce water loss. Ascorbic and citric acids were used asanti-browning agents and two different packaging solutions were tested. Heads were divided into homogeneous lots of 50 heads, and after the processing slices were placed in PET trays and packed in ordinary atmosphere using a Cast Polypropylene film and NatureFlex™ compostable BIObased film. Bags were hermetically sealed and stored at 4 °C for 14 days. Analysis of weight losses, texture, respiration rate, colour degradation, polyphenol and ascorbic acid content and microbiological analysis were performed. Our results highlighted as cv. Spinoso sardo proved to have suitable qualitative characteristics for industrial processing; both packaging films used ensured a prolonging of shelf life that seems to be enough for produce distribution to the local markets.

Keywords: biodegradable, browning, colour degradation, compostable, respiration rate

1. INTRODUCTION

Globe artichoke [*Cynaracarduncul*us var. *scolymus* (L.) Fiori] is one of the most important vegetable crops in the Mediterranean Basin, with an annual production of ~1,4 Mt of heads (FAOSTAT, 2013). Among the most commonly grown traditional cultivars, Spinoso sardo is greatly appreciated for fresh consumption due to its pleasant and mild sensory attributes. However, the presence of long sharp spines on bracts and leaves limits its consumption on a wider scale. This boasts the development of new products, such as minimally processed ones, which could increase the level of Spinoso sardo consumption thanks to their ease of preparation and convenience. One of the main problem with fresh-cut globe artichoke heads is the high browning rate of the cut surfaces (receptacle and bracts) caused by oxidation; therefore, different chemical compounds were tested to overcome this problem, together with different packaging solutions. Weight loss is another phenomenon that negatively influences globe artichoke marketability (CAMPUS *et al*., 2006); this may be attributed to respiration and other senescence-related metabolic processes during storage. The aim of this study is to propose a suitable processing to obtain fresh-cut globe artichoke heads using active compounds, to delay browning and to select the best packaging solution to defeat water loss.

2. MATERIALS AND METHODS

Experimental field trial was conducted during the 2014–2015 growing season in a farm located in Cassibile in the Siracusa Plain (Sicily, Italy). Spinoso sardo, an early maturing cultivar that is normally harvested between November and April, was harvested during March-April2015 at marketable stage (MAUROMICALE and IERNA, 2000). Homogeneous lots of 50 heads were prepared by removing the outer bracts and cutting the floral stems to 5 cm in length and the top of internal bracts. Then, they were sliced, washed in chlorinated water, immersed for 5 min in the anti-browning solution (0,5 and 2% citric and ascorbic acid), dried in a manual centrifuge. About ten slices were placed in PET trays and packed in ordinary atmosphere using a Cast Polypropylene film (OTR 3000 cc/m^2/24h, 23°C 0% RH) and a compostable BIObased film (OTR 55 cc/m^2/24h, 8°C 70% RH) kindly provided respectively by Rotocalco Mediterranea (Siracusa, Italy) and InnoviaFilms (Novara, Italy), identified throughout the manuscript as SS_C and SS_BIO, respectively. Bags were hermetically sealed, and stored at 4 ± 2°C for 14 days. Analysis of weight losses, texture using a ZwickRoell z 0,5 (Zwick GmbH & Co. KG, Ulm, Germany), respiration rate through a Dansensor A/S Checkpoint (Ringsted, Denmark), colour degradation (Handycolorimeter NR 3000; Nippondenshokuind. co. ltd) and microbiological analysis were performed. Total polyphenol content (TPC) was measured by Folin–Ciocalteu assay (SINGLETON &ROSSI, 1965) reading the absorbance at 760 nm using a Shimadzu 1601UV–visible spectrophotometer (Tokyo, Japan). The ascorbic acid (AAC) was determined according to the method proposed by RESTUCCIA *et al*. (2014). All the reagents and solvents, of analytical or high-performance liquid chromatography grade, were purchased from Sigma-Aldrich (Milan, Italy). All the chemical analyses were performed in duplicate.

3. RESULTS

As expected, weight loss infresh-cut heads of *cv*. Spinoso sardo was higher in samples packed in the BIObased film than in samples packed in PP cast bags (Fig. 1). In fact, the

compostable polymeric film shows a high water vapor transpiration rate (200 g/m^2.24 hrs, 25°C 75% RH). With reference tothe texture (shear tests were performed on 6 different slices), the compostable BIObased film showed the best performance even if with a high standard deviation among samples (Fig. 2).

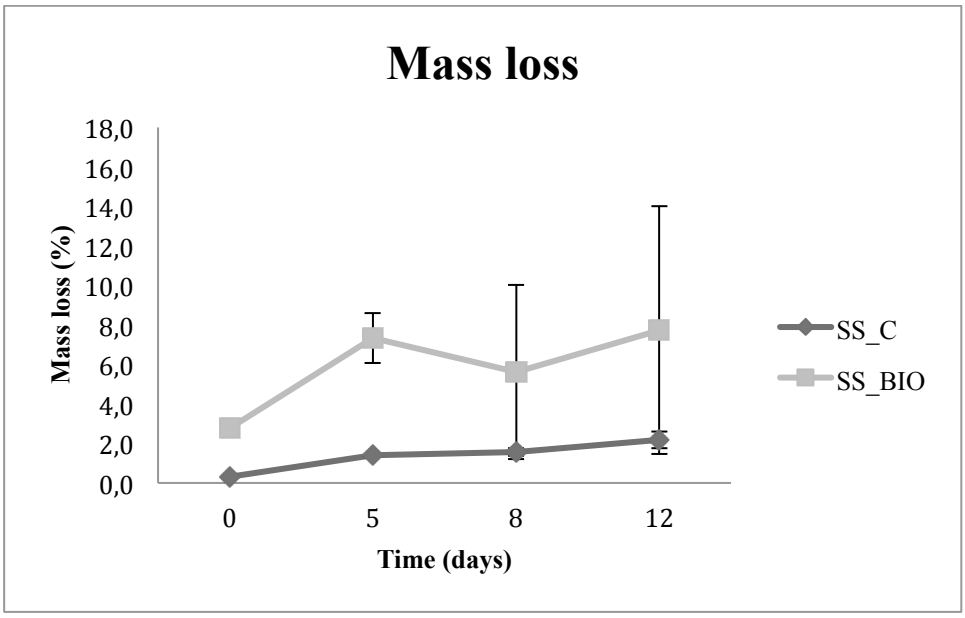

Figure 1: Effect of packaging film on the weight loss (%) of minimally processed globe artichoke heads during cold storage.

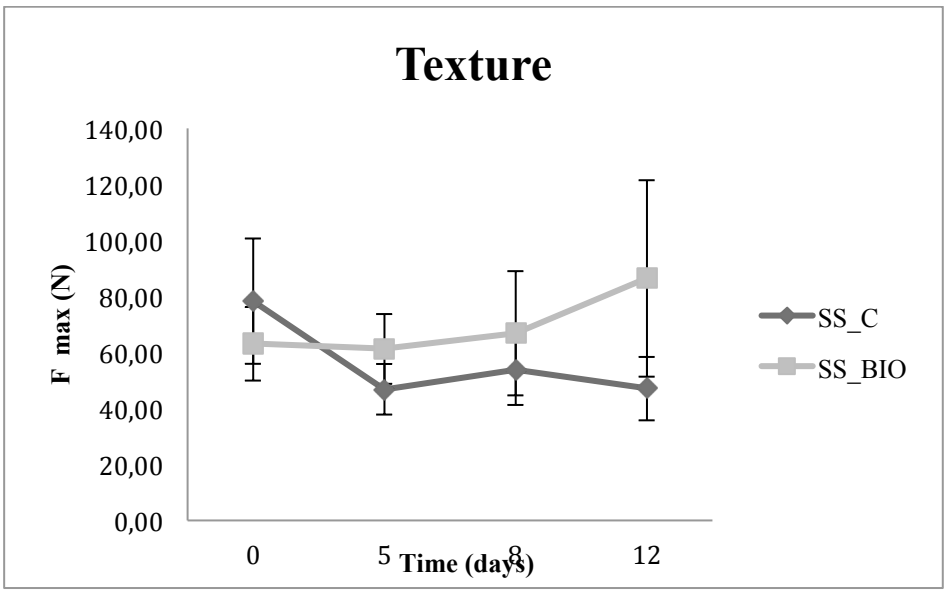

Figure 2: Effect of packaging film on the texture (N) of minimally processed globe artichoke heads during cold storage.

There were not differences between the packaging films for the colorimetric parameters (data not shown). Also for the respiration rate, both analyzed gases (oxygen and carbon dioxide) did not show significant differences between the packaging films (Figs. 3 and 4).

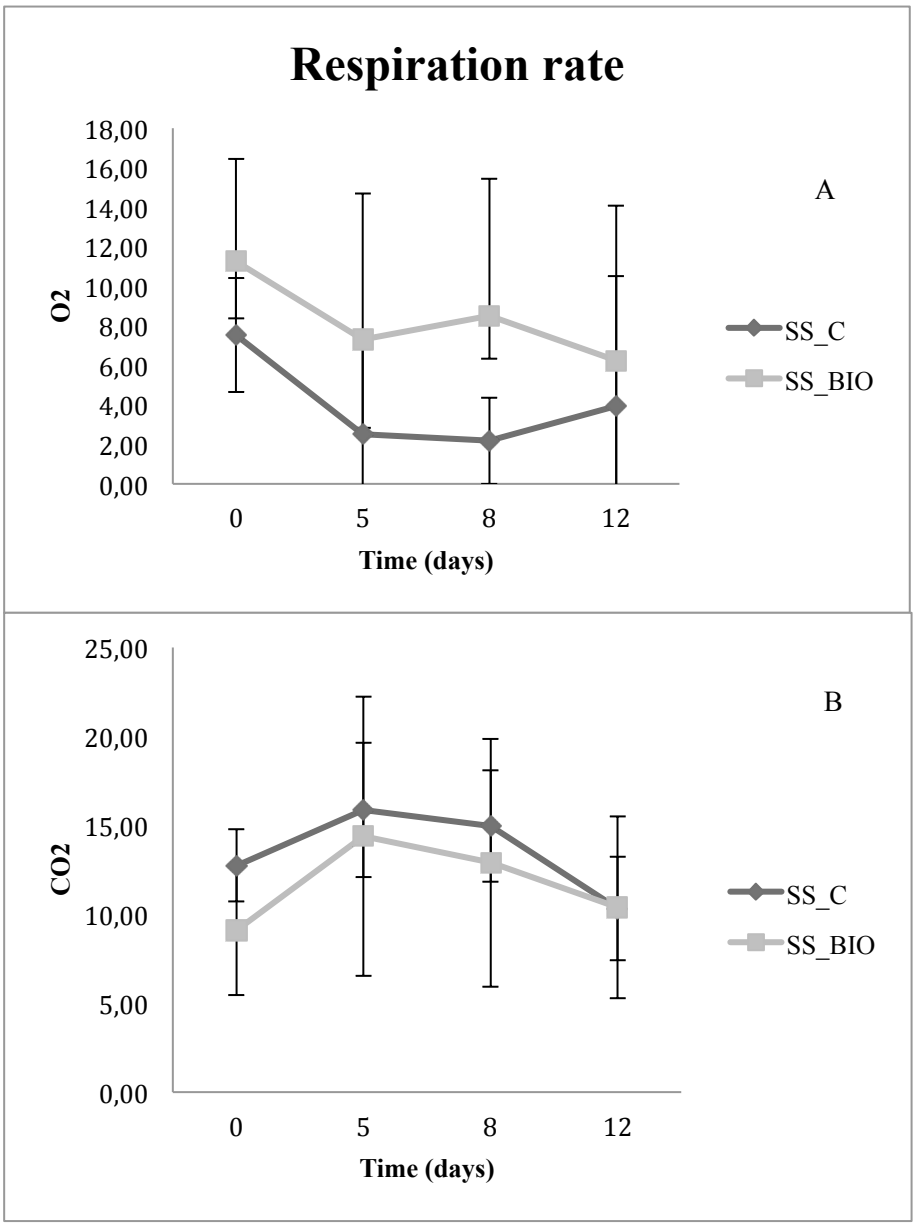

Figure 3: Respiration rate of minimally processed globe artichoke heads during cold storage, as effected by packaging film. Oxygen rate (A); Carbon dioxide rate (B).

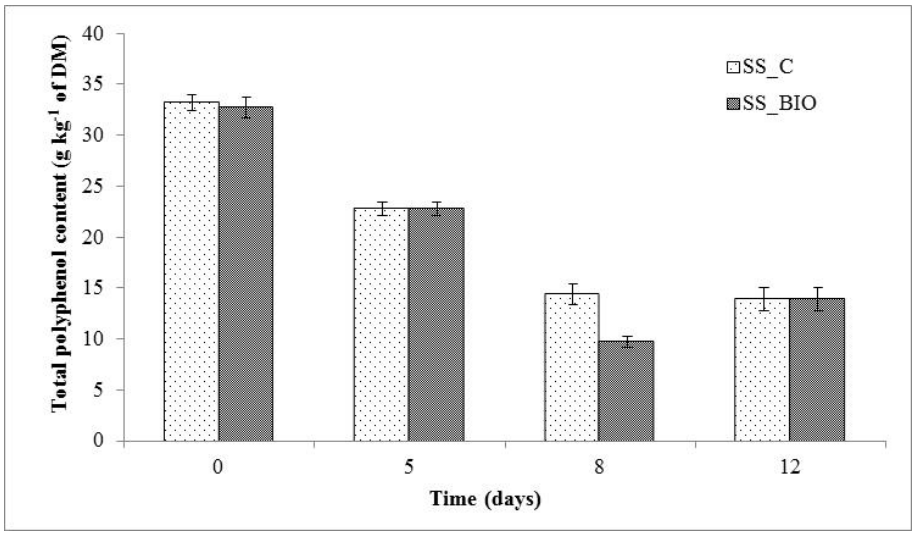

Figure 4: Effect of packaging film on the total polyphenols content of minimally processed globe artichoke heads during cold storage.

At each sampling time, no differences were observed between the packaging films under study for both TPC and AAC (Figs. 5 and 6). In particular, the TPC significantly decreased throughout the storage time up to 13.9 g/kg of DM for both packaging films, as observed previously by RICCI *et al*. (2013) due to an increase in electrolytic leakage during cold storage. Analogously, AAC significantly decreased from the processing day up to 12 days of cold storage. This phenomenon may be attributable to AA conversion into dehydroascorbic acid (DHAA), which is less stable than AA (DAVEY *et al*., 2000). With reference to microbiological counts, all the microbial groups gradually increased throughout the refrigerated storage.

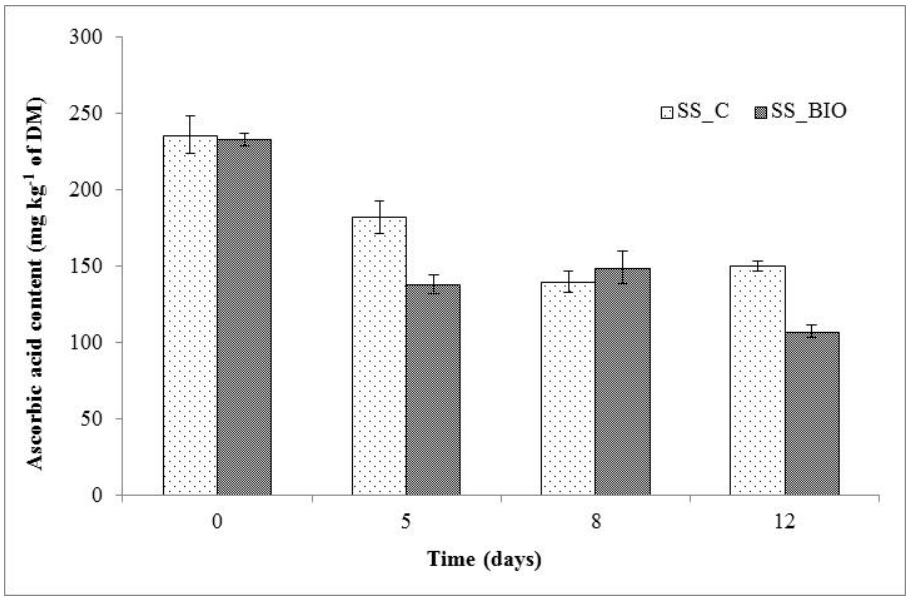

Figure 5: Effect of packaging film on the ascorbic acid content of minimally processed globe artichoke heads during cold storage.

The different films used for packaging did not affect the microbial growth, except for mesophilic bacteria and yeasts and moulds whose count increase was slightly reduced by the BIObased film.

4. CONCLUSIONS

Our results demonstrated as *cv*. Spinoso sardo is suitable for industrial processing, due to its physiological, nutritional, sensory and microbiological traits of ready-to-use heads. Despite the results, we think that the opportunity to use a compostable BIObased film is interesting in order to reduce wastes and the environmental impact.

ACKNOWLEDGEMENTS

This work was financially supported by the project "SHELF-LIFE - Integrated use of innovative technological approaches to improve the shelf-life and preserve the nutritional properties of food products" carried out by the Cluster Sicily Agrobio and Fishing Industry and funded by the Research Fund PON R&C 2007-2013, DD 713/Ric. (PON02_00451_3361909).

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STRATEGIES FOR IMPLEMENTING NANO-CELLULOSE COATINGS IN FLEXIBLE PACKAGING

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ABSTRACT

The coatings of cellulose nanocrystals (CNCs) on different common plastic films have proven to be an excellent barrier to oxygen permeation, able to make feasible a significant reduction in the final thickness of flexible packaging materials, adding a very thin layer of a bio-based material. The main hurdles to a possible application of such a new technology, however, is still represented by the low water vapor resistance and the lack of thermoplastic properties.
In this work, a thin PET film (23 μm), has been coated by a CNCs suspension (8% in water w/v) obtaining a final coating thickness of less than 700 nm. The coated PET films have been laminated, by water and solvent based polyurethanes adhesives to both CPP and LDPE films. The laminated structures obtained were subsequently tested for their resistance to delamination, sealable properties, and oxygen permeability, following ASTM standards procedures. The results obtained demonstrated the feasibility of such novel multilayer structures in high demanding food packaging.

Keywords: Adhesive lamination, cellulose nanocrystals, flexible packaging, mechanical properties, oxygen permeability

1. INTRODUCTION

Cellulose, a crystalline β-1,4-glucan, is the world's most abundant biopolymer. Cellulose nanocrystals (CNCs), obtained by Ammonium Persulphate treatment from cotton linters, show peculiars chemical/physical characteristics as the high cristallinity and the high content of carboxyl groups onto the surface (LEUNG *et al*., 2011). Thanks to these properties it can be possible to use CNCs as coating onto common plastic film for food packaging application, because the CNCs coated plastic films show excellent oxygen barrier, at very low thickness (700 nm). The main hurdles to a possibile application of such a new technology for food packaging are represented by the low water vapor resistance and the lack of thermoplastic properties(LI *et al*., 2013).
The chances of using CNCs coated plastic films seem streactly linked to the possibilities of laminating them to a polyolefinic layer, in order to obtain a final structure, sealable and with a good barrier both to oxygen and water vapour, thus able to compete in the market as a high performing flexible packaging material.
The aim of this study was to evaluate the feasibility of novel multilayer structures composed by PET film, CNCs coating, polyuretane adhesive (solvent and water based), polyolefin film with final high gas and vapour barrier, for high demanding food packaging applications and as a possible route for an increase of more sustainable flexible packaging materials.

2. MATERIALS AND METHODS

2.1. Materials

The cotton linters used as raw materials to produce CNCs were kindly supplied by INNOVHUB SSI (Milan); Ammonium Persulphate ≥98% was purchased from Sigma-Aldrich (Milan); polyethylene terephthalate PET film 12±0,5 μm thick, co-extruded polypropylene (CPP) film, 50±1 μm thick, low density polyethylene LDPE film 100 μm tick, waterborne polyurethane adhesive (PUD), waterborne polyurethane adhesive with hydro dispersible hardener (PUD_HH) and solvent base polyurethane adhesive (PBS) were supplied by Sapici S.p.a. (Cernusco sul Naviglio, Milan).

2.2. Preparation of CNCs coating and lamination process

CNCs were extracted from cotton linters by Ammonium Persulphate treatment as reported elsewhere (LEUNG *et al*., 2011). A 8% wt CNC water dispersion (pH 8) was coated onto a PET 12 μm corona treated (Arcotech GmbH, Monsheim, Germany) by an automatic applicator (model 1137, Sheen Instruments, Kingston, UK) according to ASTM D823-07, practice C. The same ASTM standard was used to perform the lamination process.

2.3. Oxygen permeability

The oxygen permeability (PO_2) of multilayer films was assessed by an isostatic method (Multiperm, Extra_Solution S.r.l., Capannori, Lucca) at 23°C and two different relative humidity values (0% and 50% RH), complying with ASTM D-3985.

2.4. Mechanical test

Peel resistance (t-peel test) and seal strenght of multilayer films were performed using ASTM D1876 and ASTM F 88M-09 standards respectly.

3. RESULTS AND CONCLUSIONS

3.1. Oxygen permeability

The permeability values shown in Fig. 1 demonstrates a general barrier effect provided by the CNCs, to indicate a good interaction between CNCs and adhesives (XU *et al.*, 2013).

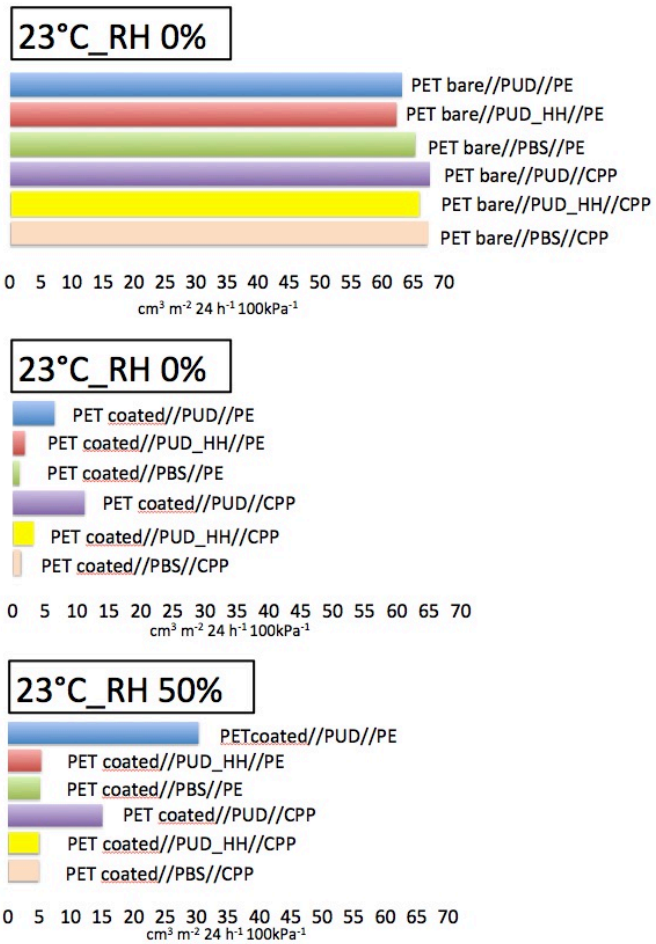

Figure 1: Oxygen permeability of multilayer films.

The laminates assembled using solvent-based (PBS) and water-based adhesives with the addition of isocyanates (PUD_HH), shown interesting barrier effects, for the two polyolefins, both at 0% RH and 50% RH. The oxygen permeability of multilayer film assembled with PET CNCs coated, PBS and CPP is 1,278 ± 0,02 cm^3 m^{-2} day^{-1} 100 kPa^{-1} at 0% RH and 4,91 ± 0,10 cm^3 m^{-2} day^{-1} 100 kPa^{-1} at 50% RH, whilst permeability of multilayer film, assembled with PET CNCs coated, PUD_HH and CPP is 3,15 ± 0,15 cm^3 m^{-2} day^{-1} 100 kPa^{-1} at

0% RH and 5,01 ± 0,08 cm^3 m^{-2} day^{-1} 100 kPa^{-1} at 50% RH. These values are much lower than oxygen permeability value of the same multilayer structure without CNCs coating, which is 67 cm^3 m^{-2} day^{-1} 100 kPa^{-1} with either PBS and PUD_HH as the adhesive in the structures. In contrast, the use of water-based adhesive alone seems not enough effective to obtain a good barrier properties, as demostrated by the results obtained: 11,2 ± 0,31 and 15 ± 0,53 cm^3 m^{-2} day^{-1} 100 kPa^{-1} at 0% and 50%RH with CPP as the polyolefin.

3.2. Mechanical test

The peel resistance values of multilayer films are shown in Table 1. Delamination occured in presence of the waterborne PUD adhesive onto CPP and, in some cases, on PE. On the contrary, the coupling of PET coated with CNCs, throught PBS and waterborne PUD_HH adhesives, with both polyolefin achieved no delamination (No Del. in Table 1), due to the good affinity between crystals and adhesive. FTIR analysis shows that CNCs coating, after delamination, remains on polyolefin side, due to the affinity between CNCs and adhesive.

Table 1: Peel resistance values of multilayer films.

Final structure		**Delamination (N/15mm)**		
	Adhesive Type		**Days of storage**	
		1	**3**	**6**
	Waterborne PUD	0.5	0.5	0.5
PET Coat/CPP	Waterborne PUD_HH	No del.	No del.	No del.
	PBS	No del.	No del.	No del.
	Waterborne PUD	0.5	1.0	0.9
PET/CPP	Waterborne PUD_HH	No del.	No del.	No del.
	PBS	No del.	No del.	No del.
	Waterborne PUD	0.5	No del.	No del.
PET Coat/PE	Waterborne PUD_HH	No del.	No del.	No del.
	PBS	No del.	No del.	No del.
	Waterborne PUD	0.5	No del.	No del.
PET/PE	Waterborne PUD_HH	No del.	No del.	No del.
	PBS	No del.	No del.	No del.

The seal strenght values of multilayer films presented in Table 2 show good sealability of the samples even in presence of CNCs. The laminates containing waterborne adhesives PUB showed the lowest value of heat seal strenght in accordance with the lamination results. The seal strenght of CPP is always so high that the polymer break or the delamination happens before the seal break.

Table 2: Seal strenght values of multilayer film.

Lamynate types	Heat-seal strength (N/mm^2)
PET bare // PUD_HH // PE	0.90±0.21
PET coated CNCS // PUD_HH // PE	1.85±0.32
PET bare // PBS // PE	9.38±2.25
PET coated CNCS // PBS // PE	2.47±1.18
Laminates with CCP	No break of seal

The results presented in this work show the possibilities of laminating a polyester CNC coated film to a poliolefinic layer with different adhesive types. The properties of the laminates are correlated to the affinity between polymer, cellulose nanocrystals and adhesive. The best results in terms of oxygen barrier properties and mechanical properties were obtained with the combination PET coated CNCs, PBS, PE or CPP. These preliminary results need to be further investigated to understand the physico-chemical correlations between the involved matrices in the final structure of the laminated films.

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SESSION II

"Long Life, High Sustainability through New Technologies"

PRE-SELECTION ON SKIM MILK OF DAIRY LACTIC ACID BACTERIA TO IMPROVE ANTIOXIDANT ACTIVITY AND SHELF LIFE OF CHEESES

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ABSTRACT

It has been shown that some lactic acid bacteria (LAB) possess antioxidant activity. The using of lactic acid bacteria to increase the antioxidant activity in cheese is a good way to improve its shelf life. Consequently, the aim of this work was to perform the clonal isolation, identification and selection of lactic acid bacteria to produce compounds with the property to improve the antioxidant activity and the shelf life of cheeses. Seventy-three strains of lactic acid bacteria, were tested on skim milk for their pH and antioxidant activity. Each strain was first precultured in MRS broth (lactobacilli) or in M17 broth (coccal-shaped) at 37°C for 24 h. These cultures were then used in inoculation (1%, v/v) of 15 mL of skim milk treated at 121°C for 5 minutes. Each strain was inoculated in triplicate for 44 h at 37°C. Then the pH was determined and the antioxidant activity was measured by analysing the radical scavenging activity using a spectrophotometric decolourization assay (ABTS) according to Virtanen *et al.* (2007). In addition, also DPPH assay was performed. The pH values have shown mean value of 4.88, with a minimum of 3.49 and a maximum of 5.89, while ABTS values (expressed as % of inhibition) mean value of 51.56, with a minimum of 24.40 and a maximum of 71.52. These results confirm that the development of antioxidant activity is a strain-specific characteristic. In detail, the tested strains exhibit a high degree of biodiversity.

Keywords: antioxidant activity; cheese; lactic acid bacteria (LAB); shelf life; skim milk

1. INTRODUCTION

Lactobacillus species are probably the most important bacteria in the food industry. They are widely used as starter cultures and have been reported to play significant roles in the production of fermented foods like cheeses (OSUNTOKI and KORI, 2010). Extension shelf life of cheeses by addition of Lactobacillus cultures is also reported (JALILZADEH *et al*., 2015).
Some LAB strains have been found with important biological functions, such as anti-ageing and antioxidant activities and they were investigated for their in vitro scavenging activity against radicals (DPPH and ABTS) as reported by LI *et al.* (2012).

2. MATERIALS AND METHODS

2.1. Preparation of LAB culture sand preparation of whey fraction

The strains (seventy-three) of Lactic acid bacteria (LAB) used in this study, isolated from dairy products during different years, were precultured in MRS (Lactobacilli) and M17 broth (coccal-shaped) at 37°C for 24 h and then inoculated as described by VIRTANEN *et al.* (2007). These strains of LAB were used to inoculate (1 % v/v at 37°C for 44 h) 15 mL of skim milk treated at 121°C for 5 minutes. The whey fraction was treated to remove nonhydrolyzed casein by acidification (with 1 M HCl) up a pH of 4.60 and centrifugation of suspension at 10000 x*g* for 20 min at 5°C (eppendorf 5804 R centrifuge). The supernatant was filtered on a 0.45-μm filter (Chromafil RC-45/25, Macherey-Nagel).

Antioxidant activity of the isolates

The antioxidant activity was assessed on whey fraction with ABTS and DPPH assays as described by RE *et al.* (1999) and by Son and Lewis (2002) respectively. ABTS radical cation ($ABTS^{\cdot\cdot}$) was produced by reacting ABTS stock solution with potassium persulfate in the dark at room temperature for 12-16 h before use. The $ABTS^{\cdot\cdot}$ solution (7 mM) was diluted with ethanol to an absorbance of 0.70 at 734 nm and equilibrated at 30°C. For the measurement of antioxidant activity, 2.90 mL of diluted ABTS radical solution was added to 100 μL of sample in water and the mixture was shaken for 10 s at 30°C and the absorbance was measured at 734 nm (30°C) 1 min after initial mixing and up to 6 min. Appropriate solvent blanks were run in each assay. All sample are evaluated in triplicate and the antioxidant activity was expressed as percentage inhibition of absorbance at 734 nm.
DPPH assay is based on the decoloration of the solution resulting from the reaction between DPPH radical and antioxidant compound. 2.95 mL of DPPH in ethanol (500 mM) was added to 50 μL of the whey fraction and the absorbance at 517 nm, was measured 1 min after initial mixing and up to 30 min. All sample were evaluated in triplicate and the antioxidant activity was expressed as percentage inhibition of absorbance at 517 nm. Results are shown as the mean values ± standard deviation. Pearson's correlation coefficients were determined to evaluate correlation between antioxidant activity and acidifying power.

3. RESULTS AND CONCLUSIONS

Results has evidenced different pH values among strains of LAB: they were divided into five pH ranges on needs of cheesemaking operations. The pH range of 3.50-4.50 where the LAB had the highest acidifying power, in particular for the n.122 strain (*Lactococcus*) with a pH value of 3.49; 4.50-4.75 where the LAB has shown values next to isoelettric point of casein, 4.75-5.00 with suitable pH values for a correct stretching of curd, 5.00-5.50 and 5.50-6.00 with the LAB with the lowest acidifying power. Significant differences for one-way ANOVA ($P < 0.01$) were found among single strain of LAB in each pH ranges as shown in Table 2 (only results about the first three pH ranges are shown). Antioxidant activity of whey fraction inoculated with strains of LAB that had a highest acidifying power (3.5-4.5 pH ranges) is showed in Table 1. In this pH ranges an inhibition rate of 37.23-71.52% was found as exhibited by different letters by Tukey's test. In particular, the variation among different LAB was about a rate of 37.23-67.72% (ABTS assay) and 1.33-9.51% (DPPH assay) for *Lactobacillus* strains, 53.11-71.52% (ABTS assay) 5.24-11.46% (DPPH assay) for *Lactococcus* strains and 45.48-50.83% (ABTS assay) and 2.69-2.89% (DPPH assay) for *Enterococcus* strains.

Table 1: Acidifying power of LAB inoculated on whey fraction. Each number identifies the LAB species distributed into five pH ranges.
Values are Means ± Standard Deviation, n=3.
*Significance at P<0.05;**Significance at P<0.01; n.s. not significant. Data followed by different letters are significantly different by Tukey's multiple range test.

pH 3.50-4.50			**pH 4.50-4.75**			**pH 4.75-5.00**		
1	4.32±0.01	ab	4	4.67±0.04	ab	2	4.80±0.06	cde
3	4.42±0.07	a	9	4.55±0.05	cd	5	4.78±0.03	de
6	4.04±0.02	de	10	4.65±0.01	abc	7	4.82±0.03	abcde
34	4.37±0.05	ab	11	4.56±0.03	cd	8	4.82±0.04	abcde
35	4.39±0.04	ab	32	4.58±0.04	bcd	12	4.96±0.05	ab
69	4.39±0.08	ab	42	4.75±0.06	a	13	4.91±0.06	abcd
122	3.49±0.05	g	43	4.53±0.02	d	14	4.91±0.06	abcd
123	4.07±0.02	cde	158	4.65±0.04	abc	15	4.74±0.02	e
127	3.81±0.02	f	-	-		18	4.89±0.04	abcd
145	4.23±0.08	bc	-	-		21	4.94±0.05	abc
146	4.11±0.02	cd	-	-		33	4.77±0.01	de
147	4.42±0.04	a	-	-		36	4.95±0.07	ab
148	4.03±0.02	de	-	-		38	4.82±0.07	bcde
151	4.47±0.09	a	-	-		40	4.89±0.03	abcde
153	3.91±0.14	ef	-	-		45	4.97±0.06	a
156	4.00±0.01	de	-	-		160	4.80±0.03	cde
157	3.90±0.04	ef	-	-		-	-	
163	4.11±0.06	cd	-	-		-	-	
Sig.	**			**			**	

Table 2: Antioxidant activity of whey fraction with a ph ranges of 3.5-4.5 after LAB fermentation.
Values are Means ± Standard Deviation, n=3. *Significance at P<0.05;**Significance at P<0.01; n.s. not significant. Data followed by different letters are significantly different by Tukey's multiple range test.

Lactic acid bacteria	Identify	pH	ABTS (% inhibition)*	DPPH (% inhibition) **
1	Lactobacillus strain	4.32±0.01^{ab}	53.79±0.40^{def}	6.61±0.75^{bcde}
3	Lactobacillus strain	4.42±0.07^{a}	51.96±2.36^{fg}	3.55±0.58^{def}
6	Lactobacillus strain	4.04±0.02^{de}	37.23±1.74^{h}	3.75±0.63^{def}
34	Enterococcus strain	4.37±0.05^{ab}	50.83±3.18^{fg}	2.89±0.76^{ef}
35	Enterococcus strain	4.39±0.04^{ab}	45.48±2.43^{g}	2.69±0.36^{ef}
69	Lactococcus strain	4.39±0.08^{ab}	53.11±0.67^{ef}	4.51±1.23^{cdef}
122	Lactococcus strain	3.49±0.05^{g}	71.52±0.55^{a}	11.46±0.48^{a}
123	Lactobacillus strain	4.07±0.02^{cde}	63.74±4.24^{bc}	5.24±2.50^{cdef}
127	Lactobacillus strain	3.81±0.02^{f}	64.48±0.52^{abc}	6.59±1.85^{bcde}
145	Lactobacillus strain	4.23±0.08^{bc}	64.77±1.53^{abc}	3.42±0.42^{def}
146	Lactobacillus strain	4.11±0.02^{cd}	63.65±1.34^{bc}	7.01±2.46^{bcd}
147	Lactobacillus strain	4.42±0.04^{a}	54.71±0.58^{def}	1.33±1.87^{f}
148	Lactobacillus strain	4.03±0.02^{de}	63.91±0.60^{bc}	9.22±1.03^{ab}
151	Lactobacillus strain	4.47±0.09^{a}	59.46±1.36^{cde}	4.31±0.32^{cdef}
153	Lactobacillus strain	3.91±0.14^{ef}	64.18±0.68^{bc}	6.15±1.13^{bcde}
156	Lactobacillus strain	4.00±0.01^{de}	67.72±1.18^{ab}	3.06±0.14^{ef}
157	Lactobacillus strain	3.90±0.04^{ef}	60.48±6.85^{bcd}	9.51±1.70^{ab}
163	Lactobacillus strain	4.11±0.06^{cd}	59.69±1.81^{cde}	7.69±1.01^{abc}
Sig.		**	**	**

*The percentage of inhibition was defined as [(A_{734} blank – A_{734} sample)/A_{734} blank] x 100 (%).
** The percentage of inhibition was defined as [(A_{517} blank – A_{517} sample)/A_{517} blank] x 100 (%).

The highest % of inhibition was found in whey fraction obtained after acidification with *Lactococcus* n. 122 strain both ABTS and DPPH assays with 71.52 % and 11.46 % respectively. These results were correlated with acidifying power of the same strains: LAB strains with a lowest pH values, had the highest % of inhibition; also a negative correlation between antioxidant activity by ABTS assay and pH values was found ($r = -0.545$, $P < 0.01$ for ABTS assay). Considering total evaluated pH range 3.50-6.00 negative correlations were evidenced between these pH values and antioxidant assays: $r = -0.649$ (ABTS) and $r = -0.306$ (DPPH). Antioxidant activity evaluated by DPPH assay had lowest values in all sample than ABTS assay, probably for the most specific activity of this last radical for this whey fraction. In conclusion, the strains of LAB were capable to have antioxidant capacity with the best results for % of inhibition by ABTS assay and clear differences with DPPH assay. This antioxidant activity was correlated with acidifying power of LAB and six strains (three *Lactobacillus*, two *Enterococcus*, and one *Lactococcus)*, exhibiting the highest radical scavenging activity, were selected for further investigation in order to produce lacto-fermented Mozzarella cheese with a longer shelf life

ACKNOWLEDGEMENTS

This research was supported by Ministero dell'Istruzione, dell'Università e della Ricerca (Prot. 957/ric, 28/12/2012), through the Project 2012ZN3KJL "Long Life, High Sustainability".

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ACTIVE COATINGS AGAINST DATES FUNGAL DECAY

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ABSTRACT

Biodegradable coatings based on chitosan incorporating either bergamot or bitter orange EOs at different concentrations were evaluated against *Aspergillus flavus*, under *in vitro* conditions, in terms of mycelium growth and spore germination inhibition, and under *in vivo* conditions on inoculated dates, stored at 20°C. Sensory analysis was carried out to evaluate the effect of the different coating treatments on the flavour and odour characteristics of the treated fruits. Combined treatments based on CH-2% (v/v) bergamot EO or CH–2% (v/v) bitter orange EO proved to be the most effective coatings to reduce conidial germination resulting in a 87-90% inhibition compared with the control. In fruit decay assays, coatings based on CH incorporating citrus oils were able to reduce fungal decay in the range of 52-62% at day 12.

Keywords: *Aspergillus flavus*, bergamot, bitter orange, chitosan, date, Locust Bean Gum, postharvest decay

1. INTRODUCTION

Date fruit, which is a rich source of carbohydrates, fibres, minerals, vitamins and antioxidant compounds, is one of the most in North Africa, Middle East and South-Asian countries. However during storage, this fruit is susceptible to infection by different spoilage fungi resulting in economic losses, especially for exporting countries. In Tunisia, the highest exporter of "deglet Nour' date variety, more than 50% of dates fruit are lost due to fungal spoilage. *Aspergillus flavus* and *Aspergillus parasiticus* have been reported to be among the most common fungal species infecting dates during storage (AHMED *et al*., 1997). Under conditions of high humidity and moderate temperature, these postharvest fungi may have the potential to produce aflatoxins which are considered to be among the most significant food contaminants regarding to their negative impact on public health and food security. In recent years, considerable attention has been directed toward natural essential oils (EOs), as a promising approach for controlling fruits' postharvest decay and reducing chemical-based treatments. However, the use of these compounds is often limited due to their intense aroma and their potential toxicity. The incorporation of EOs into edible coating formulations has been investigated as an effective approach to solve some of these problems, by lowering the diffusion processes and maintaining high concentrations of active molecules on the surface of the fruit. Among the polysaccharides used in the edible coating formulations,
chitosan (CH) and Locust Bean Gum (LBG) have been reported to be of interest as potential coating components due to their excellent film forming properties as well as their ability to act as effective matrices for the entrapment of bioactive compounds including EOs (PERDONES *et al*., 2012).
This study aimed at screening the antifungal activity of five citrus EOs (bergamot, bitter orange, sweet orange, mandarin and lemon) against *A. flavus* in *in vitro* conditions and investigating the potential application of CH and LBG coatings enriched with the most efficient oils in controlling postharvest decay in inoculated dates.

2. MATERIALS AND METHODS

2.1. Screening for the most effective EOs against *A. flavus*

The Poison food medium method was used in order to screen the most effective citrus oil against *A. flavus* as previously described by ALOUI *et al*. (2014). The most effective EOs were selected for all the further experiments.

2.2. Determination of selected EO effective concentration

Based on Poison Food medium method, bergamot and bitter orange EOs were identified as the most effective against *A. flavus*. In order to determine the minimum effective concentration of the selected oils, an in vitro conidial germination inhibition assay was carried out, using the "cavity slide" technique as previously described by ALOUI *et al*. (2014). A concentration of 2% (v/v) EO was selected for the evaluation of the *in vitro* and the *in vivo* antifungal activity of the combined treatments based on CH or LBG enriched with either bergamot or bitter orange EOs.

2.3. Preparation of the film forming dispersions

CH solutions (1%, w/v) were prepared by dissolving CH powder in an aqueous solution of glacial acetic acid (1%, v/v), while that of LBG (0.5%, w/v) were obtained by dispersing LBG powder in heated distilled water with constant agitation. The selected amount of either bergamot or bitter orange EOs (2%, v/v) was then added to CH and LBG film forming solutions, before being homogenized at 13,500 rpm for 4 min, using an Ultra-Turrax T25 (IKA, Labortechnik GmbH., Munich, Germany).

2.4. *In vitro* antifungal activity of combined treatments

Conidial germination inhibition assays was carried out in order to evaluate the antifungal potential of CH and LBG either alone or in combination with either bergamot or bitter orange EOs at a concentration of 2% (v/v).

2.5. In *vivo* antifungal assay: Fruit decay

The antifungal activity of the most effective coating treatments was evaluated according to ALOUI *et al*. (2014). Briefly, dates previously washed with sodium hypochlorite (0.01%) were injured and dipped in a conidial suspension of *A. flavus* at a concentration of 10^5 conidia/mL for 1 min and dried at room temperature for 2 h. Inoculated fruits were then immersed in the different coating solutions (30 dates for each treatment) for 1 min and air-dried at room temperature before being stored at 25°C, 75% RH for 12 days. Disease incidence, expressed as the number of infected dates out of the total number of fruits per treatment, was daily evaluated.

2.6. Sensorial analysis

The effect of the different coating treatments on the sensory characters of dates has been evaluated 24 h after coating application, using the sensory profile method (UNI, 10957, 2003).

3. RESULTS AND DISCUSSIONS

3.1. Effect of combined treatments on conidial germination

As it can be inferred from Table 1, pure CH inhibited conidial germination by 17% compared to the control and the pure LBG treatment. Combined treatments based on CH–2% bergamot EO and CH–2% bitter orange EO were the most effective in reducing conidial germination allowing an inhibition in the range of 87-90% compared with the control ($p < 0.05$), followed by those based on LBG enriched with either bergamot or bitter orange for which conidial inhibition was in the range of 73-82%. In agreement with our results, DOS SANTOS and AGUIAR (2012) reported an inhibition by more than 90% of conidial germination of *Rhizopus stolonifer* and *A. niger* when CH was assayed in combination with origanum EO. According to these authors CH and origanum EO inhibited germination through an interaction with the cell wall of conidia.

Table 1: Effect of different treatments on conidial germination inhibition of *Aspergillus flavus*. Mean values and standard deviation.

Treatments	Conidial Germination Inhibition (%)
Control	0.00 ± 0.00^{a}
LBG	0.00 ± 0.00^{a}
CH	17.50 ± 2.50^{b}
LBG-2% bitter orange EO	73.57 ± 1.24^{c}
LBG-2% bergamot EO	82.20 ± 2.31^{d}
CH-2% bitter orange EO	87.12 ± 0.82^{e}
CH-2% bergamot EO	93.61 ± 1.20^{f}

[a-f]: different superscripts indicate significant differences among treatments ($p < 0.05$).

3.2. Fruit decay

As shown in Table 2, pure CH coatings led to a significant delay in the rate of fungal decay. At day 11, although all the uncoated dates were infected, only 55% of those treated with pure CH were decayed compared to the control and those treated with pure LBG ($p < 0.05$). This result seems to be related to antifungal activity of CH previously proved against postharvest fungi including *Aspergillus*. Statistical analysis revealed CH coatings enriched with either bergamot or bitter orange to be the most effective in controlling fungal decay of dates allowing a reduction in the range of 52-62% compared with the control at day 12 ($p < 0.05$).

Table 2: Effect of coating treatments on the decay percentage of inoculated dates during storage at 25°C. Mean values and standard deviation.

Storage time (days)	Fungal decay (%)					
	Control	LBG	CH	LBG-2% bergamot EO	CH-2% bitter orange EO	CH-2% bergamot EO
1	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$
7	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$	$0.00\pm0.00^{a,u}$
8	$43.33\pm4.71^{b,u}$	$41.66\pm2.35^{b,u}$	$8.33\pm2.35^{b,v}$	$10\pm0.00^{b,vw}$	$3.33\pm0.00^{b,wx}$	$0.00\pm0.00^{a,x}$
9	$66.66\pm4.71^{c,u}$	$63.33\pm4.71^{c,u}$	$16.66\pm0.00^{c,v}$	$18.33\pm2.35^{c,v}$	$3.33\pm0.00^{b,w}$	$0.00\pm0.00^{a,w}$
10	$81.66\pm2.35^{d,u}$	$78.33\pm2.35^{d,u}$	$31.66\pm2.35^{d,v}$	$36.66\pm4.71^{d,v}$	$11.66\pm2.35^{c,w}$	$6.66\pm0.00^{b,w}$
11	$100\pm0.00^{e,u}$	$100\pm0.00^{e,u}$	$54.99\pm2.35^{e,v}$	$59.99\pm4.71^{e,v}$	$36.66\pm0.00^{d,w}$	$24.99\pm2.35^{c,x}$
12	$100\pm0.00^{e,u}$	$100\pm0.00^{e,u}$	$73.33\pm0.00^{f,v}$	$78.33\pm2.35^{f,v}$	$48.33\pm2.35^{e,w}$	$38.33\pm7.07^{d,x}$

[a-f] : different superscripts within a column indicate significant differences among storage time ($p < 0.05$).
[u-x]: different superscripts within a file indicate significant differences among treatments ($p < 0.05$).

Moreover these coatings were able to delay the onset of disease symptoms and slow down mould growth during storage period. After 7 days of storage, more than 40% of dates were decayed; no signs of fungal decay, however, were observed for fruits treated with CH coatings enriched with both citrus oils. In agreement with our results, a further reduction in the fungal decay of inoculated dates has been observed by PERDONES *et al*. (2012) when lemon EO was added to CH coatings.

3.3. Sensorial analysis

Sensory evaluation revealed significant differences in four descriptors including colour, gloss, citrus odour and flavour. A significant decrease in both glossiness and colour of dates was observed after coating application ($p < 0.05$). This decrease was more pronounced in samples treated with formulations containing citrus EOs. A significant loss of surface glossiness has been also observed by PERDONES *et al*. (2012) in CH-coated strawberries when lemon EO was added to CH matrix. Such behavior was ascribed to the increase in the opacity of the film as a result of oil droplet aggregation during drying process. On the other hand, a relatively high intensity of citrus odour and flavour was detected by the panel in dates treated with formulations containing either bergamot or bitter orange EOs. However, none of the judges revealed the presence of off-flavours and/or off-odour.

4. CONCLUSIONS

Treatments based on CH or LBG incorporating citrus EOs were proven effective in inhibiting *A. flavus* conidial germination and in controlling postharvest decay in infected dates. CH coatings enriched with citrus oils were the most effective in reducing fungal decay of inoculated dates (52-62% at day 12). These results and the complete absence of off-flavours and off-odours demonstrate the potential of CH coatings carrying citrus EOs as a promising alternative to synthetic antifungal agents for controlling postharvest growth of *A. flavus* in dates.

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SHELF LIFE OF STORED NOT PASTEURIZED OLIVE-BASED PÂTÉS

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ABSTRACT

Among ready-made sauces, pâtés play an important role on the market, including olive-based pâtés that result one of the most common vegetable pâtés. They are generally subjected to pasteurization treatment that causes chemical and nutritional degradation of the product. For similar products, such as meat-based pâtés and pesto genovese, the effect of spices and aromatic plants, and modified atmosphere packaging (Map), respectively, was investigated. In this study, the effects of a natural antimicrobial commercial extract (Ex) and packaging atmosphere (Map), were evaluated to extend the shelf life of not pasteurized olive-based pâté. The microbiological and physico-chemical parameters were assessed at 1 and 28 days of storage in refrigerated conditions (4°C). The addition of extract and the use of modified atmosphere packaging allowed to keep lower levels of both microbial population and oxidative degradation up to 28 days.

Keywords: antimicrobial extract, olive-based pâtés, packaging atmosphere, shelf life, storage time

1. INTRODUCTION

In the middle of a revolution that is changing the concept of food and our way of eating, both the growth in consumers' awareness of the health benefits of fruit and vegetables and more demanding contributes to a continuous need for new products, have increased the demand for ready-to-use vegetable products with good flavor and adequate shelf life. In this context, a vast range of seasonings (ready sauces) for pasta or rice is available on the market. Among vegetable pâtés, olive-based ones result very common. This kind of product usually is subjected to thermal stabilization process which, besides extending shelf life and ensure the safety of the product, causes modifications of the sensorial characteristics and a decrease of the nutritional value (COSMAI *et al*., 2013). To minimize these undesirable effects, several non-thermal stabilization processes have been proposed and currently applied for vegetable preserved food. Also natural and chemical food preservatives have been used to inhibit the growth of food borne bacteria and to extend the shelf life of processed food, even if consumers are increasingly demanding processed foods free or with a reduced level of chemical antimicrobial agents (WITKOWSKA *et al*., 2013). Many culinary herbs, spices and aromatic plants exhibit an antimicrobial activity and so could provide a source of acceptable and natural alternatives to the chemical food preservatives. A further contribution also is represented by modified atmosphere packaging (Map), in which the composition of the atmosphere surrounding the food in the package is changed, that resulted particularly effective in developing markets for chilled, short shelf life low-acid foods, on cheese and pesto genovese (FABIANO *et al*., 2000). The antimicrobial effect of plant essential oils as well as of modified atmosphere packaging was investigated for thermally stabilized meat-based pâtés (ESTÉVEZ *et al*., 2006; SOLER-RIVAS *et al*., 2011) and pesto sauce (FABIANO *et al*., 2000), respectively, whereas no study is present in literature for vegetable pâtés, to the best of our knowledge. On the basis of these assumptions, the objective of this work was to extend the shelf life of not thermally stabilized olive-based pâtés by means of the combined effect of a natural antimicrobial commercial extract and Map, during refrigerated storage.

2. MATERIALS AND METHODS

Olive-based pâtés were produced with 77% of cv. *Bella di Cerignola* table olives, 15.5% cv. *Coratina* extra-virgin olive oil and 7.5% of grilled zucchini, all purchased from local retailers. After preliminary operations such as washing and pitting of table olives, the ingredients were mixed by means of a homogenizer (Waring LB20 ES, Rome, Italy). The pâtés were produced both without (control, C) and with the addition, during the homogenization phase, of 1 g/kg of a commercial garlic extract (Ex, Proallium, DOMCA, Spain). After mixing, the product was transferred into 70 g plastic trays, packed by means of a heat sealer (Orved, model VGP 25n, Italy), with two different atmospheres: i) no modified atmosphere (no-Map) and modified atmosphere (Map, 70% N_2 - 30% CO_2). The products were stored at 4 °C, and sampled at T1 (one day after production) and at T28 (28 days of storage). Three independent production trials were carried out.
Rheological properties were evaluated by Back Extrusion test using a Texture Analyser (Z1.0., Zwick Roell, Germany) with a load cell of 1 kN. Samples were scooped into glass cylindrical container. Afterwards, the plunger (4.5-mm-diameter) penetrated to a depth of 8 mm within the sample through one cycle compression, at a constant cross-head speed of 1.5 mm/s. The maximum force (N) was recorded as index of hardness. Hexanoic acid was sampled by solid-phase micro-extraction (SPME) and analyzed by a gas-chromatographic system equipped with mass spectrometer (GC/MS) (DE ANGELIS *et al*., 2015).

Microbiological analysis included total bacteria, lactic acid bacteria, *Pseudomanas*, *Enterobacteriaceae* (DE ANGELIS *et al*., 2015), molds and yeasts (PARADISO *et al*., 2015).

3. RESULTS AND CONCLUSIONS

The results of Back Extrusion test of the different pâtés as function of Ex and Map, at T1 and T28, were showed in Figs. 1 and 2, respectively. As shown, no evident differences were observed among different samples at the two storage times considered, with values ranging between 11.60 and 12.30 N at T1 and 9.90 and 11.00 N at T28. The significant decrease of hardness observed after 28 days of storage might be due to the loss of the water-holding capacity, due to the dissolution of CO_2 during storage, and favored by low temperature, as reported by other authors (GOULAS and KONTOMINAS, 2007).

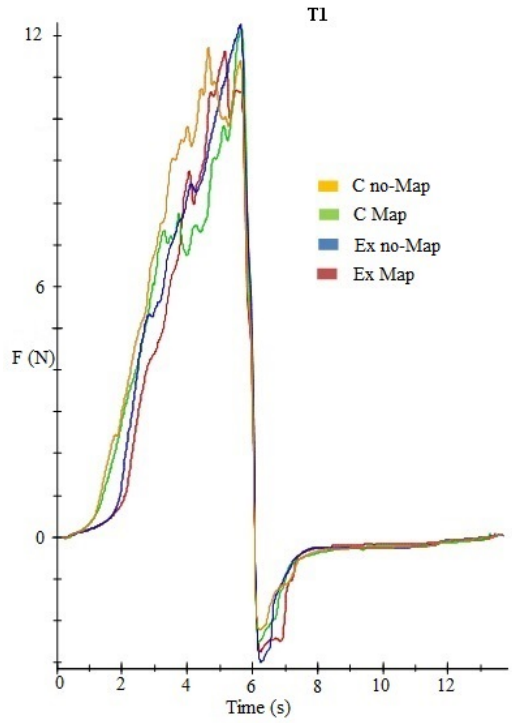

Figure 1.

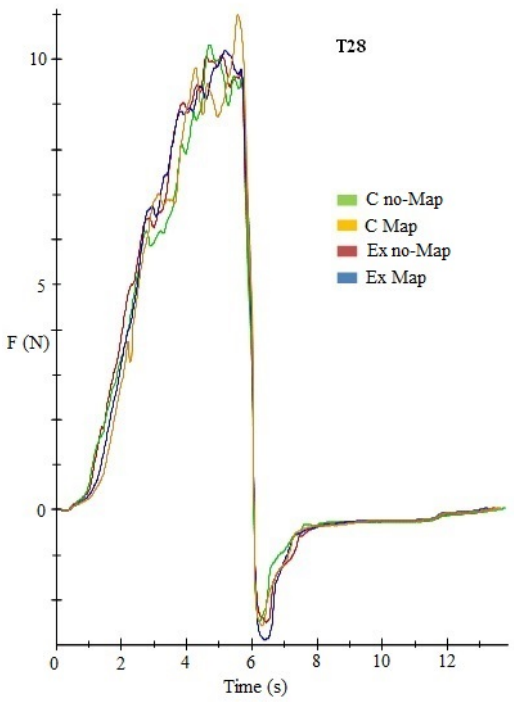

Figure 2.

The oxidative degradation – evaluated considering the hexanoic acid (Fig. 3), that represent an oxidation marker volatile compound and responsible of rancid off-flavor (PARADISO *et al*., 2008) – showed no substantial differences among samples at T1, while at T28 the C no-Map samples showed the higher increases (more than 2.5 mg/kg) than the other samples, for which the amounts of hexanoic acid were lower than 1.5 mg/kg. Similar trend was found by panel test (data no shown).
The relative cell density of the main microbial groups of the samples after 1 and 28 days of storage was showed in Fig. 4. As expected, all samples showed similar cell density at T1.
Presumptive *Pseudomonas*, responsible of appearance of bitter and rancid taste (WIEDMANN *et al*., 2000), and *Enterobacteria* increased during storage only in C no-Map.

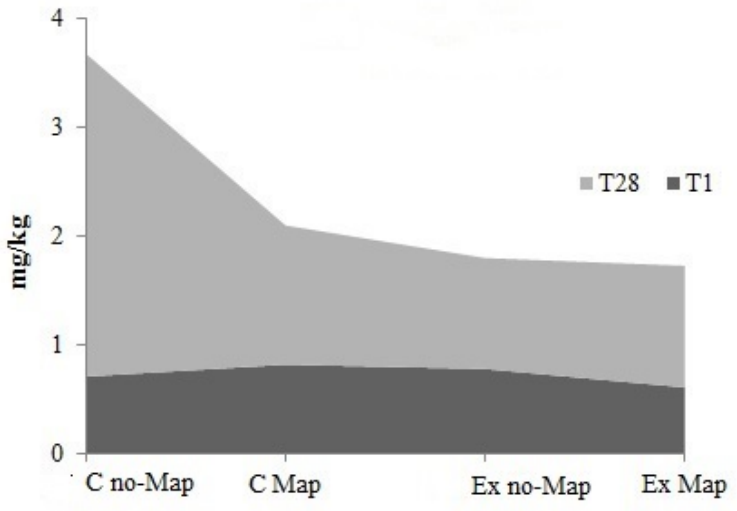

Figure 3.

In addition, presumptive molds and yeasts were not found in Ex no-MAP at T28. The combination of Ex and Map resulted in different microbial interactions/competitions driving in a lower relative abundance of total bacteria and a higher percentage of molds and yeasts compared to the control and MAP samples.

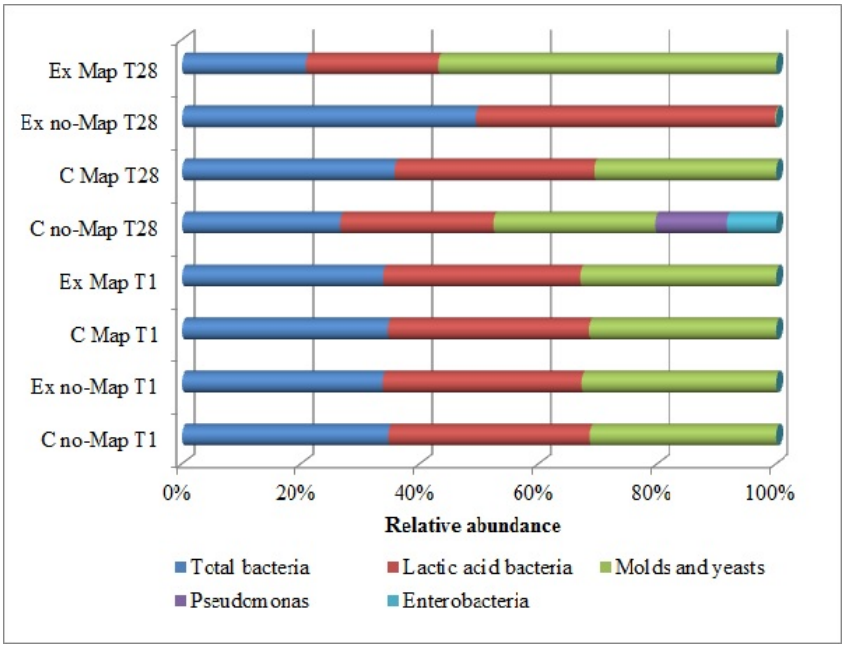

Figure 4.

In conclusion, the storage time caused a decrease of hardness in all samples; the use of extract and of modified atmosphere packaging allowed to keep lower levels of both microbial population and oxidative degradation improving the shelf-life of the products.

Finally, the influence of modified atmosphere packaging was more evident for samples in which the extract was not added.

ACKNOWLEDGEMENTS

This research was supported by Ministero dell'Istruzione, dell'Università e della Ricerca (Prot. 957/ric, 28/12/2012), through the Project 2012ZN3KJL "Long Life, High Sustainability".

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ECONOMIC ASSESSMENT OF FRENCH FRIES PRODUCTION, COMPARING DIFFERENT EDIBLE COATING

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ABSTRACT

Deep-fat frying is widely used to prepare tasty foods, but oil uptake in fried foods has become a health concern. Food coatings may become a good alternative to solve this problem. A coating at the surface of the foods determines mechanical and barrier properties characteristics of the used substrate.
This study seeks to determine the economic convenience on the use of some edible films on french fries positioned in the consumption market to reduce oil uptake.
Six different edible coating were tested: methylcellulose, chitosan, locust bean gum (LBG), pectin from citrus, Aloe Vera, Plantago psyllium.
Therefore, the effects of such innovation will be evaluated from a technical, economic and social point, in relation to the most suitable edible film able to reduce the absorption of commercial peanut oil during frying processing. The innovation aimed to improve the qualitative food human health protection, therefore a contribution to the healthy lifestyle for consumers.

Keywords: consumers, economic sustainability, edible coating, social sustainability

1. INTRODUCTION

Recently there have been a number of studies performed on the use of edible film coatings to perform different functions in food, in particular, it has been found that various hydrocolloids, especially cellulose derivatives, forms gels which can be used in frying to reduce oil adsorption (KROCHTA *et al*., 1994). The soft and moist interior together with the crispy crust are desirable characteristics of most fried foods. Lipid content of french fries increases from 0.2 to 14%, thus, oil uptake in fried foods has become a health concern. Food coatings may become a good alternative to solve this problem. For this product innovation, industries would intervene to the existing production line including two new equipment (a tank for dipping and a hot air tunnel for drying) but the associated costs would be offset by the total additional profit margins for producers.
Six different edible coating were chosen, some of them are the most studied and used commercially to improve appearance, to prevent water loss that leads to shriveling and subsequent loss of marketability and to maintain quality through delayed ripening and senescence. In particular, we compared edible films available on the market some of which are likely to be prepared directly in the company with greater assurance of environmental (ALLEGRA *et al*., 2015) and social (DI VITA *et al*., 2015) sustainability.
The aim of this study was to determine the economic convenience on the use of some edible films on french fries positioned in the consumption market to reduce oil uptake.

2. MATERIALS AND METHODS

In relation to the objectives of technical-economic and food security of this work, it was necessary to start the research identifying the types of edible coatings to be tested to evaluate the best formulation for the fries, as well as to ensure its producers and corresponding prices.
Methylcellulose (400 cP, Sigma-Aldrich), chitosan (800 cP, Sigma-Aldrich), locust bean gum (LBG, Sigma-Aldrich) and pectin from citrus (Alfa Aesar) were purchased and tested to assess the best formulation. Aloe Vera and Plantago Psyllium were provided by local producer as a *free sample* to be tested and to evaluate the simplicity of use, the excellent formulation, maintaining the natural character and food security; in fact, they may also be prepared directly in the company. For these reasons, the last two coatings were considered priceless, so as to postpone, in this work, the preparation of financial statements, considering the evaluation of application on chips limited to technical and social aspects.
Tubers were washed, peeled and cut into strips, than immersed in distilled water for 2 min. Next, they were blanched in water at the temperature of 80°C for 2 min. Potato strips were dipped in the different coating for 2 min and then coated sticks were allowed to dry by placing them in a forced air oven at 30°C for 2 hours. Potato coated sticks were fried in peanut oil at the temperature of 180°C for 3 min.
To establish the total cost we assessed the direct and indirect costs. The first refer to the primary means employed in the machining process (potato, peanut oil, labor, distilled water, coating, etc.), while the seconds are deducted from capital assets that complement the original processing line (depreciation and interest for the steel tank and dryer), and by facility costs in varying proportions for management expenses, administrative, accounting.

3. RESULTS AND CONCLUSIONS

The addition of the edible coating to the industry of french fries, does not make any structural changes to the relative transformation process. The only fixed capitals that integrate the processing line ad hoc are a steel tank and a heater for drying dimensioned with the production capacity of chips production, for which the amount of depreciation and interests, considering the total cost of production are very limited; especially for the steel tank which has a technical-economic rather long enough.
With the same technical conditions, is the choice of the edible coatings to be used to determine different levels of total costs per unit of french fries that is strictly dependent from the nature of the product. The difference are between the ready to use powder such methyl cellulose, chitosan, locust bean gum and pectin, or the raw material that needs to be processed in the industry like Aloe Vera and Plantago Psyllium.
In french fries coated industry, the cost of production of the partial solution in distilled water decreases from methylcellulose to chitosan, to locust bean gum to pectin. In particular the costs are 0.77 €, 0.31 €, 0.11 € and 0.08 € per 100 ml respectively, which should then be recalculated considering the quantity of chips transformed (Fig. 1).

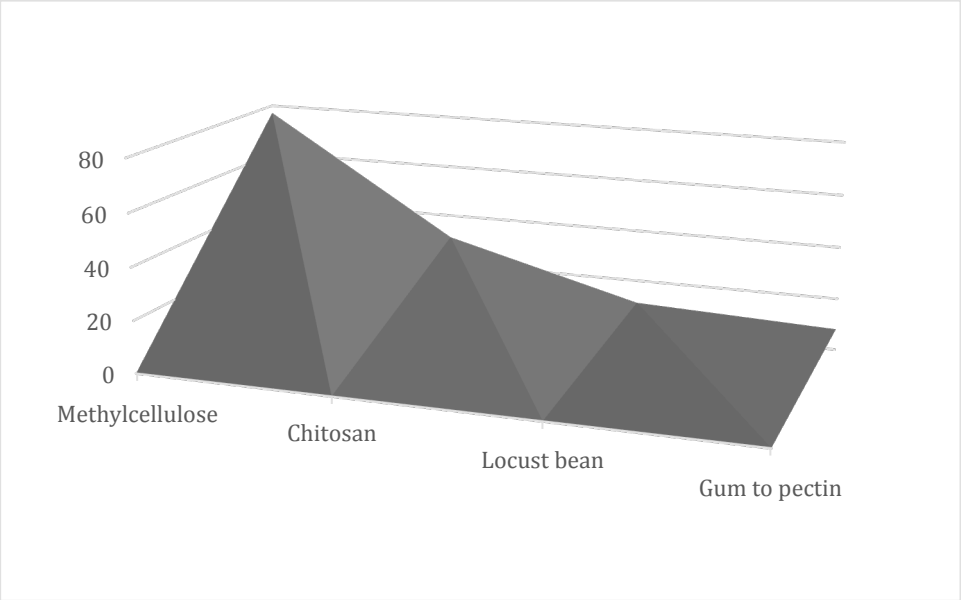

Figure 1: Early indications of the coating production cost (Value euro cents).

Looking at the significant difference between the coating considered, at the time the choice fall back on the technological aspects and health. Considering the fat percentages, all coated samples shown a lower fat intake respect to the control (Fig. 2).
The percentages of fat reduction calculated for dipped french fries is higher in samples treated with Aloe Vera and pectin; the worst results are from LBG film perhaps for the difficulties in producing an homogeneous coating around samples (Fig. 3).

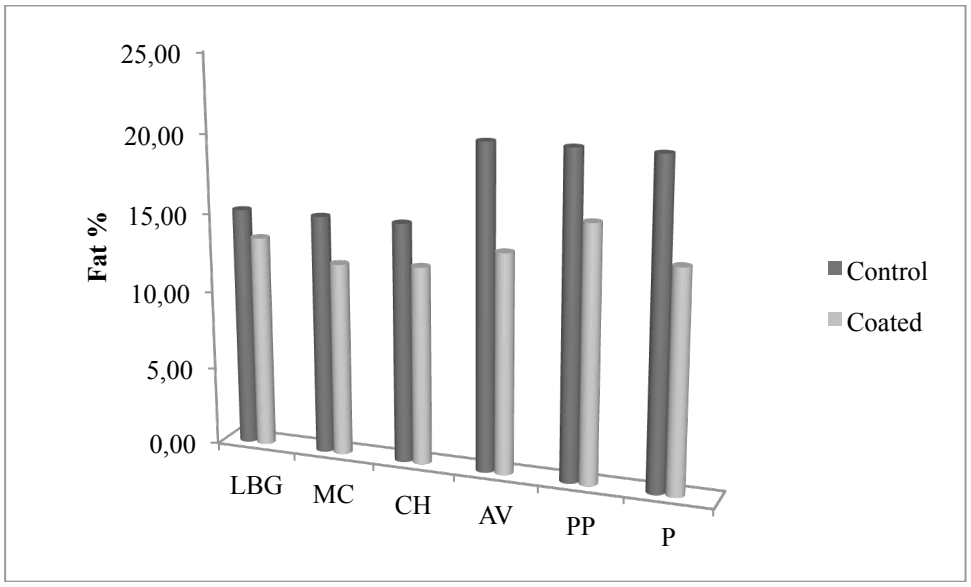

Figure 2: Fat percentage of different tested coating applied on french fries sticks (%) in control and coated samples. (LBG=locust bean gum, MC=methylcellulose; CH=chitosan; AV=Aloe Vera; PP=Plantago Psyllium; P=pectin).

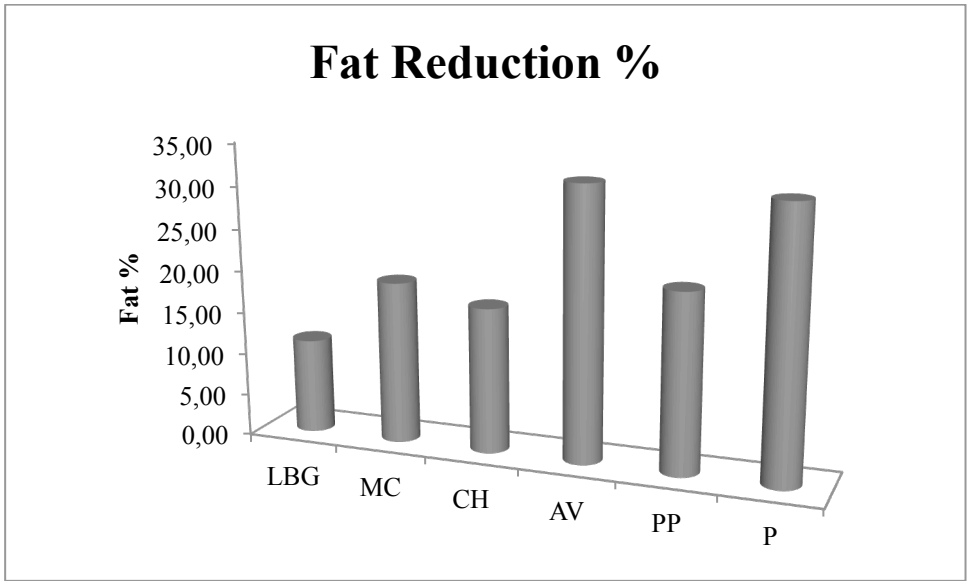

Figure 3: Fat reduction (%) of different tested coating applied on french fries sticks (%).(LBG=locust bean gum, MC=methylcellulose; CH=chitosan; AV=Aloe Vera; PP=Plantago Psyllium; P=pectin).

Many authors verified the effects of applying mono-layer or double-layer or triple-layer coatings (Daraei Garmakhany, et al., 2014). An industrial production able to obtain an effective results in reducing oil uptake and increasing the moisture retention during deep-fat frying is of course interesting from economic point of view, linked with the opportunity to sell the product with an "healthy message" to the consumers.

ACKNOWLEDGEMENTS

This work was financially supported by the project "SHELF-LIFE - Integrated use of innovative technological approaches to improve the shelf-life and preserve the nutritional properties of food products" carried out by the Cluster Sicily Agrobio and Fishing Industry and funded by the Research Fund PON R&C 2007-2013, DD 713/Ric. (PON02_00451_3361909).

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SHELF LIFE EXTENSION OF A CHEESE CAKE WITH ANTIMICROBIAL ACTIVE PACKAGING

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ABSTRACT

The shelf life extension of a cheese cake subjected to modified atmosphere (MAP) and active packaging (AP) was evaluated. Samples were packaged under N_2/CO_2 (70/30) (MAP), or by inserting labels with the essential oil (EO) of *Cinnamomum zeylanicum* inside trays (AP). A control batch was packaged under air. Changes in microbial growth, chemical-physical parameters, and sensory attributes were monitored for 42 days at 20°C. MAP allowed a mould-free cheese cake shelf life of 28 days, while AP was ineffective in extending the shelf life of product, with respect to control samples, which spoiled after only 7 days. Panellists judged MAP cakes over the acceptability threshold during their whole shelf life.

Keywords: active packaging, modified atmosphere packaging, sensory analysis, shelf life

1. INTRODUCTION

Cheese cakes may be classified as ambient cakes with intermediate or high moisture values and shelf life is limited by microbial growth, mainly appearing as moulds (JONES, 2000; SMITH *et al*., 2004). The control of post baking contamination is the preferred mean to control microbial spoilage (SMITH *et al*., 2004). Out of MAP, the application of AP through the use of antimicrobials appears as a promising application (SALMINEN *et al*., 1996; GUYNOT *et al*., 2003; PRASAD and KOCHAR, 2014).
The objective of this work is, thus, to extend the mould free shelf life while maintaining sensory quality of a cheese cake, by using both the consolidated technique of MAP and a more promising approach of AP.

2. MATERIALS AND METHODS

Cheese cakes were acquired by a local plant and soon packaged. Samples for MAP (N_2/CO_2 – 70/30) were placed inside multistrate (EVOH/PS/PE) gas barrier trays, (Aerpack B5-30, Coopbox Italia, Reggio Emilia, Italy), which were wrapped with a multistrate (EVOH/OPET/PE) gas and water barrier film (54 mm, EOM 360B, Sealed Air, USA). Control and AP samples were placed in gas barrier XrPET trays and wrapped with a XrPET film (Coopbox Italia, Reggio Emilia, Italy). Four batches of AP trays were prepared by sticking inside them 1, 2, 3 and 4 shelf-adhesive (20x20mm) labels containing the essential oil (EO) of *Cinnamomum zeylanicum* (provided by the GUIA group from the University of Zaragoza), respectively. Samples were stored at 20°C and inspected at regular intervals for chemical-physical, microbiological and sensory parameters. Chemical-physical measured attributes were pH, dry matter (%) and water activity (a_w) and were obtained with routine laboratory methods. For microbiological analyses 10 grams of samples were homogenised in 90 mL of sterile 0.1% peptone solution, decimal dilutions were prepared and plated on specific media (PCA, Baird Parker and Rose Bengal) to enumerate total microbial count, *Staphylococci*, moulds and yeasts. Sensory analysis involved 32 untrained consumers that evaluated the acceptance of the samples by using a hedonic scale from 1 to 9 for the attributes that best represent the product and are more susceptible to changes during storage, that are: flavour (1, none; 9, very intense) off-odours (1, none; 9, very strong) and softness (1, not soft; 9 very soft), An overall acceptability score was also asked on a scale from 1 to 9 (1, extremely dislike; 9, extremely like). Headspace evolution of EO was analysed by SPME extraction and GC/MS. Data were analysed with a two-way analysis of variance with thesis and storage time as group factors and means separated by Duncan's Multiple Range Test at $p \leq 0.05$.

3. RESULTS AND DISCUSSIONS

The dough of freshly baked cakes showed a_w and dry matter values of 0.889 and 78.2%, while filling values were 0.916 and 70.6% respectively. The dry matter content tended to increase in the dough and in the filling during the storage, surely to the evaporation of water from the samples to the surrounding atmosphere.
The a_w values of the product make it very susceptible to mould alterations and to the possibility of *Staphylococci* growth. Colony count on PCA medium at the start of the experiment evidenced a low number of microorganisms, whereas viable cells did not appear in Rose Bengal. Plates made from control and AP cakes evidenced yeast and moulds after seven days in storage, in fact moulds appeared on cakes of both batches,

while MAP delayed yeast and mould growth up to 28 days. Although the PCA count was very low in MAP trays after 28 days it is possible that oxygen was present in the headspace, thus allowing the mould growth after a long lag phase. *Staphylococci* were not detected in any sample.
The results of acceptability during storage did not show significant changes for flavour, off-odours, softness, friability. Moreover, preference was not significantly different among the three batches.
In conclusion, only MAP extended considerably the shelf life of cheese cakes, while the essential oil of *Cinnamomum zeylanicum* had no effect on delaying the mould growth. Further studies are in progress to test the efficacy of other essential oil on the shelf life of this food specialty.

ACKNOWLEDGEMENTS

This research was supported by Ministero dell'Istruzione, dell'Università e della Ricerca (Prot. 957/ric, 28/12/2012), through the Project 2012ZN3KJL "Long Life, High Sustainability" and by the Gobierno de Aragón given to GUIA group (T-10).

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SESSION III

"Long Life, High Sustainability through New Shelf Life Testing"

SHELF-LIFE EXTENSION OF FRESH PRODUCE BY EDIBLE COATING

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ABSTRACT

Fresh and fresh-cut produce get a growing share on the worldwide food market as they combine the aspect of convenience food with healthy nutrition. Nevertheless, these non-treated food products bear a risk of microbial contamination with pathogenic microorganisms and have limited shelf-life due to their fresh character and big surface. Edible coatings can comply with several functions to protect those sensitive food products. In this study alginate was used as coating for fresh-cut cantaloupe melon to act as a barrier to water loss and a carrier for antimicrobial hop extracts. For mung bean sprouts chitosan, which is antimicrobial by nature, was used. Both experiments showed that edible coatings can enhance the shelf-life of these products. The edible coatings have to be designed for each produce individually.

Keywords: edible coating, fresh-cut, cantaloupe melon, hop extraxt, alginate

1. INTRODUCTION

Fresh and ready-to-eat convenience products like fresh and fresh-cut fruits and vegetables are an upcoming food market sector [1]. Because of their fresh nature, mild processing technologies and increased surfaces as consequence of the cutting process, these products are susceptible to quality changes like desiccation, enzymatic reactions, oxidation, loss of the cellular integrity and microbial deterioration by a primary microflora [2]. The latter is a major problem if the products are consumed in a raw state. Edible coatings are an innovative way of packaging fresh and fresh-cut produce each individually and thus protecting them from quick quality decay. Edible coatings comply with several functions to protect the coated food from mechanical damages or water loss, to represent a specific barrier to water vapor, oxygen or CO_2 and to act as a carrier for functional additives like antioxidants or antimicrobials. Especially fresh-cut products host a large microbial population, particularly bacteria [3]. Antimicrobials like chemically synthesized preservatives get more and more rejected by the consumer. Natural substances are preferred instead. Beta-acids containing natural hop extracts exert high bactericidal effect on various food-related bacteria. Especially gram-positive bacteria such as *Listeria* are very sensitive to hop beta-acids [4, 5]. Other edible coating materials like chitosan are antimicrobial by nature [6]. Thus, to exert an antimicrobial effect, no addition of an antimicrobial is needed. As every produce has different surface properties and spoilage mechanisms, edible coatings have to be designed individually. The individual design will be shown on two different examples, fresh-cut cantaloupe melon coated with alginate and the addition of antimicrobial hop extracts and mung bean sprouts coated with chitosan.

2. MATERIALS AND METHODS

2.1. Application of alginate edible coating on fresh-cut cantaloupe melon

Cantaloupe melons were peeled and cut into pieces. Coating solution was prepared by dissolving 1.25% sodium alginate, 2% glycerol, 0.125% sunflower oil, 0.1% Tween 40 and 0.6% Span 80 in sterilized water while stirring at 70°C. 0.0625% of a beta-acid hop extract was added as antimicrobial. In order to induce the cross-linking reaction, 2% calcium lactate was dispersed in distilled water. 0.0625% hop extract was added as antimicrobial. After each dipping process, pieces were allowed to drain for about 2 min. As a reference, pieces of melon without any coating treatment were used. The pieces were transferred into EPS trays and closed with stretch films. The melons were stored at 8°C for up to 8 days. After several time intervals a total viable count of at least three independent samples was performed.

2.2. Application of chitosan edible coating on fresh mung bean sprouts

Mung bean sprouts samples were washed for 20 sec with tap water with the addition of 0.5% chitosan solution, while control group samples were washed with tap water only for 20 sec. Samples were transferred into aPET trays and stored at 4°C for up to 9 days.

2.3. Assessment of the total viable count

30 g of cantaloupe melons and 10 g of mung bean sprouts respectively were homogenized in Tween 80 added ringer solution (270 ml and 90 ml respectively) in a stomacher for 2

min. Decimal serial dilutions were pour-plated in appropriate dilutions with sterile plate count agar and incubated at 30°C for 48 h.

2.4. Assessment of the weight loss

Percentage weight loss was determined by recording the weight on the initial day and the testing day, using a laboratory balance. Determination was done in triplicate and average was obtained. The percentage of weight loss was calculated.

3. RESULTS AND CONCLUSIONS

The alginate coating had a positive impact on the weight loss of the melon pieces. The average weight loss of the uncoated pieces was about 1.5%, whereas the alginate coated pieces never lost more than 0.5% of their initial weight. Additionally, the hop extract incorporated into the coating and the cross-linker had bactericidal effect for the first two storage days. Throughout the storage time the coated melon pieces had a 1 to 2 log-cycle reduced total viable count compared to the reference pieces (Fig. 1). According to the German Society of Hygiene and Microbiology [7] the microbiological limits for fresh-cut melons is 10^7cfu/g. The uncoated melon reached this value after 6 days, whereas the alginate coating with hop extract could extend this period to the 8th day of storage.

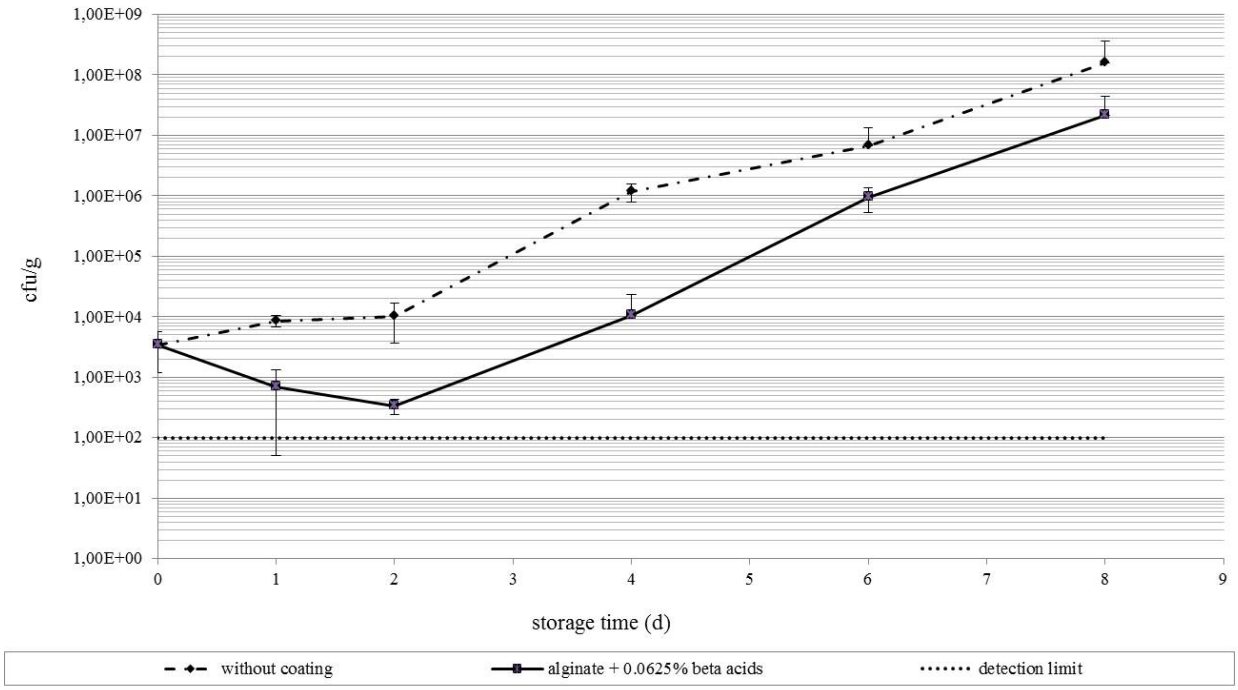

Figure 1: Development of the total viable count on fresh-cut cantaloupe melon without coating and after dipping in alginate and Ca-lactate solution with addition of beta-acid containing hop-extract (0.0625%) over 8 days packed in EPS trays; storage temperature: 8°C; data are expressed as means ± SD, n=3 [8]

Chitosan does not only serve as a coating matrix in already very thin layers, but also has an inherent antimicrobial effect. Therefore the chitosan was applied in the washing process of the sprouts without the addition of further additives. During the first four storage days the chitosan washed sprouts behaved similar to the just washed samples; the total viable count increased slightly about 0.5 log-cycles. But after this adaption period the total viable count decreased. At the end of the storage period, the microbial load was almost 2 log-cycles lower than the control group (Fig. 2). In summary, edible coating had a positive effect on different aspects concerning shelf-life of fresh and fresh-cut produce.

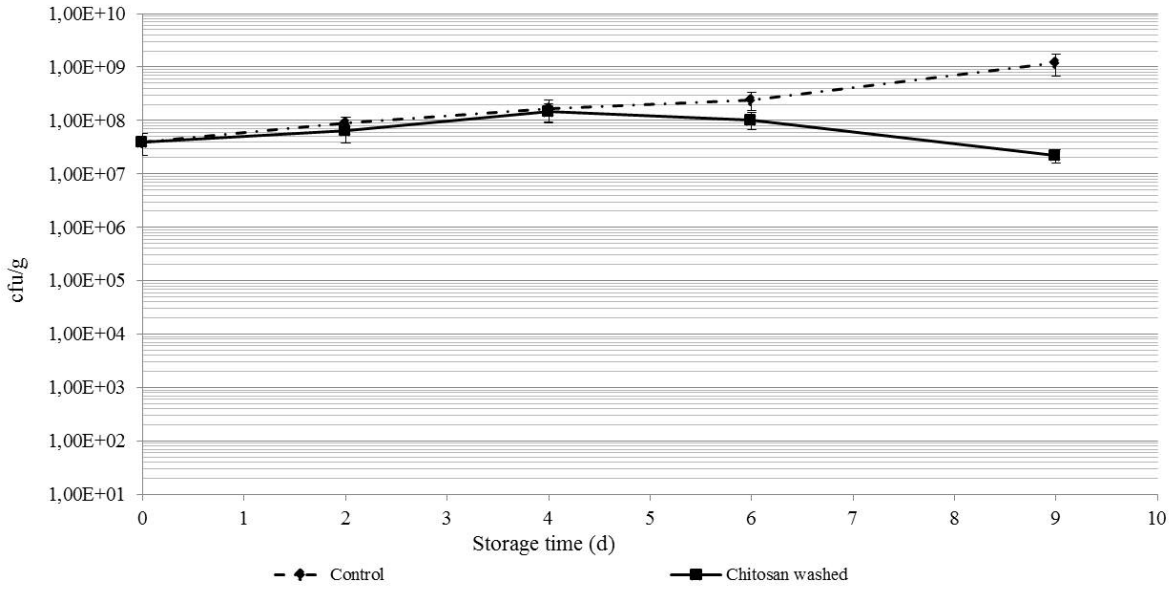

Figure 2: Development of the total viable count on mung bean sprouts after washing in tab water (control) and in tab water with addition of 0.5% chitosan over 9 days packed aPET trays; storage temperature: 4 °C; data are expressed as means ± SD, n=3 [9].

ACKNOWLEDGEMENTS

The research was conducted in the IGF-project 95EN/1 which was implemented under the CORNET initiative and carried out under the auspices of the German Federation of Industrial Research Associations in the framework of the program to promote collective industrial research (IGF) and financed within the budget of the German Federal Ministry of Economics and Technology (BMWi).

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THEORETICAL EVALUATION OF OXYGEN BARRIER ON COFFEE POD

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ABSTRACT

Due to the coffee's nature, the evaluation of packaging barrier properties is often requested; it is widely known that the presence of oxygen can compromise the quality of the coffee content in pods and, considering the cost of the final product, it's important that the pod works exactly as it is projected for. Another important point is the recent introduction of bio materials for the production of pods, which have a low environmental impact but with the same request to protect the coffee inside. The permeability analysis with traditional machines is well known to require adequate accessories, particularly when it is request in function of the geometry of the package, with long times to process data.
Otherwise, considering the properties of materials and the flow ratio O_2/CO_2 across them, it is also possible to make some consideration and theoretical evaluations. For these reasons a method for oxygen barrier evaluation has been developed; a closed system in which pods are put in and where is fluxed carbon dioxide has been created and times for carbon dioxide to accumulate inside have been calculated.
The system provides that after some days from the initial conditioning it is possible to analyze every pods with a GC TCD method. All data are processed at the end of the analyses using a data sheet and by this way it is possible to calculate the oxygen barrier of coffee pads. This method could be preferred in some occasions, because of the rapidity of the analysis and the low cost. Furthermore, making a rapid GC TCD analysis gives the possibility to compare an elevate number of pads and, by this way, to process a statistical evaluation that is not possible with traditional machines; also considering this important aspect, it could be interesting to compare the use of this GC TCD method to others and, in case of low differences, try to individuate a conversion factor.

Keywords: coffee pod, gas barrier, permeability test

1. INTRODUCTION

Because of the interest to individuate new materials to make coffee pods and due to the necessity to have a pod that is barrier to oxygen, to individuate a system to evaluate permeability is important. A classic permeabilimeter has some disadvantages: it must have apposite heads, it needs time for gas carrier to equilibrate with atmosphere, it has hermeticity trouble, it needs time to prepare sample and, not less important, it requires a big investment. For these reasons a theoretical premise has been elaborated using Polymer handbook, in particular the chapter "Permeability and Diffusion Data".
The formula that defines Permeability is

$$Permeability = \frac{(quantityofpermeant)x(filmthickness)}{(area)x(time)x(pressuredropacrossthefilm)}$$

Starting from this formula it is possible to calculate permeability of materials and to convert a unit of P to another using the Fig. 1. If Permeability to Oxygen and to Carbon Dioxide is available, it is possible to calculate the Ratio between them of a material of interest, using the Chart 1.

B. CONVERSION FACTORS FOR VARIOUS UNITS OF THE PERMEABILITY COEFFICIENT

	Multiplication factors to obtain P in		
	$\frac{[cm^3]\,[cm]}{[cm^2]\,[s]\,[cm\ Hg]}$	$\frac{[cm^3]\,[cm]}{[cm^2]\,[s]\,[Pa]}$	$\frac{[cm^3]\,[cm]}{[m^2]\,[day]\,[atm]}$
From			
$\frac{[cm^3]\,[cm]}{[cm^2]\,[s]\,[cm\ Hg]}$	1	7.5×10^{-4}	$6.57 \times 10^{+6}$
$\frac{[cm^3]\,[mm]}{[cm^2]\,[s]\,[cm\ Hg]}$	10^{-1}	7.5×10^{-5}	$6.57 \times 10^{+5}$
$\frac{[cm^3]\,[cm]}{[cm^2]\,[s]\,[atm]}$	1.32×10^{-2}	9.87×10^{-6}	$8.64 \times 10^{+4}$
$\frac{[cm^3]\,[mil]}{[cm^2]\,[day]\,[atm]}$	3.87×10^{-14}	2.90×10^{-17}	2.54×10^{-3}
$\frac{[in^3]\,[mil]}{[100\ in^2]\,[day]\,[atm]}$	9.82×10^{-12}	7.37×10^{-15}	6.45×10^{-1}
$\frac{[cm^3]\,[cm]}{[m^2]\,[day]\,[atm]}$	1.52×10^{-11}	1.14×10^{-14}	1
$\frac{[cm^3]\,[cm]}{[m^2]\,[day]\,[bar]}$	1.54×10^{-11}	1.16×10^{-14}	1.01
$\frac{[cm^3]\,[cm]}{[cm^2]\,[s]\,[Pa]}$	1.33×10^{3}	1	$8.75 \times 10^{+10}$

Figure 1.

2. MATERIALS AND METHODS

The necessity to evaluate permeability to gases without using a permeabilimeter conduced to elaborate a laboratory test equipped with a glass drier, a CO_2 tank, tubes, 100 ul syringe, GC-TCD AGILENT 7890. Tubes are used to connect the CO_2 to the glass drier, where carbon dioxide can accumulate and equilibrate with pods put inside. The system provides carbon dioxide to flush into a becker.
With this equipment it is possible to start the analysis; immediately in case of empty pods with top, after waiting about one night in case of pods without top. In this last case there is the possibility to put them on a glass with glue and analyze the system by this way. It has been calculated the time that is necessary to let pods equilibrate with carbon dioxide inside, that is about five days. After this conditioning under temperature control, pods can be analyzed using a GC TCD.

3. RESULTS AND CONCLUSIONS

After the analysis of atmosphere inside pods with GC TCD instrument, it is possible to calculate permeability; before, it is necessary to take into account that there is carbon dioxide into the atmosphere and so a blank of air and CO_2 has to be considered and a response factor between CO_2 and O_2 has to be calculate.
Using the formulas of Permeability described before, considering the area of CO_2 and O_2 obtained and also taking into account Chapter 1, it is possible to calculate Permeability. In the Chapter 2 there in an example of data obtained on four types of pods.

Chapter 1.

Polymer	T (°C)	Permeability Coefficient to O_2 x 1013 (*)	Permeability Coefficient to CO_2 x 1013 (*)	Ratio PCO_2/PO_2
LDPE (low density polyethylene)	25	2,2	9,5	4,3
HDPE (High density polyethylene)	25	0,3	0,27	0,9
PP (Polypropylene)	30	1,7	6,9	4,1
PS (Polystyrene)	25	2	7,9	4
PMMA (Polymethylmethacrylate)	25	0,89	3,8	4,3
PAN (Polyacrylonitrile)	25	0,00015	0,0006	4
SAN (Polyacrylonitrile-co-styrene 57/43)	25	0,14	0,27	1,9
PVAL (Polyvinyl alkohol)	25	0,0067	0,0092	1,4
PVC (Polyvinyl chloride unplasticized)	25	0,034	0,12	3,5
PVDC (Polyvinylidene chloride)	30	0,0038	0,022	5,8
PTFE (Polytetrafluoro ethylene)	25	3,2	7,5	2,3
PET (Polyethylene terefhthalate)	25	0,044	0,23	5,2
PA 6 (Polyamide 6)	30/20	0,029	0,066	2,3

	theoretical flow of O_2 (cm^3 / 24 h air)			
NP	blue pods	red pods	yellow pods	orange pods
1	0,053	0,052	0,051	0,052
2	0,051	0,051	0,052	0,051
3	0,051	0,051	0,053	0,051
4	0,053	0,051	0,052	0,053
5	0,053	0,052	0,051	0,050
average	0,052	0,052	0,052	0,051
s.d.	0,0010	0,0005	0,0007	0,0009
e.u.	0,0020	0,0010	0,0015	0,0018
min	0,049	0,050	0,050	0,049
MAX	0,055	0,053	0,055	0,055

In conclusion, the advantages of this method are the following: no errors due to hermeticity, work at 1 bar (security conditions), easy to conditionate pods, economic and rapid answer from TCD rivelator, easy to test a sufficient population of samples (about 60 pods in 8 hours).

DEGRADATION KINETICS OF CAROTENE IN CHOLESTEROL-FREE MAYONNAISE CONTAINING RED PALM OLEIN

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ABSTRACT

This research is aimed to determine the degradation rate of carotene in cholesterol-free mayonnaise containing red palm olein during storage. Cholesterol-free mayonnaises containing 74 wt% red palm olein stabilized with 0.25 wt% whey protein concentrate and 0.5 wt% modified starch were prepared. The mayonnaise samples were stored at 10, 25 and 35 °C for 8 weeks and the peroxide value, color and total carotene were evaluated. The results indicated that the peroxide value increased during the induction period and then decreased after the longer storage. There was no significant difference for the overall color of mayonnaise in the bottle but the color at the surface was changed over the storage period at all storage temperatures. Total carotene in mayonnaise decreased with the increase of the storage temperature and time, and respected to the second-order kinetics at the studied temperatures with R^2 of 0.80-0.84. The carotene degradation rate (k_{Ca}) was 0.0158, 0.0215 and 0.0258 ppm day^{-1} for the storage temperature of 10, 25, and 35°C, respectively. The rate constants were temperature dependent according to the Arrhenius equation (R^2 » 1.0) with the activation energy and Q_{10} values of 15.79 kJ mol^{-1} and 1.02, respectively.

Keywords: degradation kinetics, carotene, red palm olein, cholesterol-free mayonnaise

1. INTRODUCTION

Mayonnaises are well known thick emulsified food dressing prepared from vegetable oil, egg yolk, vinegar, some spices and water. The presence of egg yolk results in the desirable flavor, color and stability of mayonnaise. However, egg yolk is composed of cholesterol that has been recognized by various medical authorities as being an undesirable constituent that could be a leading cause for heart attack. This consideration has led to the development of cholesterol-free mayonnaise by using other proteins or emulsifiers (GHAZAEI *et al*., 2015: NIKZADE *et al*., 2012). Mayonnaise is generally regarded as a high-fat and high-caloric food. A positive relationship between dietary fat and development of cardiovascular diseases, hypertension and obesity was reported previously. Therefore, the traditional vegetable oil in mayonnaise was changed to healthy oils, e.g. fish oil or red palm olein. Red palm olein contains high concentration of carotenes, natural antioxidant and pro-vitamin A. For this reason, carotene in red palm olein could be considered to be a promising substance to reduce the incidences of certain chronic diseases (NAGENDRAN *et al*., 2000). However, the use of carotenes in foods is currently limited due to their chemical instability. This research is therefore aimed to determine the degradation rate of carotene in cholesterol-free mayonnaise containing red palm olein during storage.

2. MATERIALS AND METHODS

The mayonnaise recipe contained the following ingredients: red palm olein (Carotino SDN BHD, Johor Darul Takzim, Malaysia) 74 wt%, modified starch (Octenyl succinic anhydride, OSA) 0.5 wt%, whey protein concentrate (80% protein) 0.25 wt%, vinegar 6.5 wt% (5% (w/v) acetic acid), salt 2.0 wt%, mustard powder 1.0 wt%, sugar 1.2 wt%, and water 14.55 wt%. The whey protein concentrate, modified starch and water were mixed together, followed by the addition of the sugar, salt, mustard using a blender (HR1613 Hand Blender, Philips, China). Red palm olein was slowly added and mixed. Then vinegar was gradually added and homogenized (Ika Ultra-Turrax T25 Basic, Germany). The mayonnaise samples (100 g) were transferred to 120 ml glass bottles with caps and stored at 10, 25 and 35°C for 8 weeks in the dark. Peroxide value was analyzed to monitor the extent of lipid oxidation using AOAC (1997) standard method. The color of mayonnaise samples was measured using Minolta colorimeter (CM-3500d, Minolta, Japan) and reported as b* value. The total carotene content was calculated from the ultraviolet (UV) absorbance value at 446 nm according to the method of CHANDRASEKARAM *et al*. (2009).

3. RESULTS AND CONCLUSIONS

The change in peroxide value of mayonnaise with time as a function of storage temperature is shown in Fig. 1. The results indicated that the hydroperoxide slightly increased during the induction period and then decreased after the longer storage. In addition, the higher storage temperature showed the faster change. These results may be due to the higher degradation rate of hydroperoxide at high storage temperature.
For the color changing, the result shows that there was no significant difference for the overall color of mayonnaise in the bottle. However, the color at the surface of the sample was changed over the storage period at all storage temperatures as showed in Fig. 2.

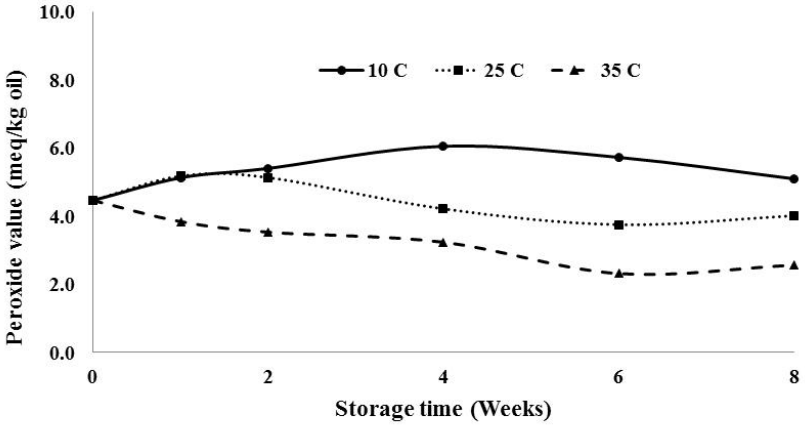

Figure 1: Change of peroxide value in mayonnaise during storage at different temperatures.

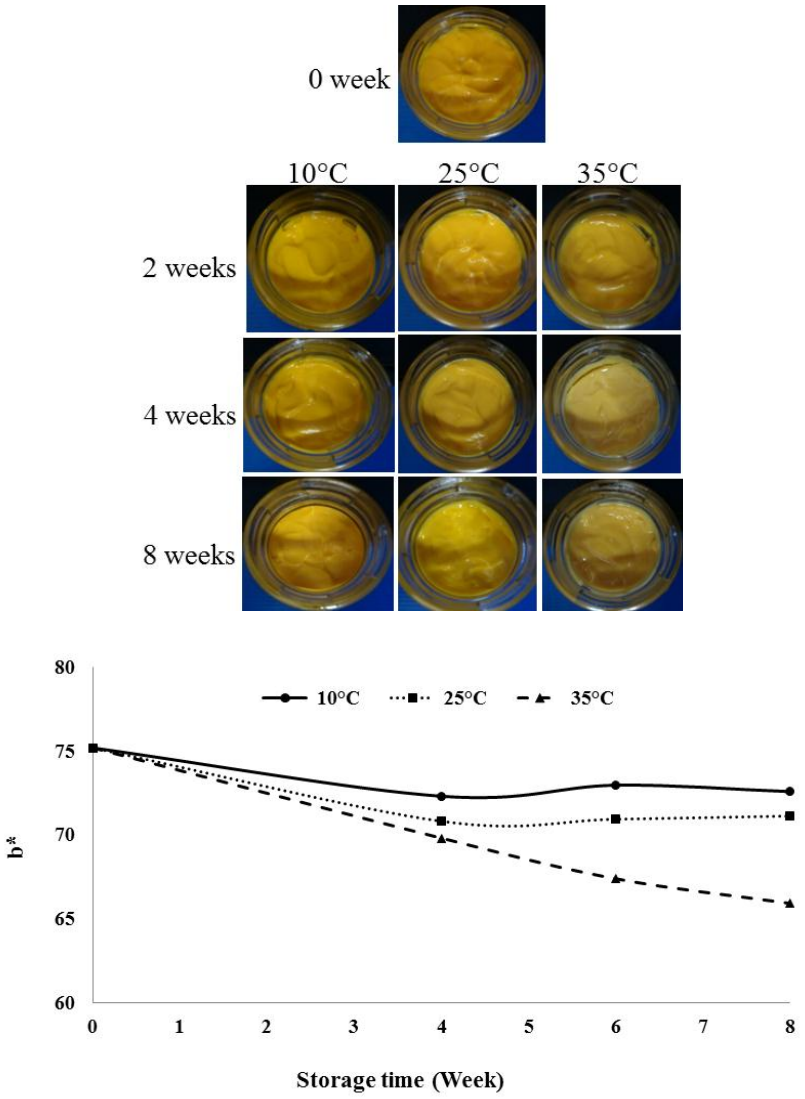

Figure 2: Digital image (A) and the b* value (B) at the surface of mayonnaise during storage for 8 weeks at different temperatures.

At 10°C, the overall color at the surface of mayonnaise was slightly changed; on the other hand, at the higher temperature, especially at 35°C, the color of mayonnaise changed from bright gold to pale yellow (Fig. 2A). After 8-week storage time, the result shows that the b* value was also decreased (Fig. 2B), which might be the cause of the degradation of carotene at the surface (RODRIGUEZ-AMAYA, 1999).

In Fig. 3, it shows the increase of carotene degradation in mayonnaise with increasing temperature. A decrease of carotene during storage was observed at all temperatures, following apparent second-order equation. The rate constants of carotene degradation increased with temperatures, following the Arrhenius equation as presented in Fig. 4.

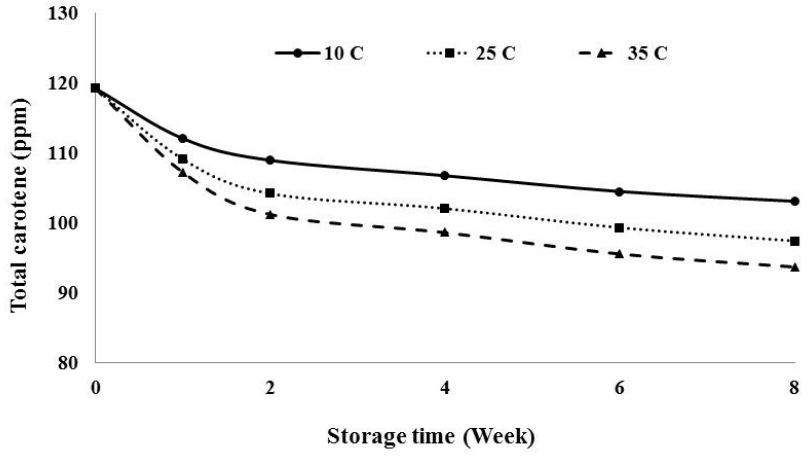

Figure 3: Degradation of total carotene in mayonnaise during storage at different temperatures.

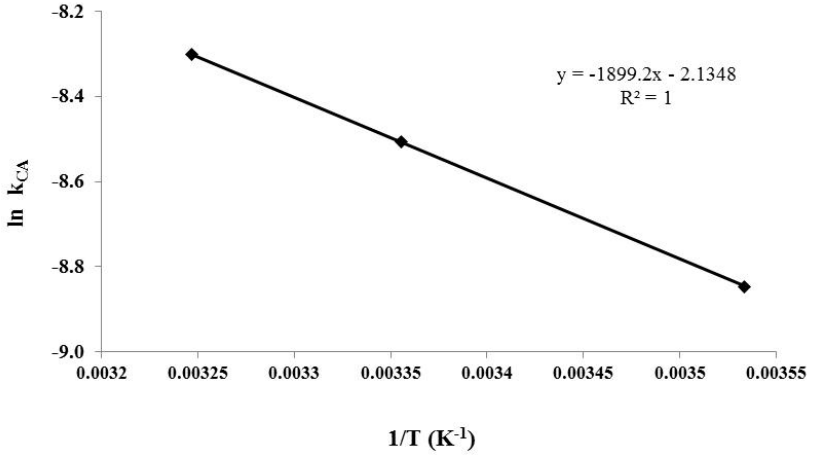

Figure 4: Temperature dependency of carotene degradation rate constant for Arrhenius equation.

When plotting the logarithmic of rate constant values (ln k_{CA}) versus inverse of absolute temperature (1/T), it indicated that the Arrhenius equation has a good correlation coefficient (R^2 » 1.0). By using these regression parameters, the activation energies (E_a), and Q_{10} numbers for the degradation reaction of carotene were calculated. The E_a was found

equal to15.8 kJ mol^{-1}. These values are in agreement with the literature data reporting typical E_a values for carotene degradation (HENRY *et al*., 1998). In the present study, the Q_{10} number for carotene degradation was around 1.0. It indicated that each 10°C increase in storage temperature of carotene enriched mayonnaise, led to the higher carotene degradation.

From the results, we suggest that the cholesterol-free mayonnaise containing red palm olein with high carotene successfully prepares by using whey protein concentrate and modified starch as emulsifiers to replace egg yolk. Moreover, the carotene degradation in mayonnaise products could be controlled by controlling storage temperature.

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CHEMICAL MIGRATION IN MINERAL WATER PACKAGED IN PET BOTTLES AND SENSORY CHANGES DURING THE SHELF-LIFE

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ABSTRACT

Polyethylene terephthalate (PET) bottles are the most widely used mineral water containers. The growth in bottled water is influenced by three public concerns of fears: a declining quality from municipal water supplies; toxic contamination of ground water sources and a general increased interest in personal health. However, very little attention has been paid to the changes in quality of water after prolonged storage in plastic containers. The aim of this research was the determination of volatile migrants from PET bottles to the mineral waters and the correlation of these data with the sensory analysis. The migrants were extracted and analyzed by SPME-GC-MS. The sensory profile of samples was performed measuring the intensity of different descriptors olfactive/flavour, gustative and tactile/chemestetic. Of interest resulted the presence of aliphatic linear aldehydes such as octanal and nonanal which were related to the plastic-like off-odor descriptor. Different amount of migrants during shelf-life resulted in agreement with the sensory results.

Keywords: aldehydes, migration, mineral water, PET, sensory analysis, SPME-GC-MS

1. INTRODUCTION

Water packaged in polyethylene terephthalate (PET) bottles is a large segment of the beverage market. It is well known that food may become contaminated with components of plastic containers by a diffusion process known as migration depending on concentration of substances in package, nature of the foods, temperature and time of contact (BHUNIA *et al*., 2013). The migration could create a sensory problem, other than toxicity. In fact, water packaged in plastic containers develops an off-taste after a short period of storage. This off-taste, often described as "plastic", is most pronounced in water packaged in polyolefin containers, but it is also noted in PET containers especially if closed with a polyolefin closure. This off-taste can be correlated with the presence of aliphatic aldehydes, particularly octanal, nonanal and decanal (STURBE *et al*., 2009; BACH *et al*., 2013).
Most of the papers reported in literature deal with the presence of phthalates (esters of phthalic acid) (CAO, 2010), diethylhexyl adipate (DEHA) (SERÔDIO and NOGUEIRA, 2006), and bisphenol A (BPA) (CASAJUANA and LACORTE, 2003) in mineral water packaged in PET bottles; otherwise limited are the information on the migration of aliphatic aldehydes during the shelf-life (DĄROWSKA *et al*., 2003). The aim of this research is the determination of volatile aldehydes in mineral water packaged in PET during the shelf-life, correlating the results obtained by SPME-GC-MS to the sensory profile of the samples.

2.MATERIALS AND METHODS

2.1. Sampling

Mineral water samples were provided by local producers, immediately after the bottling in PET containers. Thirty bottles of 500 mL were collected from the same batch and stored at room temperature up to five months. Every month instrumental and sensory analysis were carried out on a bottle stored at room temperature (Rt) and one stressed at 60°C for 12 h (St) to simulate the storage effect.

2.2. Volatiles extraction: HS-SPME

Extraction was performed in the headspace vial with a DVB/CAR/PDMS fiber, 50/30 μm film thickness; extraction time, 35 min. Thermal desorption onto the capillary GC column, 3 min.

2.3. Volatiles analysis - GC-MS

A Varian GC-MS instrument equipped with a polar capillary column (CP-Wax 52 CB, 60 m, 0.25 mm i.d., 0.25 μm film thickness) was used. Each compound was identified using mass spectral data, NIST'11 library, linear retention indices (LRIs), literature data and the injection of standards where available.

2.4. Sensory analysis

The definition of the sensory profile of the water samples, according to the method UNI 10957 (2003), was carried out by a panel of 10 trained judges (UNI EN ISO 8586, 2014). The

samples were presented to each judge, randomizing the order of presentation, in a sensory laboratory (UNI EN ISO 8589, 2014).

3. RESULTS AND CONCLUSIONS

Aliphatic aldehydes from C_8 to C_{10}, saturated and unsaturated, were identified and quantified in the mineral water samples (Table 1). The total amount of aldehydes was included between 2.01μg/L (Rt0) and 6.38 μg/L (St5). Decanal prevailed in all the samples, nonanal and (E)-2-nonenal followed. From the statistical elaboration of the data statistically significant increases were observed during the shelf-life both among samples stored at room temperature and among the stressed samples, whereas no significant differences between the samples stored at room temperature and the stressed ones, excluding (E)-2-nonenal and octanal only at t1, with the highest amounts in the stressed samples. The amount of the identified aldehydes was little more superior to their odour threshold in all the samples except for (E)-2-nonenal. These data agree with the literature since it has been demonstrated that the migration of these substances is related to their amount in the bottle wall and directly dependent on temperature and time of storage (BATCH *et al*., 2013).

Table 1: Aldehydes quantified[a] (μg/L) in mineral water sample packaged in PET.

Compounds	LRI[b]	Rt 0	St 0	Rt 1	St1	Rt 5	St5
Octanal	1291	0,23a[c]	0,26a	0,29a	0,34b	0,36b	0,41b
Nonanal	1396	0,4a	0,43a	0,63b	0,64b	0,82c	0,89c
Decanal	1498	1,1a	1,48a	1,95b	2,5b	3,6c	4,2c
(*E*)-2-Nonenal	1457	0,28a	0,38b	0,41b	0,53c	0,76c	0,88d
All		*2,01a*	*2,55a*	*3,28b*	*4,01b*	*5,54c*	*6,38c*

[a] Average quantity.
[b] Linear retention indices calculated on CP-Wax 52 CB column according to Van den Dool and Kratz equation.
[c] Different letters in the same row represent significant differences at $P < 0.05$ by Duncan's multiple range test.

Figure 1 reports the sensory analysis results for the samples at t0, t1 and t5 showing that samples St0 and St1 were significantly different respect to Rt0 and Rt1 for four descriptors: sulfur odor, chlorine odor, medicinal flavor and off-flavor.
The sample St1 showed the highest intensity for all these descriptors, while the sample St0 showed the highest intensity only for medicinal flavor and off-flavor. Samples at t0 vs t5 didn't showed any significant differences between Rt and St except for the descriptor sulfur odor with the high intensity in St5. Plastic odor and plastic flavor descriptors showed the highest intensity in all the St samples. Generally, it has been concluded that the plastic-like off-odor in mineral water arises from aldehydes such as octanal, nonanal.
Lubricant agents, commonly unsaturated fatty acid amides, can oxidize upon exposure to ozone, high temperature and UV light leading to aliphatic aldehydes reported in the literature as the main reason for this off-odor. The differences observed in the intensity of

the plastic odor descriptor could be correlated with octanal, nonanal and decanal since their amount was higher to their odor threshold in all the samples.

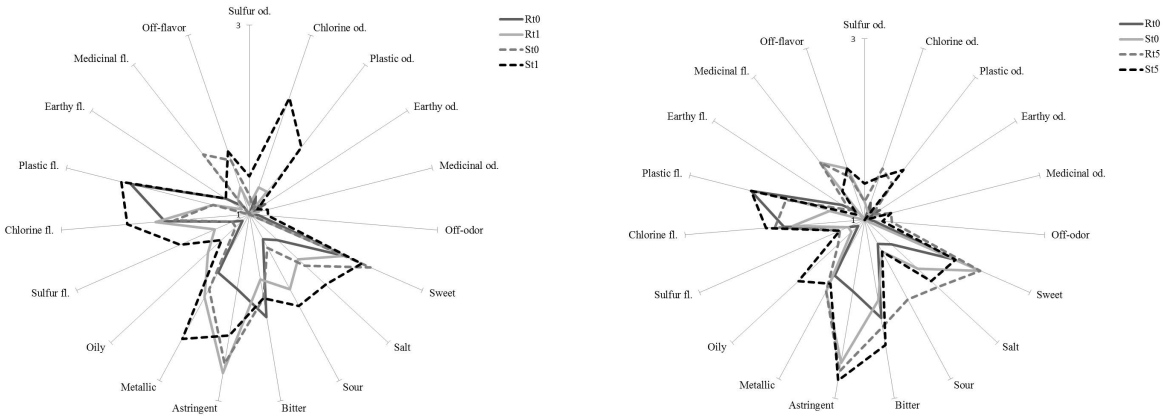

Figure 1: Sensory profile of the mineral water samples during the shelf-life.

This study has highlighted changes of the sensory characteristics of water with an increase of the smell of sulfur and plastic off odor. Volatile compounds analyzed by gas chromatography showed the presence of aldehydes whose amount, although below the legal limits, negatively affected the sensory quality.

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FROM SCREENING ANALYSIS TO EXPOSURE ASSESSMENT: NEW INSTRUMENTS FOR RISK ASSESSMENT EVALUATION ON FOOD CONTACT MATERIAL (FCM)

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ABSTRACT

Screening tests have been introduced by Regulation (EU) N. 10/2011 for alternative compliance test approaches for overall and specific migration. Screening tests can be based either on experimental-analytical testing methods or on theoretical migration estimations via calculation or migration modelling. As a matter of principle, screening approaches need always to be at least as conservative as the verification method. Therefore, test conditions which are at least as severe should be applied. Since, from experience, screening results will be in most cases conclusive concerning positive compliance declaration, screening tests offer advantages over verification methods with regard to time and costs. Screening tests can be performed stepwise within a gradual approach system, starting from the assumption of total mass transfer via very quick and cheap extraction tests on food contact materials and determining the residual content with more refined tests and migration consideration. In case of substances which represent a risk for human health, it is necessary to verify the migration of substances into the food in the real condition of use. Therefore food consumption information must be collected and provided detailed intake and exposure estimates crucial for risk assessment analysis.

Keywords: Exposure evaluation, risk assessment, screening analysis approach

1. INTRODUCTION

Considering that screening analysis revealed a large number of residual and potentially contaminating substances, it can be considered an effective risk assessment technique, since it allows to select critical compounds with excessive migration (over 60mg/kg). Their highest nominal concentrations are used for calculations as worst cases, according to the assumption that the maximum migration can be calculated by assuming total migrant transfer for the given material thickness at the appropriate surface-to-volume ratio.
It was decided to apply screening approach to evaluate the compliance of multi material multilayer films for food packaging application.

2. MATERIALS AND METHODS

Analyses were conducted for the purpose of determining the compounds subject to restriction according Annex I of Reg. UE 10/2011 and to investigate other residual volatile and nonvolatile contaminants. The analyses have been carried out under GC conditions listed in the Table 1 - GC analyses operative condition for research of volatile contaminants, and Table 2 - GC analyses operative condition for research of nonvolatile contaminants.

Table 1.

GAS CHROMATOGRAPH AGILENT 7890	
Column	Agilent HP5-MS 30mx0,25mmx1,0μm
Autosampler	Gerstel Head Space MPS
Injection volume	1500 μl
	T°initial 40°C x 2 min
	Ramp at T1 60°C in 5°C/min
Temperature program	Ramp at T2 90°C in 7,5°C/min
	Ramp at T3 190°C in 10°C/min
	Ramp at T4 290°C in 20°C/min
Total	25 min
Injection mode	Split 1:10
Injection temperature	250°C
Carrier flow	Helium at costant flow 1,7 ml/min
MASS SPECTROMETER AGILENT 5975C	
Acquisition mode	SCAN
Acquisition range	m/z=33 to m/z=550
Delay	1,5 min
LOQ	0,1 mg/kg
Calibration	Pool of volatile substances

Table 2.

GAS CHROMATOGRAPH AGILENT 7890	
Column	Agilent HP5-MS 15m x 0,25mm x 0,1μm
Autosampler	Gerstel MPS
Injection volume	1 μl
	T°initial 60°C x 0 min
	Ramp at T1 105°C in 30°C/min
Temperature program	Ramp at T2 170°C in 10°C/min
	Ramp at T3 295°C in 15°C/min
	Final Isotherm at T 295°C in 8.67 min
Total	25 min
Injection mode	On column
Carrier flow	Helium at costant flow 0,9ml/min
MASS SPECTROMETER AGILENT 5975C	
Acquisition mode	SCAN
	m/z=33 to m/z=400 until 10 min
Acquisition range	m/z=33 to m/z= 500 from 10 to 15 min
	m/z=33 to m/z= 750 from 15 min
Delay	1,5 min
LOQ	5 mg/kg
Calibration	Internal STD Methyl Heptadecanoate

3. RESULTS AND CONCLUSIONS

The screening analyses results are shown in the Table 3 - Volatile residual substances detected by HS-GC-MS analyses and Table 4 - Substances extracted from the plastic matrix. A substance emerged from analyses could be represent a risk for human health, so it has been decided to evaluate the specific migration limit via theoretical calculation collecting safety data on substance and food consumption data in order to estimate relative exposure for different population.
Specific migration evaluation of substances detected by screening analyses is shown in Table 5.

Table 3.

	Volatiles HS-GC-MS 125°C 30 min		**FILM**	
RT min	**COMPOUND**	**Average mg/kg**	**s.t.**	**s.t. %**
3.10	Ethyl acetate	19.8	2.2	11
21.15	N.I. (m/z 176, 55, 99, 84, 110)	0.4	0.02	5.2
4 ÷ 15	BAH C8 ÷ C12	14.8	0.6	4.0

Legend. BAH CX = Branched Aliphatic Hydrocarbons with X carbon atoms.

Table 4.

RT min	EXTRACTION IN ULTRASOUND BATH 60 ° C 24 HOURS COMPOUND	FILM Average mg/kg	s.t.	s.t. %
3.30	Penta methyl heptane	14	2.2	15
9.08	Adipic acid ester	21	1.3	6.4
9.63	1-isocyanato-3-isocyanatomethyl-3,5,5-trimethylcyclohexane (CAS 4098-71-9)	31	1.4	4.4
10.09	N.I. (m/z 173,55,99,84,111,155)	45	4.8	11
12.74	prob. phthalate N.I. (m/z 149,193,104,76,132)	57	7.3	13
14.84	tributyl prop-1-ene-1,2,3-tricarboxylate (CAS 7568-58-3)	16	1.6	9.8
15.43	Acetyl TriButyl Citrate (ATBC)	1183	12	1.0
17.79	erucamide	18	1.7	9.6
18.02	N.I. (m/z 215,55,129,111,343,428)	40	1.6	4.0
19.38	N.I. (m/z 173,99,55,389,155)	19	2.4	13
19.65	N.I. (m/z 235,149,104,69,363,448)	30	1.7	5.8

Table 5.

COMPOUND	Ref.	CAS	SPECIFIC MIGRATION mg/kg	LMS mg/kg
ethyl acetate	30140	0000141-78-6	< 0.1	60
Branched aliphatic hydrocarbons C8 ÷ C12	'---	'---	< 0.1	60
adipic acid ester	'---	'---	< 0.1	(*)
1-isocyanato-3-Isocyanatomethyl-3,5,5-trimethylcyclohexane (CAS 4098-71-9)	19100	0004098-71-9	11 (**)	1 (**)
tributyl prop-1-ene-1,2,3-tricarboxylic (CAS 7568-58-3)	'---	0007568-58-3	< 0.1	(*)
Acetyl Tributyl Citrate (ATBC)	93760	0000077-90-7	0.6	60
erucamide	52720	0000112-84-5	< 0.1	60

(*) : not identified. For not identified peaks, the higher mass peaks detected are reported in brackets.
(**) : maximum residual quantity in the object.

The food contact material has a residual content of 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethylcyclohexane (CAS 4098-71-9) equal to 31 mg / kg of total and 11 mg / kg if intended as isocyanate group (NCO), in excess of the amount permitted in the regulations in force of 1 mg / kg (Reg. (EU) No 10/2011, Annex I, Table I, Ref. 475). The material constitutes the top film of a sales units containing fatty foods with high power extraction, it was decided to proceed with the assessments of the risk of food contamination and relative ingestion by the consumer, on the basis of bibliographic data in this document and assumptions assumed to simulate the worst case scenario.

Whereas the application in terms of food contact of the material that goes to form a top film of which weight is about 4 g, comparing the residual content found in the analysis GC-MS to the weight of the object are obtained the following amount of contaminants in the Table 6 - Residual content of critical substance in FCM article.

Table 6.

COMPOUND	Ref.	CAS	Residual content detected via screening analysis on the FCM in mg/kg	Residual content of single top film in mg/object
1-isocyanato-3-Isocyanatomethyl-3,5,5-trimethylcyclohexane (CAS 4098-71-9)	19100	0004098-71-9	31	0.124

Due to the lack of available data on oral exposure scenario, therefore it was decided to use the US data to CEDI (Cumulative Estimate Daily Intake) for exposure assessments, on the basis of the following assumptions:

1) the entire residual content of 1-isocyanato-3-isocyanatomethyl-3,5,5-trimethylcyclohexane (CAS 4098-71-9) on top film is absorbed by the packed fatty food (220 g)
2) all the contaminated food product is taken by the same person of 60 kg body weight
3) the acceptable daily intake of food additives or contaminants in food is based on the assumption of a substance daily during the life cycle of an individual, and whereas it is unlikely that a consumer is exposed to the same contaminant in equivalent amounts every day, using this conservative approach, considered sufficient to protect consumers, to assume the risk resulting:

if we consider samples of membranes found not to comply, and if we assume that an individual's consumption of 60 kg 70 grams of food to die (source EFSA Food Consumption Database – Composite Foods A.01.001789-Adults population-INRAN survey), this would assume full 0,039 mg / day. This amount is lower than the value of CEDI consulted which is defined as equal to 0.00093 mg /kg bw / day resulting in no more than 0.00065 mg / kg bw / day.
Screening tests appear to be a valid method to prove compliance of the food contact materials and above all to provide a lot of information about potential residual contaminants to take into consideration in risk assessment evaluation. The methodology used is affected by uncertainties but it can help steer the evaluation of food contamination by residual NIAS or IAS, and to establish the safety of the material to be used for food application in function of the reference population.

SHELF LIFE EVALUATION OF SWEET BAKERY FOODS: TWO CASE STUDIES

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ABSTRACT

The shelf life of two bakery products was evaluated under real storage conditions. Amaretti cookies and Savoiardi biscuits were purchased from 2 local plants and stored in their pack at 20°C. Chemical-physical and texture parameters, and sensory attributes were checked at regular intervals.
The sensory analyses allowed to determine the shelf life of Amaretti and Savoiardi, as panellists discriminated fresh from stored samples. Texture data obtained at the acceptability limit will be considered in future studies of shelf life evaluation of these two products.

Keywords: Cookies, sensory analysis, shelf life, texture analysis

1. INTRODUCTION

The food quality deterioration can be followed by checking microbiological, chemical, nutritional and sensory attributes (SINGH, 2000; NICOLI, 2012). In order to estimate the shelf life of a product is fundamental to define the level to which the quality modification is acceptable over time. The identification of the critical descriptors and of the relevant acceptability limit are of paramount importance before and after monitoring of quality loss under simulated conditions (real or accelerate) (KILCAST AND SUBRAMANIAN, 2011; CALLIGARIS and MANZOCCO, 2012). To adequately follow all these aspects it is necessary to know the food characteristics (ingredients, processing, chemical-physical and microbiological attributes, packaging, distribution and consumption methods).
The aim of this work was to determine the actual shelf-life of the Amaretti cookies and Savoiardi biscuits, in order to extend it, in future studies, through packaging or mild formulation changes. The critical descriptor of both cookies is hardening, due to water loss or redistribution.

2. MATERIALS AND METHODS

Cookies and biscuits were manufactured and packaged by 2 local plants. An adequate number of samples was stored at 20°C and evaluated at regular intervals for chemical-physical, texture and sensory parameters. Chemical-physical attributes were dry matter (%) and water activity (a_w) and were obtained with routine laboratory methods in triplicate.
Texture analysis (cut test) involved checking for hardness changes on the top and bottom parts of 10 cookies with a TA.XT plus (Stable Microsystems, Surrey, UK) texture analyzer equipped with a 35 kg load cell. A blade set with knife edge (mod. HDP/BS) and a slotted blade insert, which acted as a guide for the blade were used. Graphs obtained were used to calculate 3 hardness indexes: the maximum force (N) reached during the text; the area (N s^{-1}) calculated starting from the starting point to the maximum force; gradient or stiffness (N s^{-1}) calculated from the start of the reading up to the maximum force.
Thirty untrained consumers evaluated the product with an acceptability and triangle tests. For the first test an overall acceptability score was asked for all the inspection times on a scale from 1 to 7 (with 4 as the minimum acceptability score). The triangle test served to check for differences between the stored and freshly prepared samples. Three samples were presented to consumers and they were asked to recognize the different one. Results were expressed on the basis of right replies to wrong ones.

3. RESULTS AND DISCUSSIONS

Consumers were able to distinguish fresh from stored samples along the whole storage period with the triangle test and gave the minimum acceptability score at 15 days for Amaretti cookies and at 30 days for Savoiardi biscuits.
Data obtained from textural test for the above cited shelf life periods have been considered as the objective limit for evaluating the shelf life period in future studies and are reported in Table 1.

Table 1: Texture data associated with the minimum acceptability score as assessed by sensory analysis.

Sample	Texture Index					
	Maximum force (N)		Area (Ns^{-1})		Gradient (Ns^{-1})	
	Top[a]	Bottom	Top	Bottom	Top	Bottom
Amaretti	46.33± 6.92	33.74± 2.89	524.7±70.57	45.6±6.53	2.54±0.39	12.72± 2.63
Savoiardi	11.31±1.46	12.88±1.62	114.7±13.91	104.7±12.43	0.54±0.12	0.72±0.06

[a]Each value is the mean of 10 determinations plus or minus standard deviation.

Appropriate sensory analysis permitted to determine the shelf life of both bakery products.
Sensory results were associated with objective data obtained by texture analysis, which will be used in the future to estimate the shelf life of these 2 products.

ACKNOWLEDGEMENTS

This research was supported by Ministero dell'Istruzione, dell'Università e della Ricerca (Prot. 957/ric , 28/12/2012), through the Project 2012ZN3KJL "Long Life, High Sustainability".

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QUALITY PARAMETERS OF WHOLEGRAIN DURUM WHEAT BREAD ENRICHED WITH CITRUS FIBRE

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ABSTRACT

This paper reports the results of the quality parameters and the sensory evaluation determined on a durum wheat wholegrain breads enriched with citrus fibre. Wholegrain breads enriched with 2.0% of citrus fibre were produced in an industrial bakery, packaged under MAP conditions and stored at 25°C for as long as 90 days. Morphometric (volume, height, weight, porosity, crust and crumb color) and chemical parameters (pH, acidity, HMF, moisture) were determined to study the effect of the addition of citrus fibre in wholegrain bread. Results showed that the addition of 2.0% citrus fiber does not imply any deterioration in the appearance of bread, nor a reduction in the quality parameters. A trained panel of 12 judges determined the sensory profiles of bread samples. The results of the sensory data highlighted that the samples differed in citrus flavour but the overall evaluation were similar. The addition of citrus fibre to durum wheat flour could be considered a promising and innovative way to obtain functional bread rich in fibre.

Keywords: *Triticum turgidum* L. ssp. *turgidum convar. durum*, wholegrain bread, physical properties, shelf-life, sensory evaluation

1. INTRODUCTION

The high nutritional value of dietary fibre is well documented and is widely accepted its role in the prevention of certain diseases and benefits to human health (RODRIGUEZ *et al*., 2006). Health benefits associated with dietary fibre include: blood glucose and cholesterol attenuation, protection against cardiovascular diseases, regulation of intestinal functions and promotion of gut health, protection against colon cancer (KING, 2005). WHO recommends on average a portion of 30 g of dietary fibre to be daily consumed. However, according to the European nutrition and health report, in most of European countries the dietary fibre intake is lower than the recommended level.
In southern Italy, and particularly in Sicily, the citriculture produces about 600.000 t/year of 'pastazzo', by-products derived from the citrus industry. It is possible to obtained citrus fibre from citrus residues, after numerous washing and purification steps, which can be added into food products. The addition of fibre to baked products lead to a reduction of loaf volume, hard crumb, a bitter flavor and a dark bread color (KTENIOUDAKI and GALLAGHER, 2012).
The present work shows the results of a study aimed at the production of bread enriched with citrus fibre in order to obtain a product with functional properties. The quality parameters of durum wholemeal wheat bread were evaluated. Loaf height and weight, porosity, moisture content and sensory changes were determined during shelf life.

2. MATERIALS AND METHODS

2.1. Bread making process

Breads were produced in an industrial company (Valle del Dittaino Società Cooperativa Agricola, Assoro, Enna, Italy), according to a consolidated industrial process and packaged under MAP conditions and stored at 25 °C up to 4 months. For each dough, 50 kg of durum wheat wholegrain flour was mixed with water, compressed yeast, salt and 2.0% of citrus fibre.

2.2. Bread quality parameters

The volume was determined according to the rapeseed displacement in a loaf volume meter; the loaf height was measured with a digital caliper (Digi-Max™, Scienceware®, NJ, U.S.A.). The CIE L*a*b* colour parameters were measured, using a Minolta spectrophotometer CR-300 (Minolta, Japan), for the crumb, in the transversely cut bread and on the crust surface, averaging 10 distinct points in each case.
Moisture content was determined on grounded samples by gravimetric method.

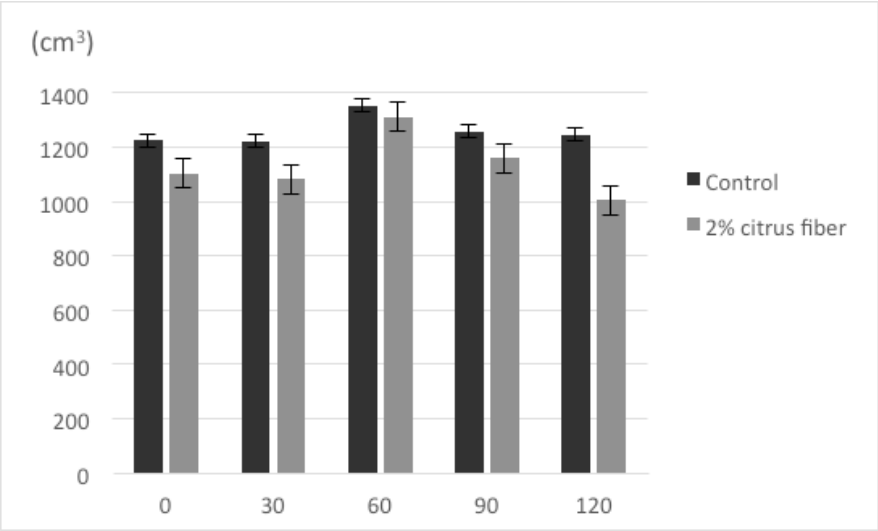
2.3. Sensory evaluation

The sensory profile was determined by applying the methods UNI 10957 2003 and UNI EN ISO 8586 2008 according to LANZA *et al*. (2011). The judges, using a scale between 1 (absence of the sensation) and 9 (extremely intense), have evaluated the intensity of the selected sensory attributes.

3. RESULTS AND CONCLUSIONS

With regard to the loaf volume, no significant differences were recorded among the bread samples and during the storage time (Fig. 1a). Bread with 2.0% of citrus fibre had a lowest loaf height (Fig. 1b). The addition of small quantities of citrus fibre on durum wheat whole flour has not increased the bread yield. Bread containing 2.0% of citrus fibre had the lowest lightness (L*) values of the crumb.

a)



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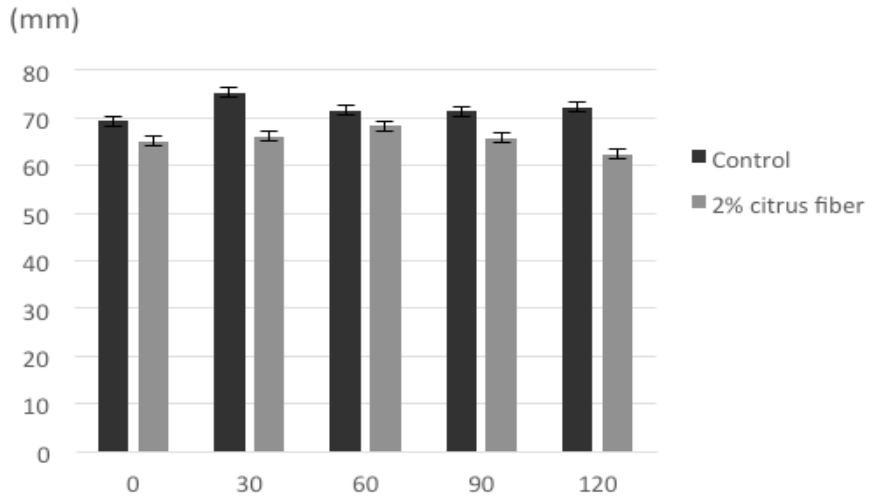

Figure 1: Effect of citrus fibre addition on physical properties of bread during storage.
a) Loaf volume; b) loaf height.

Moisture content in control bread ranged from about 38% in the freshly baked to 23% after 120 days of storage. Bread added with 2.0% of citrus fibre had the highest moisture level ranging from 42% in the freshly baked to 33% at the end of storage period. In both bread

samples at 60 days of storage, moisture content decreases quickly. At 90 and 120 days of storage, no remarkable differences were evidenced among bread samples. SPINA *et al*., (2015) reports a similar trend of moisture content in durum wheat bread during storage.
Sensory evaluation performed on the freshly baked bread samples highlights that control bread had the highest intensity of bread flavor, softness, moisture and overall judgment, while bread added with 2.0% of citrus fibre had the highest intensity of yeast odor, salty, off flavor and crumb color (Fig. 2). During storage, samples were statistically different for the citrus odor and flavor attributes (Fig. 2).
The results of this study demonstrated that the addition of citrus fibre to durum wheat flour could be considered a promising and innovative way to obtain functional bread rich in fibre.

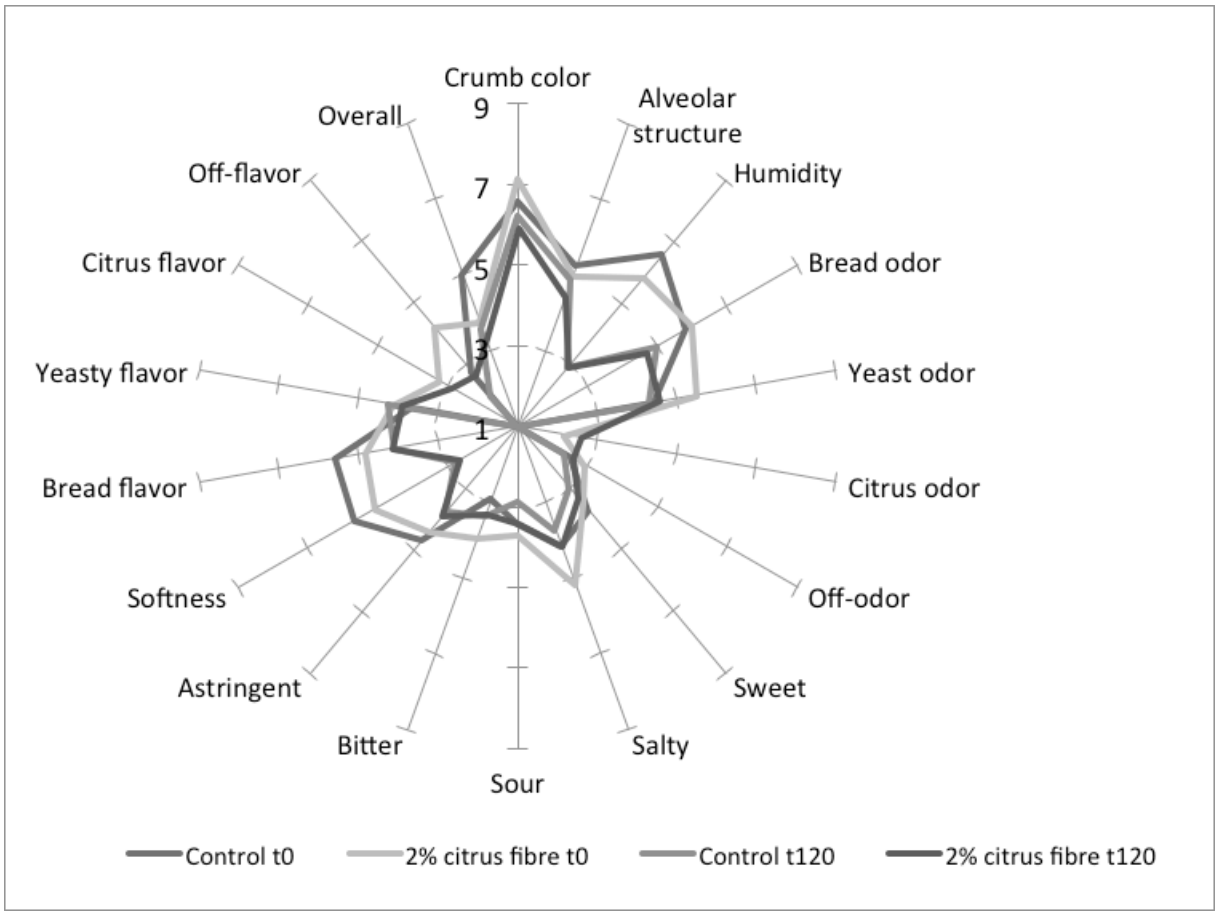

Figure 2: Evaluation of the sensory attributes in freshly baked bread samples and at 120 days of storage.

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DURUM WHEAT BREADS ENRICHED WITH CITRUS FRUITS PECTIN AND FLAVONOIDS

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ABSTRACT

The present work shows the results of a study aimed at the production of bread enriched with citrus fruit pectin and flavonoids in order to obtain a product with functional properties. The first phase of the work was focused on the qualitative characterization of durum wheat wholegrain flour and citrus fruit pectin and flavonoids. The second phase was focused on the production of bread in industrial bakery with wholemeal flour enriched with 1.0% of pectin and/or 2.0% of citrus flavonoids. Chemical (pH, total acidity, moisture) and morphometric (volume, height, weight, porosity, crust and crumb color) parameters were determined to study the effect of the different level of enrichment on the final product and during storage. Results showed that the addition of citrus flavonoids, associated with pectin have shown a better bread volume and improved the other quality parameters. Moisture content was comparable between bread samples independently from the levels of pectin and flavonoids. The results obtained indicate that the addition of citrus fruit pectin and flavonoids to durum wheat flour could be considered an innovative way to obtain functional breads.

Keywords: wholegrain flour, durum wheat bread, flavonoids, shelf-life, pectin

1. INTRODUCTION

Dietary fibers and polyphenols are recognized as active nutrients responsible for the health benefits of fruit and vegetables. Interest in incorporating bioactive ingredients such as dietary fiber and flavonoids into popular foods like bread has grown rapidly, due to the increased consumer health awareness.
Many attempts have been made to increase the nutritional value of bread by adding dietary fibre and/or other bioactive compounds as for example grape seeds, pomegranate peel powder, apple pectin and lemon pomace fiber (ALTUNKAYA *et al*., 2013; CHANG *et al*., 2015; MERAL *et al*., 2013; SIVAM *et al*., 2011). Nevertheless, fiber enrichment is usually associated with various technological problems, and there is the need to optimize the composition of the blends employedwithout impairing the technological quality of the final product (ŠKARA *et al*., 2013).
The aim of this work was the evaluation of citrus flavonoids (F) and citrus pectin and flavonoids (FP) addition on the properties of durum wheat bread. Breads were subjected to quality and chemical evaluation (attributes including moisture, color, porosity, weight and volume) during 90 days of storage.

2. MATERIALS AND METHODS

2.1. Bread making process

Bread samples were produced in an industrial bakery (Valle del Dittaino - Agricultural Cooperative Society a.r.l.,Assoro, Enna, Italy), packaged under MAP conditions using 70 % N_2:30 % CO_2 gas combination and stored at 25 °C up to 3 months. For each dough 50 kg of durum wheat semolina was used and mixed with tap water and compressed yeast and addedwith flavonoids and pectin. Code for samples and composition of flavonoids and pectin of dough were reported in Table 1.

Table 1: Code for samples and composition in flavonoids and pectin of the dough.

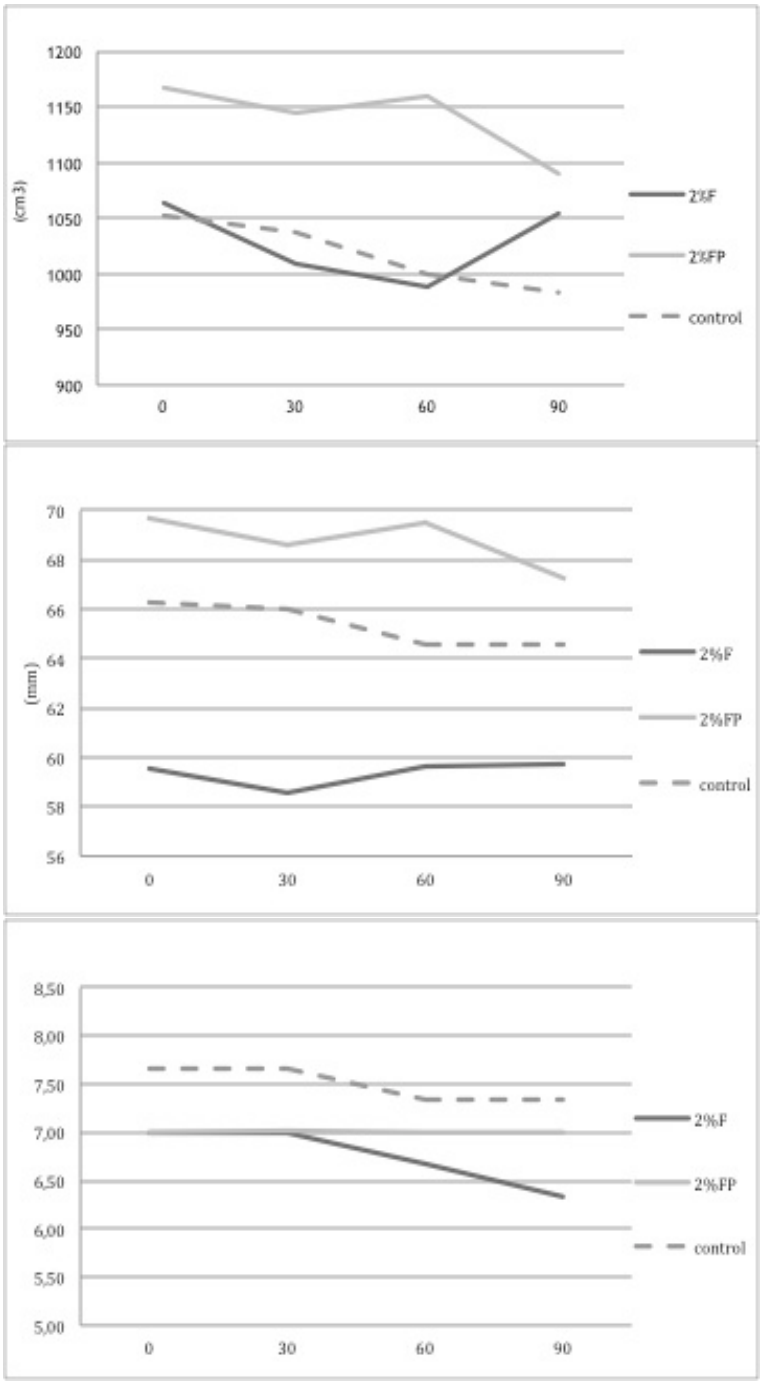
Sample code	Citrus flavonoids (g/100 g)	Citruspectin (g/100 g)
Control	-	-
2% F	2	-
2% FP	2	1

2.2. Bread quality parameters

The volume was determined according to the rapeseed displacement in a loaf volume meter; the loaf height was measured by a digital caliper (Digi-Max™, Scienceware®, NJ, U.S.A.). The internal structure was visually estimated by eight evaluator subjects, and the crumb porosity was estimated using the Mohs scale, complemented by a DALLMANN (1958), based on the analysis of visual and subjective 8 photos representing different cross sections of loaves with different porosity. Crumb color was measured by Minolta CR 300Colorimeter.
Moisture content was determined on grounded samples by gravimetric method. pH and total titratable acidity were determined according to SPINA *et al*. (2015).

3. RESULTS AND CONCLUSIONS

Regarding bread volume (Fig. 1a) FP bread samples showed, during the entire storage period, a higher bread volumecompared to control and F samples considering the structuring action of the soluble fiber (pectins). Similar trend was observed for loaves height (Fig. 1b) as the two parameters are usually related. In this case, however, the F bread sample reported during the entire storage period less voluminous loaves, probably due to the inhibitory effect on microorganisms of flavonoids during fermentation.



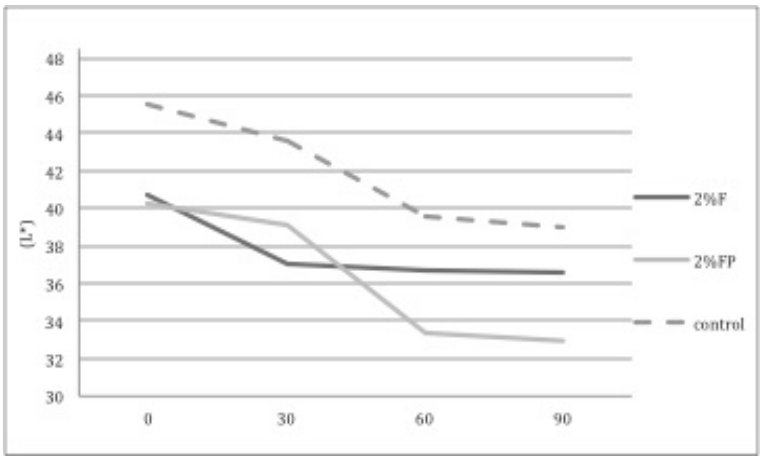


Figure 1: Effect of pectin and flavonoid addition on physical properties of bread during 90 days of storage. a) Loaf volume (cm^3); b) Height (mm); c) Porosity (8); d) Lightness (L*).

As regards for the porosity (Fig. 1c) the control bread showed a limited development of the alveo (value 7.5) during 90days of storage, while the other samples, up to 30 days, had an equal performance (value 7). At 60 days of storage bread sample with flavonoids (F) maintained a better porosity (value 6.5).
With regard to crust color (Fig. 1d), F and FP bread samples have shown lower values of Lightness (L*) compared to the control due to the effect of the flavonoid addition. During storage a decrease of crust brightness in all bread samples, as expected, was determined.
Moisture content in the control bread and in FP bread samples was about 38% (Fig. 2).

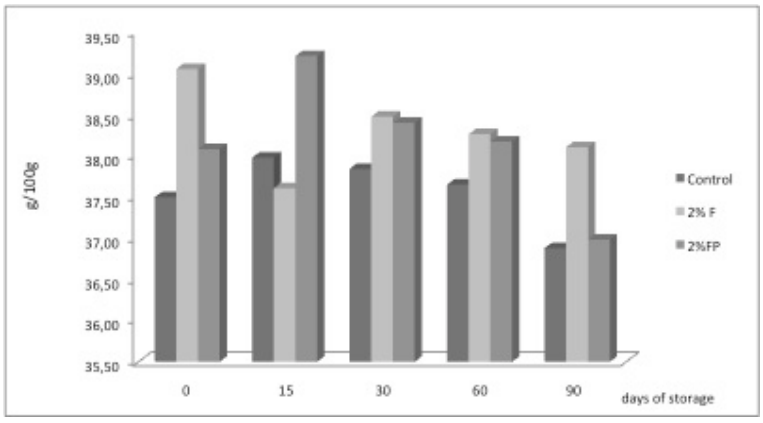


Figure 2: Evaluation of the moisture content of the bread samples during storage.

During storage, moisture levels remain almost constant in all bread samples but generally, control bread had the lowest moisture level, indicating a favourable effect induced by the addition of pectin and flavonoids on the ability to retain water by the dough.pH and total acidity of bread samples were similar independently from the presence of the flavonoids and pectin. pH in fresh samples was about 6.1, and remained almost constant during storage, as well as total titratable acidity.

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USE OF A NATURAL LOW Na^+ SALT IN DURUM WHEAT BREAD

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ABSTRACT

Processed foods are the main source of sodium in the diet and bread is one of the main contributors to dietary sodium intake, especially in countries where the bread has an important role in the daily menu. The aim of this work was the evaluation of the effects of the reduction of sodium content and its replacement with a natural low Na^+ salt on the quality parameters of durum wheat [*Triticumturgidum* (L.) subsp.*turgidum* (L.) convar. *durum* (Desf.)] bread. Breads were produced in an industrial company, packaged under MAP conditions and stored at 25 °C for 90 days. The physicochemical characteristics of the bread samples and the sensory changes were evaluated to highlight the products evolution during the shelf life. The loaf volume and height, the crumb porosity, the moisture content and the pH were determined in bread samples at different time of storage. Sensory changes were also studied to understand the effect of the Na^+ reduction on the major bread attributes. Loaf volume and height showed, as expected, a decrease of these parameters correlated with the reduction of salt concentration. The moisture content did not show differences during storage, while HMF showed the typical fluctuating trend independently from the salt level of the bread samples. The results of the sensory data showed that breads differed in salt attribute, as expected, and had similar trends during storage.

Keywords: durum wheat bread, potassium chloride, shelf-life, sensory evaluation

1. INTRODUCTION

The negative effects of an excessive salt intake with the diet, today, are well known. EFSA (2006) recommended an intake of 3-4 g salt/day, but generally, among European population the daily salt intake is more than double. One of the several initiatives undertaken for the reduction of the salt intake with breads was from the Italian Ministry of Health in 2007, who signed with various bakery associations a progressive reduction of the salt content in bread. NaCl plays an important role in bread-making. Decreasing salt addition causes a reduced dough resistance to extension, changes in gas holding capacity of the dough, influence on crumb structure and hardness during storage (LYNCH *et al*., 2009). A partial replacement of NaCl with KCl and yeast extract in durum wheat bread as a possible strategy to reduce salt intake, was proposed by SPINA *et al*. (2015). The NaCl replacement with KCl higher than 30% gives metallic and astringent taste (SALOVAARA, 1982). The aim of this work was the evaluation of the effects of the sodium chloride reduction, from 1.7% to 0.15%, and its replacement with a natural low Na^+ salt on the quality parameters of durum wheat bread. Loaf height and weight, porosity, moisture content and sensory changes were determined during shelf life.

2. MATERIALS AND METHODS

2.1. Bread making process

Breads were produced in an industrial company (Valle del Dittaino Società Cooperativa Agricola, Assoro, Enna, Italy), according to a consolidated industrial process and packaged under MAP conditions and stored at 25°C up to 90 days. For each dough 50 kg of durum wheat semolina was used and mixed with water, compressed yeast and added with different level of NaCl (1.7, 0.15%) or natural low Na^+ salt (1.7, 0.15%). Code for samples and the composition of the salts of dough were reported in Table 1.

Table 1: Code for samples and composition of the salts of the dough.

Sample code	NaCl (g/100 g)	Low natural Na+ salt (g/100 g)
1.7% NaCl	1.7	-
1.7% Lns	-	1.7
0.15% NaCl	0.15	-
0.15% Lns	-	0.15

2.2. Bread quality parameters

The volume was determined according to the rapeseed displacement in a loaf volume meter. The internal structure was visually estimated using the Mohs scale. Moisture content was determined on grounded samples by gravimetric method. HMF extraction and HPLC conditions were according to SPINA *et al*. (2015).

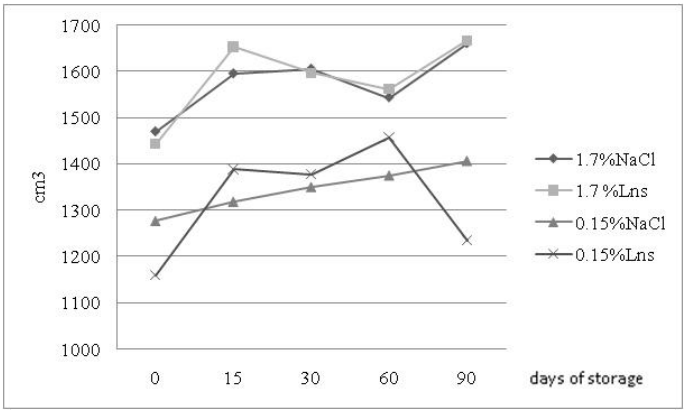
2.3. Sensory evaluation

The sensory profile was determined by applying the methods UNI 10957 2003 and UNI EN ISO 8586 2008 according to LANZA *et al*. (2011). The judges, using a scale between 1 (absence of the sensation) and 9 (extremely intense), have evaluated the intensity of the selected sensory attributes.

3. RESULTS AND CONCLUSIONS

Bread samples (1.7% NaCl and 1.7% Lns) reported similar values of loaves volume (Fig. 1a), independently from the type and content of salt used. On the contrary, bread samples (0.15% NaCl and 0.15% Lns) reported a reduction of about 300 cc. After 60 days of storage, a decrease in loaves volume was reported only in 0.15% Lns samples.
As regards to the bread weight, loaves samples (1.7% NaCl and Lns) showed an average weight between 450 and 500 g, maintaining it until the end of storage (Fig. 1b). Concerning the internal structure bread samples showed a variable trend during the 90 days of storage (Fig. 1c).

a)



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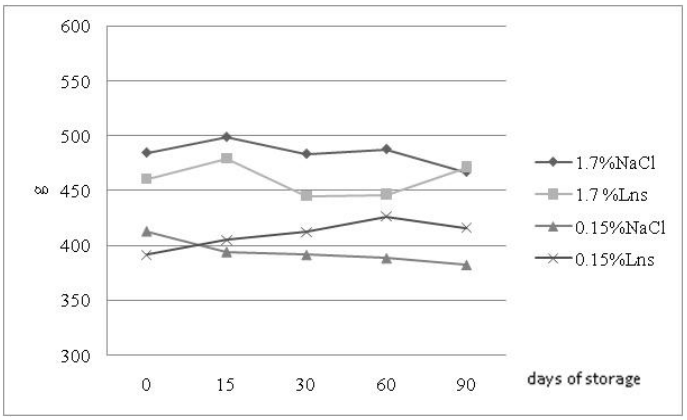

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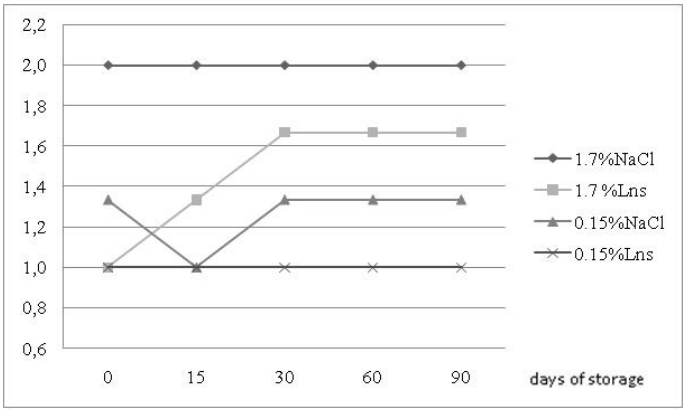


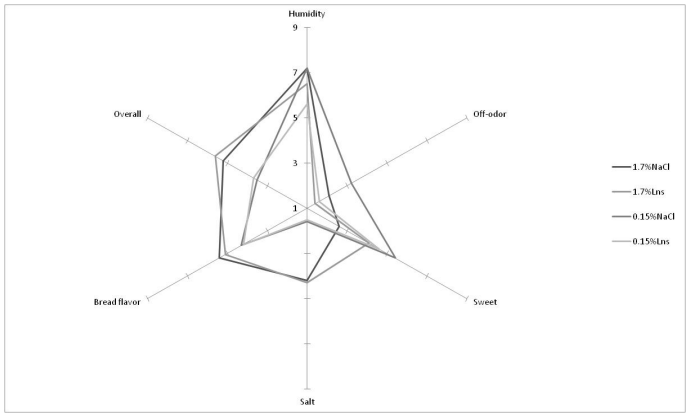
Figure 1: Effect of different salt concentration on physical properties of bread during 90 days of storage. a) Loaf volume (cm^3); b) Weight (g); c) Crumb structure (1: irregular, 2: regular).

Moisture content was about 34% in bread samples with 1.7% of salts, while bread samples with 0.15% of salts had the lowest moisture content (about 32%). During storage the moisture content remained constant in all samples, suggesting a good performance of the packaging materials.

HMF is a well-known index of thermal treatments and its formation is related to the presence of sugars in the dough and time and temperature of the oven baking (RAMÍREZ-JIMÉNEZ *et al*., 2000). The HMF levels in freshly baked bread samples ranged from approximately 23 mg/kg to approximately 39 mg/kg, but it is not possible to attribute these differences in the HMF levels between samples nor to salts content neither to NaCl replacement. Probably other factors such as slight differences in the oven baking time influences more the HMF content respect to the different salt levels in the dough. After three months of storage the HMF level in all bread samples was lower than the concentration determined before storage.

Sensory evaluation performed on the freshly baked bread samples highlight a comparable overall judgment of breads having the same levels of salts, indicating no off-odour development in bread produced with the natural low Na^+ salt (Fig 2a.).

a)



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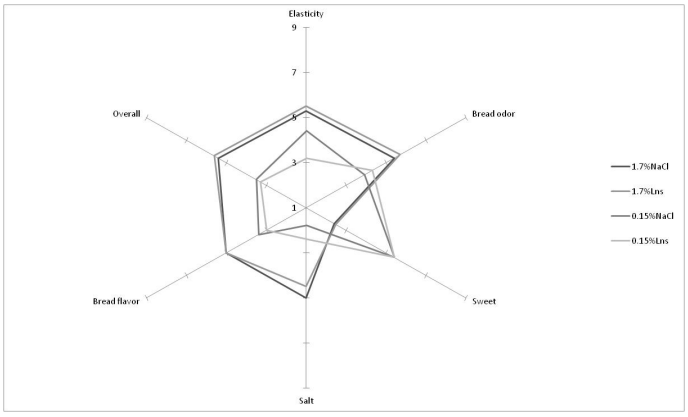

Figure 2: Evaluation of the sensory attributes in freshly baked bread samples (a) and after three months of storage (b).

Bread samples with the highest salt level (1.7%) had the highest intensity of bread flavor while bread samples with the 0.15% of salts had the lowest intensity of the same attribute. After 90 days of storage the overall judgment, the bread flavor, the bread odor and the elasticity were similar between bread samples having the same salt level (Fig. 2b).

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SESSION IV

"Long Life, High Sustainability through Shelf Life Modelling"

FOOD SAFETY AND SHELF LIFE MODELLING FOR A BETTER DIMENSIONING OF THE FOOD/PACKAGING SYSTEM

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ABSTRACT

Food shelf life is mainly related to the dynamic of mass transfer in the food / packaging system. For instance, microbial safety of fresh products in Modified Atmosphere Packaging (MAP) implies to take into account the dynamic of O_2 and CO_2 exchanges in the system and its effect on microbial growth. Fresh fruits and vegetable decay is related to respiration rate that could be minimized using packaging material with the suitable permeation properties. We developed mathematical models of gas transfer (permeation through packaging and solubilisation/diffusion within food) coupled with models of food shelf life such as predictive microbiology or respiration. These mechanistic models, once validated, and connected to dedicated food and packaging databases, could be used as a Decision Support Tool (DST) in order to optimize the storage conditions of the product or to identify the packaging material the most suitable for the product.

Keywords: Decision Support Tool, Food Shelf life, Mass Transfer, Mathematical Modelling, Modified Atmosphere Packaging

1. INTRODUCTION

Half of the fresh fruit and vegetable production is lost before consumption and most of these losses occur during distribution/consumption, when packaging is involved (GUSTAVSSON *et al.*, 2011). There is a huge effort to put on the development of eco-efficient packaging solutions able to extend food shelf life and consequently reduce food losses. Among all possibilities, Modified Atmosphere Packaging (MAP) could really help to improve food quality during storage and extend food shelf life (FLOROS and MATSOS, 2005). Food degradation and thus shelf life is mainly related to gas exchange in the food/packaging system such as microbial growth which is strongly related to O_2 and CO_2 exchanges in the system (CHAIX et al., 2015) or oxidation which is definitively related to the level of O_2 ingress inside the pack (PÉNICAUD *et al.*, 2011) or respiration of fresh fruits and vegetable that relies on the maintaining of an optimal O_2 and CO_2 concentrations in headspace (FLOROS and MATSOS, 2005). In all the aforementioned cases, the choice of a packaging material with the suitable permeation properties that permit to either maintain the gas mixture at its initial level (active MAP) or favour gas exchange to ensure aerobic metabolism (passive MAP) is crucial. However, the choice of a packaging material for a given application still relies on an empirical approach (or "pack and pray" approach) where the user arbitrary chooses his packaging material tests it on his product (Fig. 1). To avoid this time and cost consuming approach, it would be preferable to use, instead, a requirement driven approach starting from the needs and requirements of the food translated into mass transfer properties for the packaging material. Once these properties are known, they are considered as constraints in the choice of the packaging material. This optimized approach, based on the use of virtual MAP modelling tools permits to avoid over-tests and to better target the packaging dimensioning.
In this context, the present paper aims at illustrating the basements of a requirement driven approach in the field of food packaging. Two examples of mechanistic models developed by our group, coupling mass transfer and reactions in the food/packaging system will be presented and used to define the targeted packaging mass transfer properties.

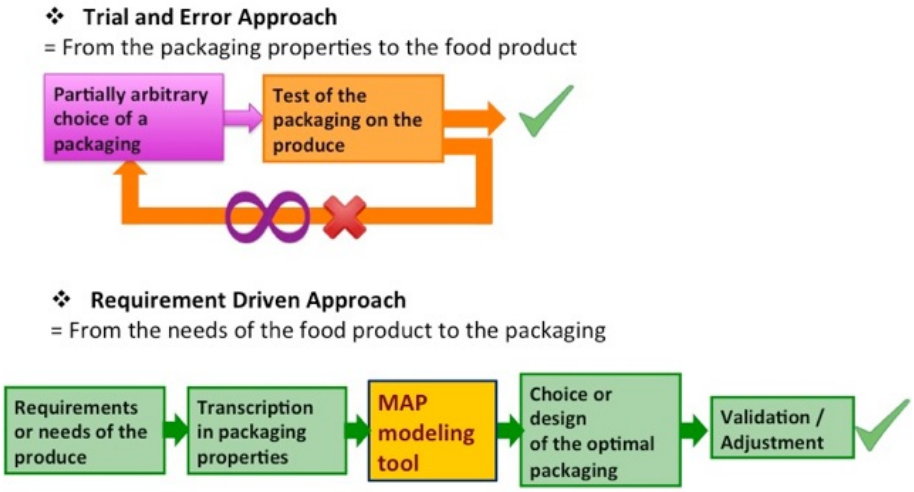

Figure 1: Trial and error approach versus requirement driven approach: interest of using mathematical modelling tools for a better dimensioning of food packaging material.

2. MODIFIED ATMOSPHERE PACKAGING OF FRESH FRUITS AND VEGETABLE

In the specific case of MAP of fresh fruits and vegetable, gas concentration in the headspace changes according to an interplay between gas permeation through the packaging material (oxygen getting in and carbon dioxide getting out), gas consumption (oxygen) and gas production (carbon dioxide) by the fresh food that respires, and until an equilibrium is reached (Fig. 2). In order to extend the shelf life, this atmosphere must be as closed as possible to the optimal and recommended one. Gas transfers in the food/packaging system can be modelled by combining mathematical equations representing on one hand gas permeation trough the material, such as Fick's law, and on the other hand gas consumption and production during respiration, such as Mickaelis-Menten based equations (GUILLAUME *et al.*, 2010). Mathematical models combining permeation and respiration can be used to predict internal gas evolution, i.e. partial pressures of O_2 and CO_2, within the food packaging. They could be also used to determine optimal O_2 and CO_2 permeabilities of the material knowing all the other parameters and considering optimal recommended O_2 and CO_2 concentration as target. The knowledge of the window of optimal gas permeabilities for a given product permits to select the right packaging material by querying dedicated database (GUILLARD *et al.*, 2015).

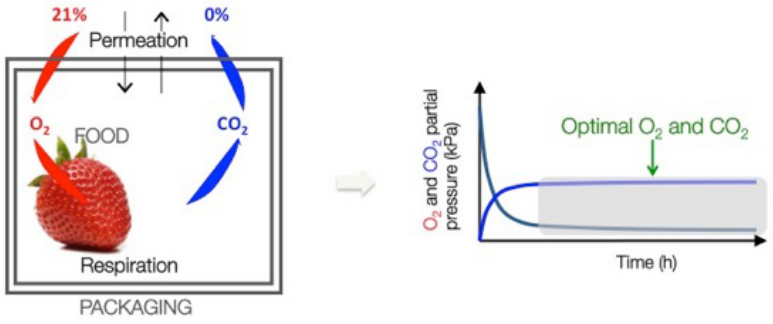

Figure 2: MAP of fresh fruits and vegetable: description of dynamic of gases exchange in the food/packaging system.

3. PREDICTING MICROBIAL FOOD SAFETY

In the case of microbial safety prediction in MAP of non-respiring food products, microorganism growth is strongly impacted by the gas composition in O_2 and CO_2 in headspace and its dynamic. Indeed, initial mixtures of gas added in the pack could vary a lot due to permeation and dissolution into the food (CHAIX *et al.*, 2015). The mechanistic model we have developed coupled for the first time mass transfer equations and predictive microbiology models and permit to identify in advance the gas permeabilities suitable to maintain microbial growth under a certain limit for a targeted application (Fig. 3). By knowing these targeted permeabilities (Fig. 4), we can query our packaging database (more than 350 permeability values currently capitalised) using a dedicated web application @Web (BUCHE *et al.*, 2011) to find the ranking of the most suitable packagings.

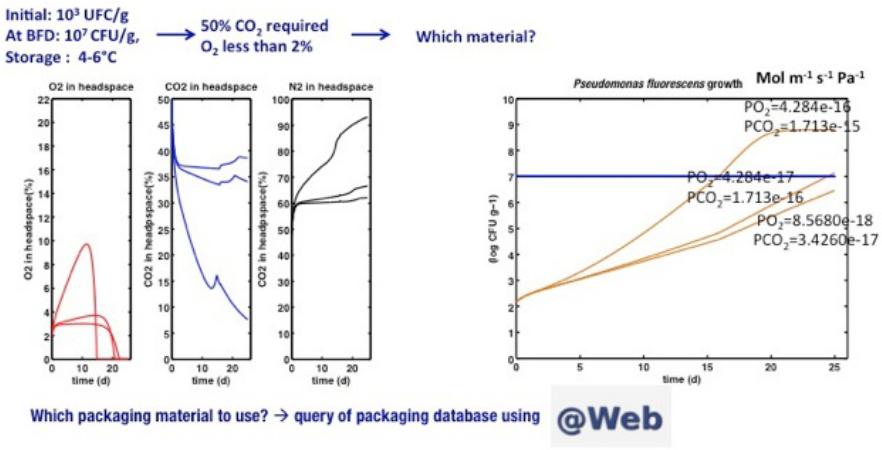

Figure 3: Identification of the couple of suitable O_2 and CO_2 permeabilities for the storage of poultry in order to obtain with an initial atmosphere of 50% CO_2 and 2% O_2 a quantity of *Pseudomonas* at the end of use-by-date lower than 10^7 CFU / g. initial load 10^5 CFU / g (*hypothetical data*).

In the present case study (Fig 4), the query made on O_2 permeability value has 2 materials in answer, both being ranked in first position. Last step is to test this material for a final validation of the selection made. This requirement driven approach is very efficient for helping food manufacturers in identifying rapidly materials for their MAP application, saving cost and time.

Figure 4: Query using @Web software of the packaging database in order to identify the best material corresponding to the criteria of O_2 permeability (case study shown in Fig. 3).

ACKNOWLEDGEMENTS

The French Agence Nationale de la Recherche (ANR) is gratefully acknowledged for its financial support through the MAP'OPT project (contract ANR-10-ALIA-002) and European Commission for its financial support in the framework of the EcoBioCAP project, Seventh Framework for Research & Technological Development (FP7/2011-2015) under the Grant Agreement FP7-265669.

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DEVELOPMENT OF MATHEMATICAL MODEL FOR TRANSPIRATION RATE OF FRESH-CUT LETTUCE

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ABSTRACT

During postharvest handling and storage, fresh fruits and vegetables continue to lose water through the process of transpiration which is a physiological process of water loss from fresh products that causes change in texture and a favourable environment for microbial growth. Iceberg lettuce is a highly perishable product due to the influence of minimal processing unit operations, such as cutting and shredding that causes disruptions of cells, which induces an increase in respiration rate, transpiration and enzymatic activities after harvest. The aim of this work was to quantify the transpiration rate (TR) of fresh cut lettuce under different storage condition (2, 6 and 10°C and 76, 86, 96 and 100% RH during 7 days). The results showed that both temperature and relative humidity affected TR which ranged from 0.04 to 2.36 (g $kg^{-1}h^{-1}$).A mathematical model was developed to predict TR as a function of temperature and humidity.

Keywords: iceberg lettuce, mathematical model, relative humidity, transpiration rate

1. INTRODUCTION

Iceberg lettuce (*Lactuca sativa* L.) is a highly perishable product with a short shelf life of 7 days at 7°C as reported on a commercial package due to the influence of minimal processing unit operations, such as cutting and shredding that causes disruptions of cells and consequently an increase in respiration, transpiration rate and enzymatic activities after harvest (LAREO *et al*., 2009). Fresh produce continue to lose water during postharvest handling and storage, through the process of transpiration which is the amount of water loss from the plant tissue per unit time and it is generally measured by conditioning the sample at a given temperature and relative humidity and weighing it at regular intervals. A weight loss of 5% or 10% of fresh weight is not acceptable because the product rots rapidly and it creates favourable environment for microbial growth; this implies that the weight loss should be as low as possible (MAHAJAN *et al*., 2008). Temperature and relative humidity (RH) of the atmospheric ambient have a significant effect on the rate of moisture loss during storage. As general trend, TR increases with the increase of temperature but decreases with increasing RH (LAREO *et al*., 2009; MAHAJAN *et al*., 2008; SOUSA-GALLAGHER *et al*., 2013; AINDONGO *et al*., 2014).
MAHAJAN *et al*. (2008) developed a simple mathematical model for transpiration rate to understand the evolution of mushrooms weight loss as a function of temperature and RH. This model was further modified and used for other products (CALEB *et al*., 2013; SOUSA-GALLAGHER *et al*., 2013).
Iceberg lettuce is packaged in polypropylene and stored under refrigeration temperature; due to the low water vapour permeability of these materials the water is accumulated into the package. The excess of condensed water is still an unresolved problem, mainly due to no available information on transpiration rate of lettuce. The aim of this work was to measure the transpiration rate of iceberg lettuce at different temperature and relative humidity and develop a mathematical model for predicting transpiration rate.

2. MATERIALS AND METHODS

2.1. Materials

Iceberg lettuce heads (*Lactuca sativa*) were obtained from Frucht Express GmbH, Groß Kreutz, Germany and transported using temperature controlled boxes in 30 minutes to the laboratory and equilibrated at the test temperature. Before the experiment started, the external leaves and the central stern were removed; then the lettuce heads were cut in pieces of 4 x 3 cm wide using a sharp knife, washed in cold tap water for 5 min and centrifuged for 2 minutes to remove the excess of water.

2.2. Transpiration rate measurements

The experiment was carried out in three containers, located in walk-in cooling room. RH was controlled by using saturate salt solutions of sodium chloride, potassium chloride, potassium nitrate and water, giving 76, 86, 96 and 100% RH, respectively. Salt solutions were placed on the bottom of the container and covered with a perforated aluminum support on which a plate containing 10 grams of cut iceberg lettuce was placed; the test was performed at 2, 6 and 10°C. Temperature and relative humidity were continuously monitored using a air humidity sensor FHA 646R (Ahlborn, Holzkirchen, Germany).
Transpiration rate was calculated as reported by SOUSA-GALLAGHER *et al*. (2013).

3. RESULTS AND DISCUSSIONS

Transpiration rate for fresh-cut iceberg lettuce ranged from 0.04 (g $kg^{-1}h^{-1}$) to 2.36 (g $kg^{-1}h^{-1}$) over all the combination of temperature and RH tested. As shown in Fig. 1 the values reported that TR was affected both by temperature and relative humidity: by increasing temperature from 2 to 10°C, TR increased and by increasing RH from 76 to 100% TR decreased.

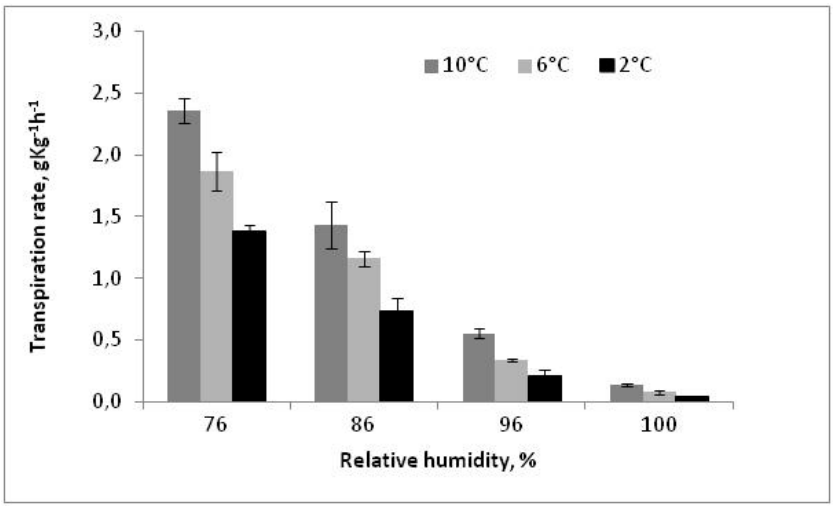

Figure 1: Effect of storage temperature and relative humidity on transpiration rate of fresh-cut iceberg lettuce.

Similar results were reported by LAREO *et al*. (2009) for butterhead lettuce leaves. The effect of temperature and humidity on transpiration rate of iceberg lettuce was quantified with a Pareto analysis at 95% significant level (Fig. 2); this analysis showed that, in the range of studied conditions, both temperature and relative humidity affected significantly the transpiration rate but the effect of humidity was more pronounced than that of temperature; moreover the interactive effects of temperature and humidity were significant. These results are in agreement with those obtained by MAHAJAN (2008) on mushrooms and Sousa-Gallagher (2013) on strawberry even if in this study the interactive effects between temperature and RH were not significant. To describe the effect of RH and temperature on TR of iceberg lettuce, the transpiration rate model developed for mushrooms has been used (MAHAJAN *et al*., 2008):

$$TR = Ki \times (a_{wi} - a_w)(1 - e^{-aT}) \quad (2)$$

Where a_w is the water activity of the container (RH/100), a_{wi} is the initial water activity of iceberg lettuce that assume a constant value of 1, T is the temperature (°C) and a is an empiric parameter to explain the effect of temperature.
To describe the TR at 100% of RH, the following equation has been used:

$$TR = b \times e^{cT} \quad (3)$$

Where b and c are the constant estimated from the experimental data and equal to 0.031 and 0.151, respectively.

Eq. (2) was combined with Eq. (3) yielding to a global model that explain the effect of RH in the range 0-100%:

$$TR = (b \times e^{cT}) + Ki \times (a_{wi} - a_w)(1 - e^{-aT}) \quad (4)$$

The mass transfer coefficient Ki and a were estimated by fitting Eq. (4) to the experimental data by non linear regression analysis. Results are reported in Table 1.

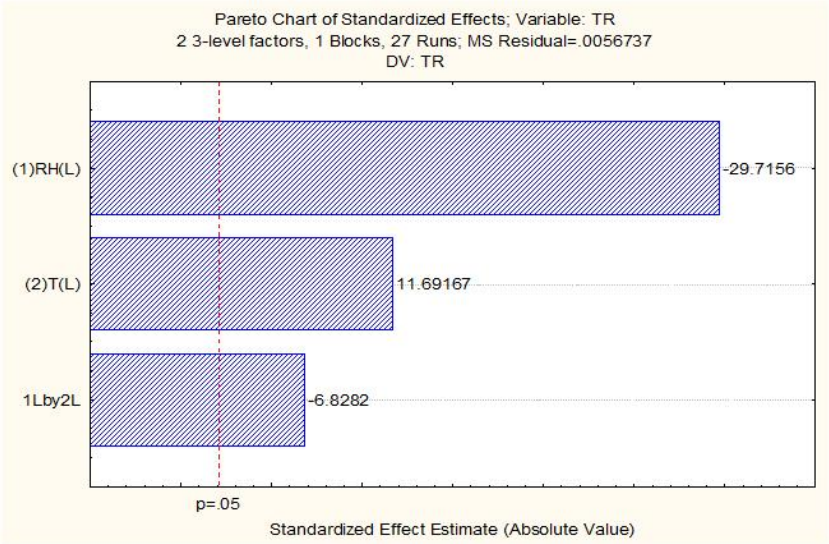

Figure 2: Pareto chart showing the effect of temperature and relative humidity on transpiration rate on iceberg lettuce at 95% significance level (indicated as a vertical dashed line).

Table 1: Parameters of the mathematical model (Eq. 4).

Ki (Kg h)	a ($°C^{-1}$)	b	c	R^2 (%)
8,92	0,42	0,031	0,151	96,6

Fig. 3 shows the relationship between experimental and predicted values of weight loss of iceberg lettuce; the values predicted by Eq. (4) were in agreement with those obtained experimentally (R>0.97).

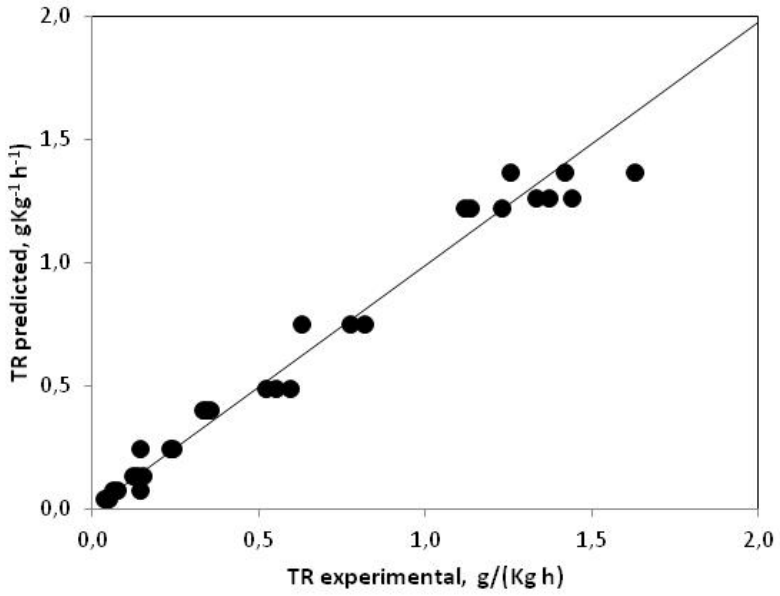

Figure 3: Relationship between experimental and predicted values of TR of fresh cut lettuce.

4. CONCLUSIONS

Transpiration rate of fresh-cut iceberg lettuce was found to be in the range of 0.04 - 2.36 (g Kg^{-1} h^{-1}) and both temperature and RH had a significant effect on transmission rate. The influence of RH in the range 0-100% and temperature has been well explained by mathematical model. This model would be useful to determining the amount of water loss and to design a proper package.

ACKNOWLEDGEMENTS

This research was performed within the project PRIN2012 "Long life, High Sustainability" supported by MIUR. Thanks to the doctoral program for the scholarship awarded to Stefania Volpe.

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RETAILERS TOWARDS ZERO-WASTE: A WALKTHROUGH SURVEY IN ITALY

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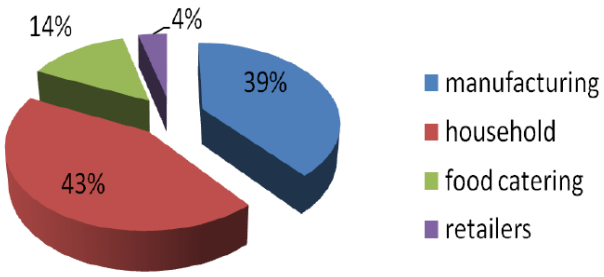
ABSTRACT

Approximately 1/3 of the food produced for consumption is lost/wasted globally being discarded by retail shops and households. Reducing food losses can thus make a significant contribution to ensuring global food supply. The purpose of this paper is to analyze how the Italy's top ten food retailers try to promote sustainable consumptions within stores. Some simple "walk through/visual observation and information collection" surveys conducted within the largest store operated by each of the best food retailers within a province in South Italy provided the empirical materials. Then, conclusions suggest how paths direct to sustainable consumptions should be inserted into the large food retailers' business models. Defining a bio-business model can move peoples towards zero-waste.

Keywords: food waste, sustainable consumption, visual observation

1. INTRODUCTION

Bio-Based Economy (BBE) could be explored such as a path toward a zero-waste humanity (ZWIER *et al.*, 2015; ADDY, 2013; SULLIVAN, 2011) addressing people's concerns about their livelihoods, safety and environment. So policies and researches trace several steps: waste prevention (WRAP, 2009); recycling/re-use of municipal waste (STEFAN *et al.*, 2012); increasing packaging waste recycling/re-using (SILVENIUS *et al.*, 2014); reducing food waste generation (FIORE *et al.*, 2015; YOUNG *et al.*, 2010). Currently, food waste has generated an immense amount across the food life cycle, determining serious environmental, social and economic issues. The development of the bioeconomy can be crucial for emphasizing the management of individual life, including the regulation and control of food consumption (ALEXANDER, 2012). In this context, a crucial role in promoting sustainable consumption beyond the throwaway society can be assigned to food retailing (FIORE *et al.*, 2015; LEBERSORGER and SCHNEIDER, 2014) providing customers with some core information (STEFAN *et al.*, 2012). But, generally retailers believe that a majority of purchases are unplanned, so they spend heavily on in-store marketing to stimulate them (BELL *et al.*, 2011). Among several initiatives to prevent food waste, Last Minute Market (LMM) is a project where retailers, shops and producers who have unsold food, which would otherwise be discarded are connected with people and charities who need food. Graphic 1 shows the percentage of food waste; household and retailers have the major percentage (respectively 43% and 39%).



Graphic 1: Percentage of food waste in manufacturing, household, food catering, retailers (Source: http://www.lastminutemarket.it).

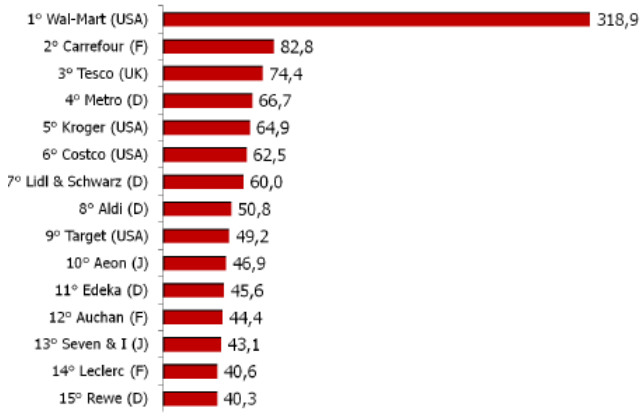
If LMM were adopted from all retailers in Italy, it is expected a huge quantities of recuperated food per year (Fig. 1). In the last decades, the supermarket industry has so become increasingly competitive: some authors show proliferation of a variety of pricing formats affect consumers' store choice behavior (BELL *et al.*, 2011); generally the philosophy of the retailer is "grabbing consumers" at the point of purchase (CHANDON *et al.*, 2009). On the other hand, other authors (JONES *et al.*, 2011) highlight the vital role of retailers in stopping 'unfair' purchases; in 2009, for example, the European Commission and a number of the UK's leading retailers along with several of their European counterparts launched a 'Retail Forum' as part of a drive to support more sustainable consumption (CHANDON *et al.*, 2009). Finally, the large food retailers are broadly recognized as having a significant impact on the environment, economy and society (JONES *et al.*, 2011) and bioeconomy can address the control of food consumption in a sustainable way (ALEXANDER, 2012). There is not a plethora of work on the retail role in promoting zero-waste model so this work try to fill this lack.

Stores' Typology	Expected quantities of recuperated food per year
Cash and Carry shops	4.850 tonnes
Superstores	52..920 tonnes
Supermarkets	136.611 tonnes
Small shops	73.518 tonnes
TOTAL	267.899 tonnes
VALUE OF RECUPERATED FOOD	1.536.396.624 euro = 636.593.035 meals in a year
CO2 emissions	319.603 tonnes of CO2 are produced because the food end up in landfills

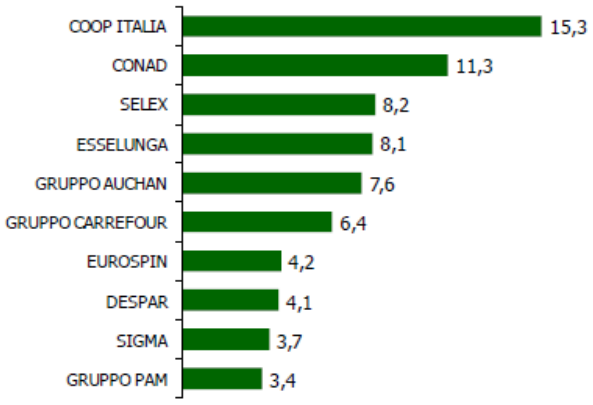
Figure 1: Typology of stores and expected quantities of recuperated food per year (Source: http://www.lastminutemarket.it).

2. MATERIALS AND METHODS

Starting from a work by JONES *et al*. (2011), the present paper carried out and collected the data by means of some simple "walk through/visual observation and information collection" surveys conducted within the largest store operated by each of the best food retailers within the province of Foggia, in South Italy; these surveys provided the empirical material for the case-study. We undertook a basic observational survey in Apulia Region, specifically in the area of Foggia and Bari. The aim of the work is to gain some insight into how the Italian top ten food retailers are currently trying to engage customers in sustainable consumption in their stores. At international level, the most important retailers (for sales) are shown in Graphic 2, while Graphic 3 highlights top ten food retailers in Italy (% on the total sales).



Graphic 2: International food retails (Source: AC Nielsen, 2013 available at: http://www.nielsen.com/us/en.html).



Graphic 3: Groups of food retail in Italy (% on the total sales) (Source: AC Nielsen, 2013 available at: http://www.nielsen.com/us/en.html).

The survey was undertaken in the store operated by each of the Italy's top ten food retailers (excluding Esselunga and PAM Group, because they are not located in the study area) namely Coop Italia, Conad, Auchan, Selex, Despar, Eurospin, Sigma, Carrefour Group: the time of collection was spread on two periods, the third week of March and the last week of April 2015. Visited stores size varies considerably from Ipermarket to superette, offering a wide range of food and non-food goods. We choose the "walk through/visual observation and recording" survey methodology (JONES *et al*., 2011) that allows us to understand the extent to which sustainable consumption messages where being used in marketing communications on banners, posters and television screens, on the shelves and shelf edges, on the products theme selves and on information leaflets and promotional leaflets and flyers. Some authors demonstrated the number of facings has a strong impact on evaluation that is wholly mediated by its effect on visual attention and works particularly well for frequent users of the brand, for low-market-share brands, and for young and highly educated consumers who are willing to trade off brand and price (CHANDON *et al*., 2009). In order to support our survey methodology, we create an accurate check list/guidelines (Table 1), in order to capture the ways in which messages about sustainable consumption were, or were not, being presented to customers within store. The guideline highlights several items: in addition to the general info (name of the group, retail and positioning), we structured our guideline analyzing setting and exhibition, commercial offer and assortment, layout, communication tools used in the store, type of messages (text messages listed in the store and any photos), institutional communication towards sustainable consumptions (used instruments and content) with evaluation of the ease of finding information in the store (for these latter questions five Likert Scales items have been adopted). The aim of our collection and analysis of the survey data is to identify broad themes and to explore how retailers are currently trying to engage customers with sustainable consumption at the point of sales, and not to provide a detailed comparison of the retailers.

Table 1: Guidelines for observing Stores (Source: our processing).

Name of the Group
Retail
Positioning
Setting and Exhibition
Used Materials: wood – steel- plastic - glass
Dividers: shelves lanes islands fridge / frozen counters shelves fridge self stand
Technologies and devices used
Commercial Offer and Assortment
Layout
Category: *Price type product origin issue other promotions*
Communication tools used in the store
Signage
Totem
Staff dedicated to promotions
Speaker signs
Communication materials (leaflets, etc.)
Info point
High Tech instruments (QR Code, computer stations, electronic signs)
Other
Type of messages (text messages listed in the store and any photos)
Institutional communication (used instruments and content) towards sustainable consumptions
Evaluation of the ease of finding information

Completely Inadequate	Major Problems	Minor Problems	Adequate	Superior
☐	☐	☐	☐	☐

3. RESULTS AND CONLCUSIONS

From the survey emerged three general themes. Firstly, the dominant sets of messages were concerned with encouraging customers to consume rather than to restrain from consumption or to promote sustainable consumption. All of the 8 top ten food retailers advertised "discount", "special discount", "special offer", "offer", "daily saving", and under that a picture of the product with the indication of its special price. This kind of messages was conveyed on posters and banners all around the shop and also on shelf edges, near the products' price indication. Other messages, focused on "multiple purchases at reduced prices", offers such as "2x1"," Buy 3 pay 2". These promotional messages may be not all pervasive but they can be seen to be setting the tone for customer expectation (JONES *et al.*, 2011). These price reduction messages can be interpreted as stimulating consumption, and not only, as retailers affirm, as providing value for money for their customers. The emerged second general theme is "customer loyalty management" by two types of messages. One that tells to the customers that they will always find lower prices, and indirectly inviting them to come back and be sure that the retail group will always provide a range of product at fixed price (obviously, low price). "Low and fixed", "Reduced prices", "100 products at reduced price", "Accustomed to convenience and quality. Coop.", " Conad discounts what it counts" are the slogan used to

help to improve customer loyalty, showing retail group commitment to guarantee stability in convenience in addiction to promotional offers. The instruments used to conveying this messages are banners posters and television screens with pictures of the product or of group of products, the slogan and, in many cases, the picture of the fidelity card of the retailer's group. Another type of messages explains how the Group is working to assure quality and the best choice for its customers, in terms of choices of range products and quality and food safety. Example are slogan as " Our Stars is the Eurospin brand dedicated to Italian and international excellence. Discover the quality of PDO, IGT and STG. This is the reason why shopping here is a smart choice", "Territories chosen by COOP: products from Apulia and Basilicata", "There is Taste by choosing a typical product. Flavors and Surroundings CONAD". The core third issue revealed by the survey is that there is limited number of messages with sustainable consumption themes. The only sustainable consumption message is about the use of reusable carrier bags. Coop, Eurospin, Auchan, Selex provide a sign at all the checkouts to encourage customers to use reusable carrier bags. For example, Eurospin store reminds customers about "The smart bag. Reuse is an advantage for you and for the environment". Beyond the latter observation, generally we can state it is hard to find institutional communication and info moving towards sustainable consumptions (8 on 8 stores have an inadequate institutional communication). Our survey includes also observational data about layout choices of the different food retailers groups, but here we have not analyzed them owing to our detailed study on semiotic that need integrated approach. In addition, we analyzed the general web site of the large scale retail distribution in Italy (www.gdoweek.it); results are in line with other ones above mentioned. To sum up, we notice a lack of messages towards sustainable consumption; the major food retailers structure communication, which are rather driven by their own commercial goals than toward a zero-waste humanity and behavior (ZWIER *et al*., 2015; ADDY, 2013; SULLIVAN, 2011).

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POSSIBILITIES FOR REDUCTION OF FOOD LOSS AND WASTE: THE CASE STUDY OF LITHUANIA'S PRODUCER COOPERATIVES

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ABSTRACT

The aim of this study is to propose organisational system providing a possibility to various size sea buckthorn producer farms to work together by merging into cooperatives and producer groups in order to improve production and logistics management and reduce food loss and waste along the entire supply chain. A semi-structured interview method was applied with respect to Lithuania's cooperatives and producer groups from neighbouring countries. Inductive reasoning method has been applied for the data analysis. The Research revealed that establishment of producer groups is directly related to the development of cooperatives with a better production and logistics management with a consequently reduction of food loss and waste and an increase of farmer profitability. The scale measuring the dependent variable contained self-reported items and such self-reports may be biased estimates of true behaviour.

Keywords: cooperation and producer, food loss and waste reduction, inductive reasoning method

1. INTRODUCTION

The competitiveness of the agro-food products is based upon: use of qualification system; improvement of both production technology and quality; traceability issues; optimisation of product costs; and, finally, promotion of products worldwide and environmental sustainability (Contò et al., 2013). This may be achieved using the best tools that are able to give sustainable results such as reduction of toxic releases damaging global environment and human health, healthier air, surface and groundwater, energy saving, reduction of chemicals production, nutritional quality improvement. Although the demand for sea buckthorn has increased in the European market, small sea buckthorn farms still prevail in Lithuania. Usually the main initiators of Lithuania's producer groups in and cooperatives are the leaders, i.e. the initiators of changes. Farmers have difficulties in ensuring timely harvesting of the sea buckthorn, as well as its processing and preparation for the market. Besides, the sales of the sea buckthorn could be a profitable activity only in case of huge volumes of production, which is properly processed and introduced to the market. This is possible only if producers are able to establish cooperatives employing intensive/advanced sea buckthorn growing/storage/marketing technologies in organising the operations in national and international markets. 156 farmers grow sea buckthorns in Lithuania, the total covered area is 1900 hectares. The majority of sea buckthorn producers are small farmers. Size of the fields varies from 5 to 10 hectares. Only 30 of them is processing more than 30 hectares. Cooperation in berry sector of the Baltic states is analyzed in a fragmentary (GULBĖ, 2003; RAMANAUSKIENĖ, 2007; PAREIGIENĖ, 2008). Joining forces via farmers cooperatives or professional associations can greatly help reduce food losses. This is achieved by establishing quality control and logistics mechanism in order to obtain fewer product rejections by wholesalers (SOYSAL *et al*., 2015), increasing understanding of the market and enabling more efficient planning, lowering individual vulnerability, improving efficiency through economy of scale.
All of these problems forced sea buckthorn producers to seek for local cooperation models. The aim of this paper is to propose organisational system providing a possibility to various size sea buckthorn producer farms to work together by merging into cooperatives and producer groups (PG) in order to reach an environmental production improvement (in terms of food loss and waste reduction) and an increase of farmers profitability.

2. MATERIALS AND METHODS

A semi-structured interview method was applied with respect to Lithuania's cooperatives and producer groups from neighbouring countries (Latvia and Poland). 26 respondents were interviewed by structured questionnaire: 4 in Poland, 2 in Latvia and 20 in Lithuania. Medium sized (10-50 acres) of sea buckthorn plantation growers participated in the survey in Lithuania and Latvia. Successful fruit cooperatives operating in the market for at least two years were interviewed in Poland. With all of the respondents were interacting directly 27 semi-structured questions were given to the respondents. Questionnaire covered common issues associated with sea buckthorn growing areas, volumes, available equipment, the products sold on the market or remain untaken away in yields and unrealized output, logistics management, the amount of loss along suplly chain and the wishes of the farmers co-operate in the setting up producer organizations. Inductive reasoning method has been applied for the data analysis.

3. RESULTS AND CONCLUSIONS

The capacity of cooperative companies and their location have been estimated so as to minimise the construction costs of future facilities, equipment and production transportation costs. The price of product unit transportation from Supplier *i* to Recipient *j* is marked C_{ij}, whereas the amount of production delivered by *i* Supplier to *j* Recipient is marked X_{ij}. The costs related to the amount of transported consignment X_{ij} will be $C_{ij} \times X_{ij}$ and transportation price:

$$\sum_{j=1}^{m} \sum_{j=1}^{n} C_{ij} \cdot X_{ij} \rightarrow min \tag{1}$$

The equipment price of *i* cooperative is marked F_i the unknown $Y_i=1$ if company will be equipped, and $Y_i=0$, if company will not be equipped. If main suppliers are the cooperatives with equipped companies, it is assumed that the amount of the supplied production will not exceed company's capacity, i.e.

$$\sum_{j=1}^{n} X_{ij} \leq A_i \cdot Y_i \left(i = 1,2,.....,m\right) \tag{2}$$

Product demand is known, therefore the condition for meeting the demand is:

$$\sum_{i=1}^{m} X_{ij} = B_j \left(j = 1,2,.....,n\right) \tag{3}$$

Task solution condition:

$$\sum_{i=1}^{m} A_i \geq \sum_{j=1}^{n} B_j \tag{4}$$

General task model looks as follows:

$$F_x = \sum_{i=1}^{m} \sum_{j=1}^{n} C_{ij} \cdot X_{ij} + \sum_{i=1}^{m} F_i \cdot Y_i \rightarrow min \tag{5}$$

In case of restrictions in the supplied production amounts by product demand and under condition of undeniable variables:

$$X_{ij} \geq 0 \left(i = 1,2,.....,m\right),$$
$$\left(j = 1,2,.....,n\right).$$

When the unknown are: $Y_i = 0$ or $Y_i = 1 \left(i = 1,2,.....,m\right)$

This is a mixed task. X_{ij} is an equivalent variable, and Y_i – whole variable. Objective function is expressed in two linear functions, where one function shows transportation and the second the equipment costs (the sum) of new companies. The application of the proposed methodology gives the following advantages for the developers of cooperative activity (producer organisations):

- Reduced distance for transportation of raw materials helps to speed-up realization of sea buckthorns. It also helps to ensure better quality of berries and reduce transportation costs/one kilogram of berries;
- There is a possibility to demonstrate advantages of a cooperative company, encourage more farmers to join sea buckthorn growing activity in smaller areas which are not suitable for growing other plants;
- Development of infrastructure facilitating the establishment of new jobs in rural areas and ensuring additional income generation in rural territories.

In conclusion the success and development of sea buckthorn growers' business is related to the ability to buy expensive harvesting and berry freezing equipment. Each farm has a limited financial capacity to purchase the necessary equipment, so the most rational way to have the necessary fruit processing capacity is to organize equipment cooperatives. Establishment of producer organisations is hindered by the size of cooperatives which cannot guarantee a required extent of trade turnover motivating to seek the support for market development allocated to producer organisations. Larger farmers merge into cooperatives and producer groups more successfully, whereas small farmers who could survive only in cooperation with others have more doubts and difficulties in merging with other companies. An integral hierarchical system for merging various size sea buckthorn producer farms into cooperatives helps to ensure a well-structures process for the establishment of local, territorial and national cooperatives and producer groups, and clearly specify the content of the activity of different level cooperatives. While optimising the activity of producer organisations (cooperatives) uniting sea buckthorn producers, a special attention should be given to justification of location of logistics objects of these organisations in the territory of the country. This is a very important factor allowing to reduce production costs, production transportation losses, and ensuring quality parameters of the berry yield. Spatial pattern analysis of Lithuanian buckthorn growers suggested to form five territorial cooperative capacity layout areas.

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CAN SHELF LIFE BE CONSIDERED AS A MEAN TO PROMOTE VEGETABLE PRODUCTS OF SUSTAINABLE LOCAL VARIETIES?

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ABSTRACT

In connection with the increasing interest for the consumption of food products of the local tradition, an investigation on the behavior of the consumers were conduct out for agricultural products also used as first subject for the preparation of ready dishes. Lately, also for the prepared alimentary products, processes of substitution it is assisted with products of the local tradition. The consumer, in fact, also for the ready foods it declares priority for the products food of the native saperis. In this context, particular the role of the grain legumes. The analysis proposed in this paper purely concerns the propensity to the consumption of legumes typical of an agricultural territory of Sicily.

Keywords: consumer behavior, local varieties, shelf life, food security, food safety

1. INTRODUCTION

The interest of the market to improve the shelf life (ALLEGRA, 2012) of the foods is strongly in increase, above all, to the agricultural products used in many food preparations of the popular tradition. In this context, the dried grain legumes have always been widely used for the consumption of prepared foods. However, constitutes a constraint, the period of retention, if the legumes are not industrially processed. In relation to the new market opportunities, interested parties are busy in judgments on whether to make use of technological tools that enhance the preservation of local tradition legumes. An important support to the evaluations can result from the knowledge of the demand motivation. Identify consumer trends means having information about buying behavior, about tastes and habits depending to age and lifestyle, about times and conditions of use of products. The survey on consumer motivations then gives concrete indications to the production and at the same time the right suggestions for the commercialization of the product concerned. In this article, we present the results of a survey on a sample of consumers of legumes of local biodiversity particularly, of a zone of Sicily with agricultural aptitude. Legumes are considered a vital source of food of the local population, but have not expressed a structural change. In particular, the investigation concerns the motivation at the origin of consumer behavior for dried grain legumes packed. Specifically, the analysis relates to the comparison between faba bean mayor, black lentil and chickpeas of the territory of Leonforte (Enna) and legumes with minimal characterization in terms of specificity. The packaging has been realized by the farmers for the variety of local biodiversity; instead, through wholesalers manipulators for legumes not characterized. In order to delineate the differences in behavior in the decision-making process of the consumer between legumes traditional and legumes of the retail chains. The results also provide interesting insights for evaluating the function of production of local varieties of legumes in relation to the promotion of environmental sustainability and income of stakeholders.

2. MATERIALS AND METHODS

The direct survey conducted on local varieties of legumes inland hills of Sicily is based on data collected at events aggregative public (festivals, fairs), through a questionnaire prepared ad hoc (ALLEGRA, 2014). This administered to 225 individuals of all ages, different sex, social class varies, of which solo 197 fit for the survey; the inquiry it was conduct in the period 2013-2015. The interviews were conducted both in the area of origin of the cultivation of legumes and other places of consumption of Sicily. As for the processing of collected, were consider only the age of the individuals, because other social personal characters do not affect the attitude of consumer the food. Each respondent, taken individually, has performed an assessment of the following attributes of legumes under investigation: perceived quality, typicality, ease of use, availability and convenience. This in order to able to identify the best alternative that maximizes the total utility in accordance with these attributes. The data collected were process in accordance with a methodology called “formal strategies of integration” which consists of the additive or linear rule “according to which the individual expresses a weight and an evaluation for each outstanding attribute of each alternative.” The value assigned to each attribute was multiply by the corresponding relative weight; results were sum to obtain an overall assessment of the alternative. Scores made to allow an alternative to implement the choice; that is, the highest value obtained.

For this purpose, we develop the additive rule formula indicated below:

$$VA = \sum_{i=l}^{n} WiV(X_{Ai})$$

In which: - *W* is the weight of the i-th in terms of the importance for the decision maker; - *V (X)* the evaluation of the attribute i-th and *n* are the attributes considered (Dalli, 2000). It should be observe that, the weight (W) is distinctive for each attribute, which does not depend on the alternative considered, however the assessment (V [x]) is attribute to the opinion expressed by the attribute of a specific alternative. Regarding the quantification, all "importance" has been assigned value between 0 and 1, the evaluation of the legume from 1 to 5. The data were process by classes of the age of the respondents, identifying a population with age: below 25 years old, between 25 and 45 years old, and finally over 45 years old.

3. RESULTS AND DISCUSSIONS

Demand is very active for grain legumes of consumers on any age group, with the expression of interest for all the concerned species. With reference to the attributes (Table 1), there is a certain uniformity as regards perceived quality, typicality and value for money, while recording differences for ease of use and availability. For these attributes, the ratings decrease going from younger adults because of poor knowledge of the peculiar characteristics of the legumes in question and in the kitchen preparation.

Table 1 - Grain legumes of the loal tradition and of the not specific origin, distribution to attribute type and age of consumers (*)

Attributes	Importange	Faba bean mayor			Black Lentil			Chickpeas			Legumes does not specified		
		Age of consumers			Age of consumers			Age of consumers			Age of consumers		
		<25	25-45	>45	<25	25-45	>45	<25	25-45	>45	<25	25-45	>45
Perceived quality	0,3	3	3	3	4	4	4	2	2	2	3	3	3
Typicality	0,3	4	4	4	4	4	4	1	1	1	3	3	3
Ease of Use	0,2	1	2	2	4	5	5	3	4	4	2	3	3
Availability	0,1	2	3	3	3	3	3	5	5	5	2	2	2
Value for Money	0,1	3	3	3	2	2	2	5	5	5	3	3	3

(*) Directly acquired data

Table 2 - Overall Assessment of the grain legumes by age consumers (*)

Attributes	Faba bean mayor			Black Lentil			Chickpeas			Legumes does not specified		
	Age of consumers			Age of consumers			Age of consumers			Age of consumers		
	<25	25-45	>45	<25	25-45	>45	<25	25-45	>45	<25	25-45	>45
Perceived quality	0,9	1,2	1,5	1,2	1,2	1,5	0,9	0,9	0,9	0,6	0,6	0,9
Typicality	1,2	1,2	1,5	1,2	1,2	1,5	0,9	0,9	0,9	0,3	0,3	0,3
Ease of Use	0,2	0,4	0,8	0,8	1,0	0,8	0,4	0,4	0,6	0,6	0,8	0,8
Availability	0,2	0,2	0,3	0,3	0,3	0,3	0,2	0,1	0,2	0,5	0,4	0,5
Value for Money	0,2	0,3	0,3	0,1	0,2	0,6	0,3	0,2	0,3	0,5	0,5	0,5
Votes obtained	***2,7***	***3,3***	***4,4***	***3,6***	***3,9***	***4,7***	***2,7***	***2,5***	***2,9***	***2,5***	***2,6***	***3,0***

(*) Our elaborations on directly acquired data

Table 2 on the overall evaluation by age, shows that legumes local tradition have higher values than to grain legumes generic. Of course, the reasons for each consumer behavior may be very different, especially in relation to the specific objective that every consumer wants to achieve as a satisfaction of a need of a food particular. From the interviews, it became clear that the decision-making process be the result of direct experience,

observation of the moment, or word of mouth. Specifically, the interviews showed that the motivation at the origin of consumer behavior varies by social group examined. In fact, in the age group over 45 years, consumer behavior is due to the memory that is the main element that influences the decision, so the choice. As for the younger age groups, the impulse is the most important factor for the choice of legumes, as the decision to purchase is determine by social and environmental conditions and the mood of the moment. In all cases, whatever the social group by age considered or the decision established, the choice of the consumer is linked to the traditional legumes to which they are increasingly recognizes the organoleptic and nutritional properties, as well as the guarantee of the maintenance of food safety standards.
Table 3 shows the results obtained from interviews with consumers be they traditional and modernists, about the reasons for purchase and the reasons for non-purchase of legumes if available at major retail outlets.

Table 3 - Orientation buying packaged legumes in grocery store (*)

Main places of food retail	Consumers of grain legumes of the local traditional							
	Traditionalists			Modernists			Respondents in total	
	<25 old	25-45 old	>45 old	<25 old	25-45 old	>45 old	N.	%
Discount		4	6		3		13	*6,6*
Supermarkets	6	5	9	2	14	2	38	*19,3*
Groceries								
- *specialist*	4	10	16	6	11	3	50	*25,4*
- *traditional*	5	18	22	2	8	5	60	*30,5*
Farmer's market		2	18	1	12	3	36	*18,2*
Total	15	39	71	11	48	13	197	*100,0*
	7,6	*19,8*	*36,0*	*5,6*	*24,4*	*6,6*	*100,0*	

(*) Directly acquired data

In this way, they highlight market segments with spaces to be covered. The survey also made it possible to achieve interesting results about the possibility of development of production of traditional legumes in the ecosystem of origin (food security).

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ACTIVE PACKAGING IN MASTER BAG SOLUTIONS AND SHELF LIFE EXTENSION OF RED RASPBERRIES (*RUBUS IDAEUS* L.): A RELIABLE STRATEGY TO REDUCE FOOD LOSS

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ABSTRACT

Small red fruits are very appreciated by consumers for their high sensory quality and their nutritional value. Furthermore, raspberries are very perishable produce, having high respiration rates and a fragile structure. Water loss, mould growth and off-flavour production during storage limit the shelf life of these fruits to few days. Currently, the high perishable characteristics of these fruits contribute to Food Loss along the supply chain (up to 75% until retailer). In this work, the possibility of extending the shelf life of red raspberries (*Rubus idaeus L.*) using active packaging solutions has been investigated, after the definition of critical indicators and cut-off criteria useful in pointing the time at which the lifetime ends. The final aim was to estimate the role of a new packaging technology in reducing Environmental Impact along the supply chain but taking into account the benefits of the Food Loss reduction derived from the actual Shelf Life Extension. The environmental impact of each packaging solution was estimated using the LCA methodology. Data were scaled with a factor that takes into account the contribute of packaging in the shelf life extension of raspberries, using lifetime data obtained in this experimental work. Results highlighted that the Shelf Life Extenstion of raspberries through an active packaging solution reduces the environmental impacts and the food losses.

Keywords: Shelf Life Extension (SLE), Food Loss, LCA

1. INTRODUCTION

Small red fruits and in particular raspberries have very short storage life due to physiological aspects. The production of raspberries in Italy consists in 1500 tons per year and ranks 16th in Europe, after the main East European states (Russian Federation, Poland, Serbia). The market, in this sector, has been increasing due to the production differentiation, profitability and sustainability of cultivation in terms of economy and environment preservation. The high perishable characteristics such as high respiration rate, loss of firmness, mould susceptibility, and breaking down tissues define the most common reasons of customers' complaints. These characteristics can contribute to important food loss and waste along the supply chain up to 75% until its arrival at the retailer and in particular WRAP study estimated the loss and waste in different steps of the supply chain in the UK. The aim of this study was to estimate the role of a new packaging technology in reducing Environmental Impact along the supply chain but taking into account the benefits of the Food Loss reduction derived from the actual Shelf Life Extension.

2. MATERIALS AND METHODS

Red Raspberries (*Rubus ideaus L.*) cv. Erika were used. Fruits were hand-harvested at commercial ripening stage and were packaged within 24h; only berries with comparable colour and absence of defects and mould were selected. The packaging solutions were: A) Lidded macro-perforated PET trays containing 125g of berries, stored in air and considered as "traditional" packaging; B) Two lidded macro-perforated PET trays containing 125g of berries inserted into master bags made of plastic materials with different permeabilities to gas and water vapor (passive modified packaging solution); C) Two macro-perforated PET trays (each containing 125g of berries) inserted into a master bag unit made of LDPE with one oxygen scavenger, a different number of pre-activated carbon dioxide emitters (500 cm^3 of capacity) and different values of film surface/unfilled volume ratio inside masterbag (active modified packaging solution), as described in Table 1. Respiration Rate (RRO_2 and $RRCO_2$); Color (Imagine analysis – clustering by k-means methods); Firmness (N*mm); Weight loss (%); Visual Rejected Berries (Molded and Damaged Berries); Headspace composition (% O_2 and % CO_2); Soluble Solid (g/100g); Volatile compounds by GC-MS (Ethanol, and Ethyl acetate), Titrable acidity (g citric acid/100g).; Dry matter (g/100g). Principal component analysis was carried out by using Unscrambler (v. 9.7) software package. The Impact Assessment was evaluated by means of the Software SimaPro v. 8.0.1 (PRé Consultants, The Netherlands), ReCiPe Midpoint (H) v. 1.08, Europe Recipe (H).

Table 1: Definition of physical and flavor descriptors used in the quantitative descriptive analysis.

Code	S/UFV* (cm^2*cm^{-3})	N° of O_2 scavengers	N° of CO_2 emitters
A	1.077	1	2
B	1.077	1	3
C	0.506	1	3
D	0.506	1	4
E	0.506	1	5

3. RESULTS AND CONCLUSIONS

In order to assess the shelf life value - in terms of a finite length of time after production and packaging, during which the food product retains a required level of quality - a multivariate approach based on PCA was used. PC1 was time related (data not shown). The slowest rate constant was set for fruits stored in solution C (1 scavenger, 3 emitters with the lowest S/V ratio). A critical PC1 score was set using the multivariate procedure proposed by Pedro and Ferreira (2006) that led to an estimation of shelf life equal to 10±0.7 days for solution C (Fig. 1). The used equation (Equation 1) was:

$$\text{Critical PC} = Xa * L \quad \text{(Equation 1)}$$

where:
Xa is the row vector of reference values for each quality index (data not shown) and L is the NXa loadings matrix of the time-related PC.

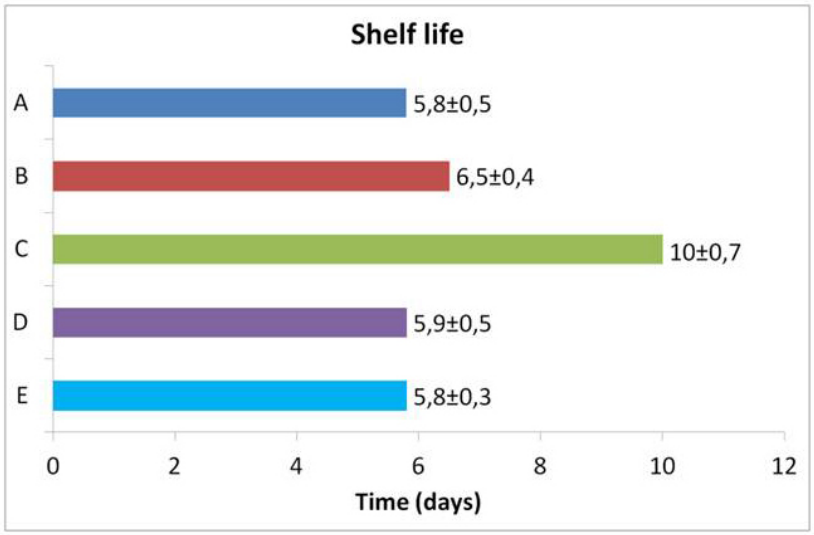

Figure 1: Shelf life value for each packaging solution estimated from the PC analysis.

A model (Equation 2) that gives the balance between the environmental impact of packaging and the environmental impact of the food losses due to changes in packaging was used (WILLIAMS *et al.*, 2011).

$$\frac{T2}{T1} < \frac{1-L2}{1-L1} + \frac{L1-L2}{1-L1} * \frac{F}{T1} \quad \text{(Equation 2)}$$

Where:
T1 is the Environmental Impact of "traditional" packaging (PET tray)
T2 is the Environmental Impact of "new" packaging (active atmosphere solution)
L1 is the Fraction of Food Loss ("traditional" packaging)
L2 is the Fraction of Food Loss (active packaging solution in master bag)
F is the Environmental Impact of food without packaging

The values used in Equation 2 and shown in Table 2 derived from the shelf life study and the calculation of the environmental load by meas the LCA approach.

Table 2: Values used in Equation 2.

T1	0.22 kg CO_2-equiv	LCA results
T2	0.25 kg CO_2-equiv	LCA results
L1	0.20	WRAP (2008)
L2	0.10	Preliminary results
F	0.12 kg CO_2-equiv	LCA results

In our solution C (active solution) the increase in environmental impact from packaging T2/T1 was below (1.15) the environmental impact generated by the food loss reduction (1.19), thus the contribution of the food "saving" was higher than the larger environmental load of the new packaging solution. Therefore, the use of active packaging solutions for raspberry fruits storage improves their shelf Life. Consequently, this longer shelf life can really contribute to reduce the food loss and the environmental burden.

ACKNOWLEDGEMENTS

This research was supported by Ministero dell'Istruzione, dell'Università e della Ricerca (Prot. 957/ric, 28/12/2012), through the Project 2012ZN3KJL "Long Life, High Sustainability").

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A RISK ASSESSMENT APPROACH ON THE UNINTENTIONAL TRANSFER OF INK COMPONENTS FROM PRINTED TO FOOD CONTACT LAYER OF FLEXIBLE FILMS

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ABSTRACT

The first step of the experiment requested a screening analysis of the structures to detect and quantify some chemical markers coming from the printed surface. The volatiles and semi-volatiles in the films have been analyzed by SPME-GC-MS, while semi-volatiles and non-volatiles substances after solvent extraction and GC-MS. A central composite cube-star design has been applied considering three experimental factors (time, temperature and reel contact pressure) at different levels. For each run, the previous selected markers have been quantified or semi-quantified on the food contact layer, assessing the statistically significant factors and interactions. The results highlighted the importance of temperature and contact pressure on the transfer mechanisms. The obtained information could suggest actions both for manufacturers and researchers involving in the comprehension of this phenomenon and in its technological solution.
Furthermore, a proper management of set-off according to GMP shall be a part of the evaluation and validation of converting processes and compliance of food packaging materials.

Keywords: printing inks, set-off, migration, PPPO (Tenax ®)

1. INTRODUCTION

Set-off is a complex mechanism in which substances used in printing inks on the external side of a material or article are transferred unintentionally on the food contact layer. Mechanisms as blocking, rubbing or peeling are potentially involved in this transfer (BRADLEY *et al*., 2005), especially considering molecules with relevant molecular weights like pigments, dyes and resins; in this case we could observe this behavior since may become, sometime, clearly visible, but also low molecular weight molecules like additives, plasticizers, generally belonging to categories IAS (Intentionally Added Substance) and NIAS (Not Intentionally Added Substance), are easily found on the surfaces of the layers of the different packaging structures (SANCHES-SILVA, 2008). When a material is stored in stacks or reels, the migration of lower molecular weight substances through the substrate can also occurred, giving origin to a non set-off mechanism based on migration (MÜLLER and RÜTER, 2012). In the case of flexible packaging, due to the low thickness of the structures and the high pressure during the storage on the reels, set-off and migration are probably both involved in this transfer. The aim of this work is to evaluate the effect of storage time, temperature and contact pressure on the set-off and migration mechanisms of flexible films, simulating their storage on the reels.

2. MATERIALS AND METHODS

The materials used in this study were the following:

A) PP Coex 20 mm // Paper 50 g/m^2 // PP Coex Met 20 mm; Printed by Rotogravure 9 colors
B) PP Coex Bioriented Bisealing 20 mm; Printed by Flexography, 7 colors
C) Paper 70g/m^2 // PP Coex 20 mm; Printed by Rotogravure 7 colors

Time of storage, temperature and contact load values were selected in the range of 10-30 days, 20-40°C and 2-20 kg$_f$ respectively, using a 2^3 screening design (Unscramble X, Camo Software, Norway). The design matrix is shown in Table 1.

Table 2: Experimental design.

	Time (day)	Temperature (°C)	Load (kgf)
BLOCK 1	30	20	20
	10	20	2
	10	20	2
	30	20	20
	30	40	2
	30	40	2
	10	40	20
	10	40	20
BLOCK 2	10	20	20
	30	20	2
	10	20	20
	30	20	2
	30	40	20
	10	40	2
	30	40	20
	10	40	2

Each film was stacked in such a way the printed layer was in contact with the food contact layer as described in Fig. 1. These two layers were isolated by an aluminum foil.

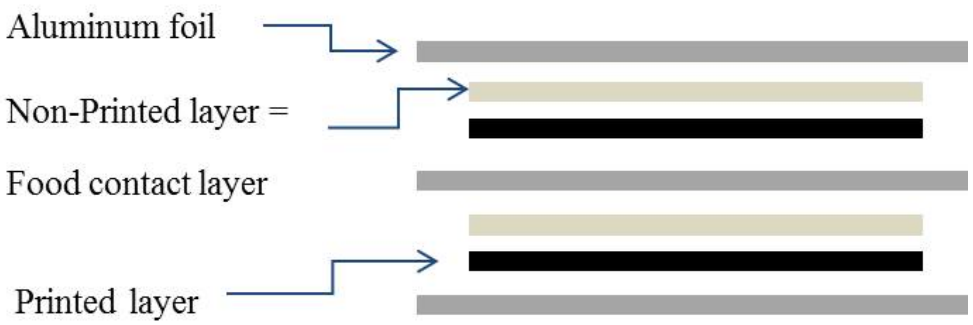


Figure 1: Stack of the films during the storage.

The following steps were carried out on the samples:

1) Solvent extraction of the printed layer before the contact, using n-hexane for 20 minutes using a single side cell. The extract was then analyzed by GC-MS as described subsequently.
2) Storage of the sample under the conditions laid down by the factorial design
3) Single side washing of each food contact layer after the storage with n-hexane. The solution was analyzed by means of a GC-MS
4) Samples (0,18 dm^2) after contact according to the experimental design were put in Petri dishes with Tenax® as food simulant to estimate real migration.

A Perkin Elmer Autosystem XL gas chromatograph equipped with a DB-5MS (30 m, 0.25mm i.d., film thickness 0.25 μm) column, a Merlin Microseal™ Septum Kit installed on the Capillary Inlet system and a Turbomass mass spectrometer was used for the analytical determination. Helium was used as the carrier gas (flow rate 1 ml min^{-1}). The results were expressed as mg/dm^2.

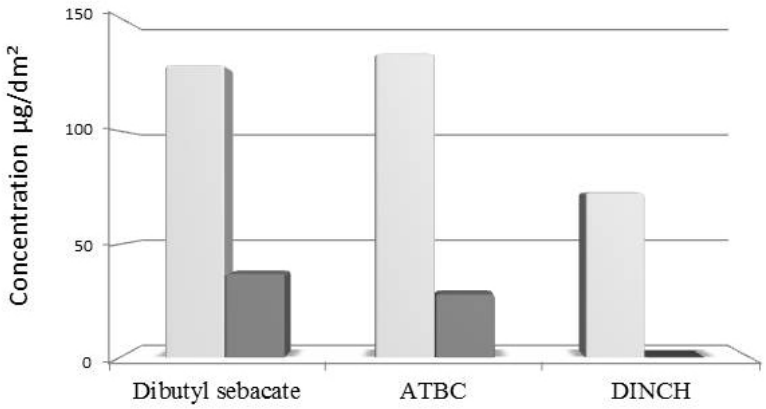
3. RESULTS AND COCLUSIONS

Temperature was the most important factor that influences the set-off and migration of some additives and their degradation products on the food contact layer. The presence of a metallized layer (sample A) reduced the transfer from the printed layer to the food contact side through migration (Table 2 for a specific combination of the DoE). For the other samples without a barrier layer, the transfer is higher, starting from the same concentration of the compounds on the printed layer (data not shown). This could be due to the combination of a set-off and migration phenomena.

Table 2: Mean transfer (%) at 40°C, 10 days and 2kgf (n.d.: < LOQ of the analytes).

Compounds	A	B	C
triacetin	n.d.	n.d.	n.d.
decanoic acid decyl ester	n.d.	n.d.	n.d.
tributyl prop-1-ene-1,2,3-tricarboxylate (Tributyl aconitate)	21,8	98,2	86
sebacic acid, dibutyl ester (Dibutyl sebacate)	23,8	n.d.	n.d.
tri-n-tributyl acetyl citrate (ATBC)	9,6	90,9	64
1,2-cyclohexane dicarboxylic acid diisononyl ester (DINCH)	0,7	n.d.	n.d.
trans-13-docosenamide	n.d.	77,2	n.d.
Octadecyl 3-(3,5-di-tert-butyl-4-hydroxyphenyl)propionate (Irganox 1076)	n.d.	n.d.	n.d.

For those runs that returned quantifiable concentration of the markers, the food contact layer of each film has been submitted to a migration study into PPPO, at 60°C for 10 days. The main results are shown in Fig. 2.
The presence of a metallized layer significantly reduced the migration phenomenon through the layers and into PPPO used as food simulant. Although the results highlighted that the tested materials did not raise safety issues, the applied methodology provides the evidence that different additives and NIAS could be transferred to the contact layer as a consequence of the set-off mechanism.



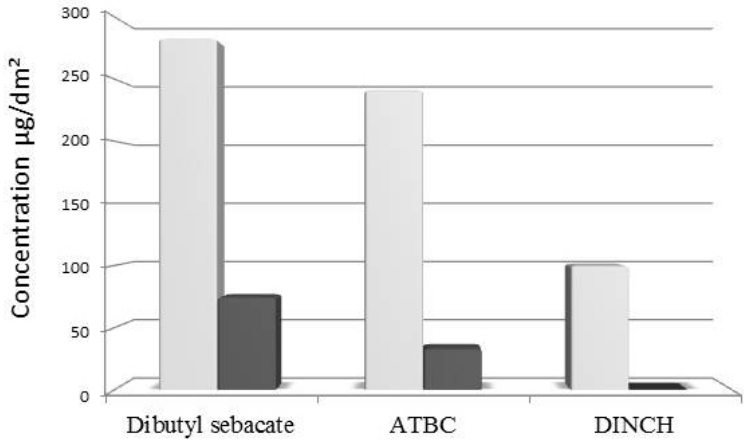


Figure 2: Concentration of substances after set-off at the combination 10 days-40°C-2kg, (light grey bars) and after migration (darker bars) for samples F1 (Fig. 2a) and F2 (Fig. 2b).

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