

PACKAGING

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Packaging challenges for novel processed food

In the last few years, the fast development of novel processing methods for food preservation to improve safety, quality and shelf life of packaged foods gave place to important gaps of knowledge that must be filled in the area of suitable packaging materials. In particular, in the European Project NovelQ (FP6-CT-2006-015710), the effect of novel processing technologies, such High Pressure (HP) as well as microwave (MW) heating on the performances and structural integrity of several types of packaging materials has been investigated along with issues related to food/packaging interactions.

HP treatment is steadily gaining as a food preservation method that maintains the natural sensory and nutritional attributes of food, extending shelf life with minimal quality loss. It consists of applying high pressure (typically in the 300-800 MPa range over a period of several minutes) to packaged foodstuff to greatly reduce the number of microorganisms and also to deactivate enzymes by mechanical action. HP pasteurisation is

conducted at 25 – 40°C while HP sterilisation is conducted at 90 – 110°C.

We discuss here some relevant issues addressed in the NovelQ project related to the effect of HP treatments on packaging materials in terms of mechanical resistance of packaging structures, of the possible reduction of their functional properties (e.g. barrier properties) and of possible migration and scalping phenomena of small molecules in conventional

plastic, novel biodegradable and nano-composite packaging materials. Moreover, in this contribution we also report on packaging/food interaction during MW heating of packaged foodstuff.

Suitability of commercial packaging materials for HP treatments

Packaging materials for HP treatments have to be flexible enough to withstand the compression forces while maintaining physical integrity without losing the properties which guarantee the adequate protection of packaged food^{1,2}. In fact, HP treatment can promote changes in crystallinity, density and orientation of plastic packaging materials that could affect, in turn, mechanical and mass-transfer (vapour and gas barrier, migration/scalping) properties of the package to a significant extent.



Moreover, interfacial stresses arising between the different elements of multilayer structures as a result of the high pressure, could bring about delamination and extensive detachment of the layers.

In the course of the NovelQ project, a systematic analysis has been performed on the behaviour of flexible single materials and multilayer commercial plastic structures (Table 1, page 45), used to package selected foodstuff (i.e. tap water, carrot juice, carrot puree and solid sliced carrots) to be HP treated.

Tests performed on films of LLDPE and of PP confirmed that these materials are capable of withstanding the HP treatment conditions, both for pasteurisation and sterilisation, without displaying significant changes or deterioration of barrier, mechanical and morphological properties, as also reported in the recent literature³. It is worth noting that LLDPE can be submitted to treatments at temperatures as high as 110°C without occurrence of melting, since HP conditions promote an increase of melting temperature.

Referring to laminated structures, the PA cast / PP cast bilayer films were found to be the

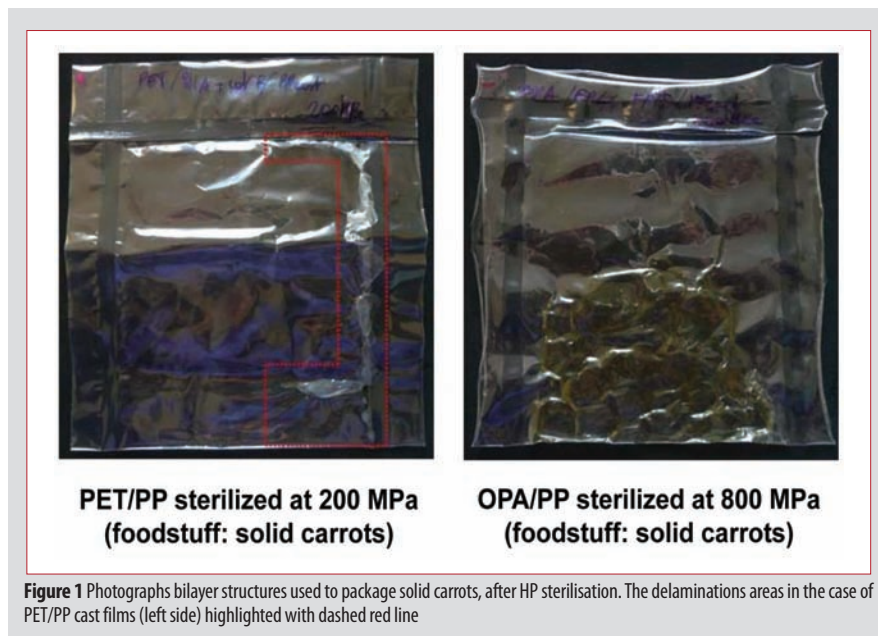


Figure 1 Photographs bilayer structures used to package solid carrots, after HP sterilisation. The delaminations areas in the case of PET/PP cast films (left side) highlighted with dashed red line

most suitable for HP pasteurisation and sterilisation up to 700-800 MPa (see Figure 1, right side), although results were to some degree dependent on the adopted lamination adhesive. Slightly worse results were obtained with OPA/PP cast films, while multilayer structures made of PET/PP were found

unsuitable for HP sterilisation due to delamination phenomena (Figure 1, left side). However, we cannot exclude that better results could be obtained with PET/PP structures made of films processed in different conditions as compared to the materials we used (e.g. different degree of orientation or different heat setting procedures

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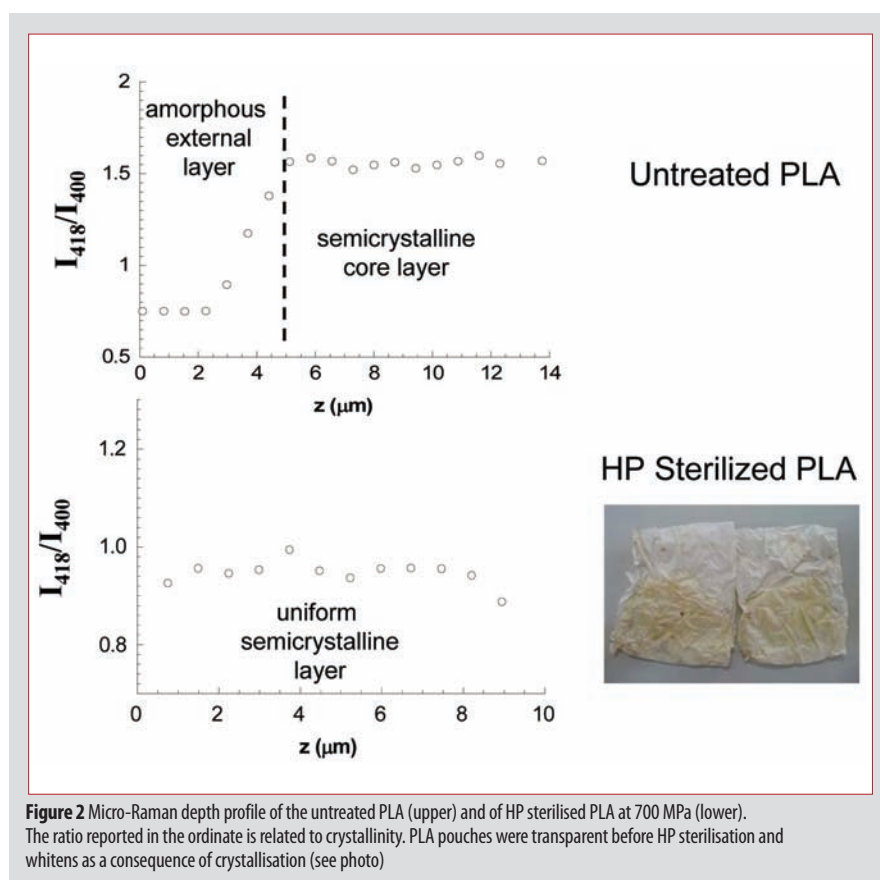


Figure 2 Micro-Raman depth profile of the untreated PLA (upper) and of HP sterilised PLA at 700 MPa (lower). The ratio reported in the ordinate is related to crystallinity. PLA pouches were transparent before HP sterilisation and whitens as a consequence of crystallisation (see photo)

of PET films). In general, the multilayer structures withstanding processing without delaminating did not display significant changes of mechanical and functional properties as a consequence of treatment. Moreover, our results confirmed that films including aluminium foil or metallised layers are unsuitable for HP sterilisation, as already indicated in some literature reports^{1,2,4} due to extensive delamination phenomena.

Finally, tested coextruded structures did not perform as well as best performing laminated structures at the highest pressure / temperature conditions.

The results reported above are in agreement with recent results reported in the literature³.

Experimental results on delamination of multilayer films have been rationalised in terms of a finite element (FEM) analysis of the process of HP treatment of pouches made by using multilayer films containing tap water. The delamination occurrence was attributed to the development of interlaminar normal and shear stresses. To summarise the findings of FEM simulations for the case of bi-layer films, it can be stated that an 'heuristic' measure of the goodness of the coupling between layers is the percentage difference between the

» Contrary to microwave heating, substantial knowledge was still required about the specific effect of pressure on food/packaging interactions at the beginning of the NovelQ project «

Young modulus of one of the films and the overall modulus of the bi-layer structure: the lower this difference, the lower the likelihood of interfacial failure. In practical terms, in the case of examined bilayer structures including PP cast film (i.e. PA cast/PP cast; OPA/PP cast and PET/PP

cast) this rough measure would predict the 'best coupling' in the case of PA cast/PP cast bi-layer films. This is exactly what emerges from the experimental evidences.

Biodegradable packaging materials for high pressure treatments

Suitability of two classes of biodegradable polymers for HP treatments were investigated analysing: i) commercial coextruded multilayer PLA structures and ii) novel laminates based on thermoplasticised zein.

PLA oriented multilayer films, made of two top sealable amorphous PLA layers and of a core of partially crystalline PLA (Treofan 121 by Biophan, 44 μm thick), has been used to prepare pouches containing tap water or solid carrots. The pouches have been treated to HP pasteurisation and to sterilisation. While pasteurisation did not promote relevant variations of the barrier properties of PLA, HP sterilisation treatments promoted relevant changes in crystallinity which was accompanied by brittleness of the material. Figure 2 shows the micro-Raman profiles of the PLA films and a picture of the PLA pouches after the HP sterilisation. The profile analysis in Figure 2 shows that the crystalline amount after the HP treatment (circles) is rather uniform along the thickness, indicating that the crystallinity in the external layers increased as a consequence of the HP treatments.

Lamination of thermoplastic zein proteins (TPZ) with hydrophobic layers of poly- ϵ -caprolactone (PCL) were also developed to prepare biodegradable flexible packaging materials with controlled barrier and mechanical properties suitable for high pressure

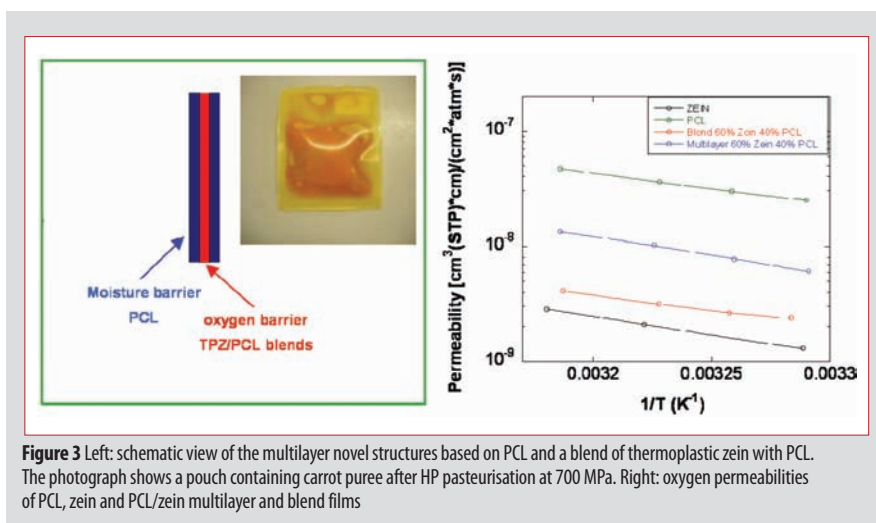


Figure 3 Left: schematic view of the multilayer novel structures based on PCL and a blend of thermoplastic zein with PCL. The photograph shows a pouch containing carrot puree after HP pasteurisation at 700 MPa. Right: oxygen permeabilities of PCL, zein and PCL/zein multilayer and blend films

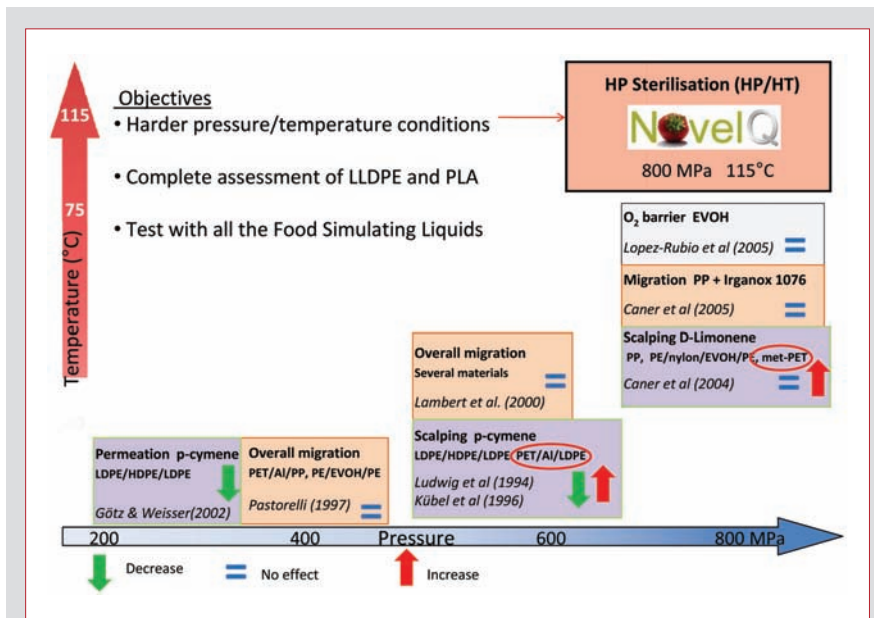


Figure 4 Overview at the starting up of NovelQ project of the available existing studies concerning impact of high pressure processing on food packaging system and synthetic presentation of the objectives of the project to go beyond the state-of-the-art (from Guillard V, Mauricio-Iglesias M, Gontard N. (in press) Crit Rev Food Sci Nutr)

multilayer TPZ-PCL structures revealed the compatibility of such structures with HP treatments at low temperatures (Figure 3, left). In particular, HP treatment up to 700 MPa did not promote detectable changes of mechanical properties as well as oxygen and water vapour barrier properties.

These laminated TPZ/PCL structures were unsuitable for HP sterilisation due to the low glass transition temperatures of the TPZ phase (~50°C) and the low melting temperature of the PCL phase (~60°C). However, we have developed TPZ that incorporates nanolayers of silicates (nanoclay) that results in bio-nanocomposite materials with improved thermo-mechanical properties (increase of glass transition temperature by ~20°C), paving the way to a possible use for HP treatments at temperatures higher than those employed for HP pasteurisation.

pasteurisation treatments. Zein, the prolamine of corn, is of industrial interest mainly for its unique hydrophobicity and barrier to oxygen. When denaturated by applying a suitable plasticiser in combination with heat and shear, zein can be thermoplasticised⁵, in a way similar to thermoplastic starch⁷, and processed by using extrusion blowing technologies⁸ to prepare single layer or multilayer films. Figure 3, page 44 (right hand side) compares oxygen permeabilities of pure zein, pure PCL, multilayer PCL/zein structures and TPZ/PCL (40:60) blend: a tailoring of oxygen permeability can be obtained by a proper selection of PCL and zein structures. HP pasteurisation performed on carrot juice and solid carrots packaged with

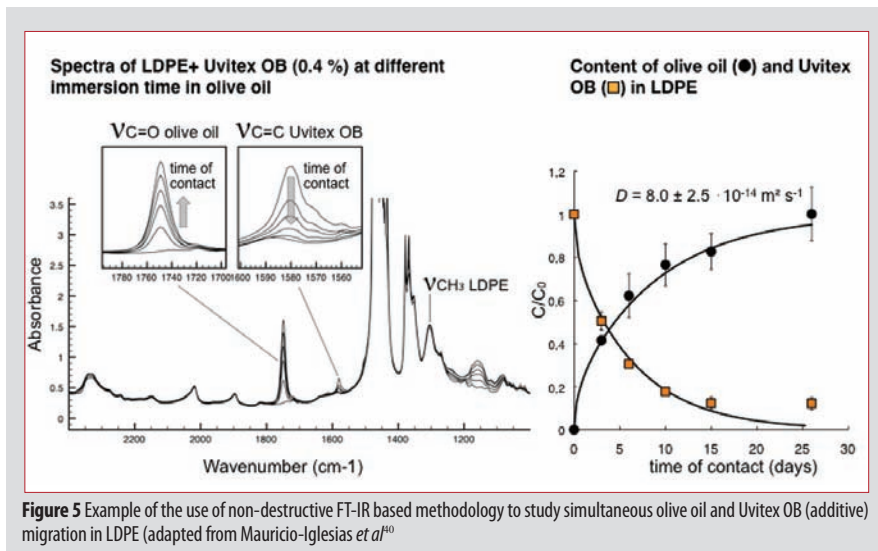


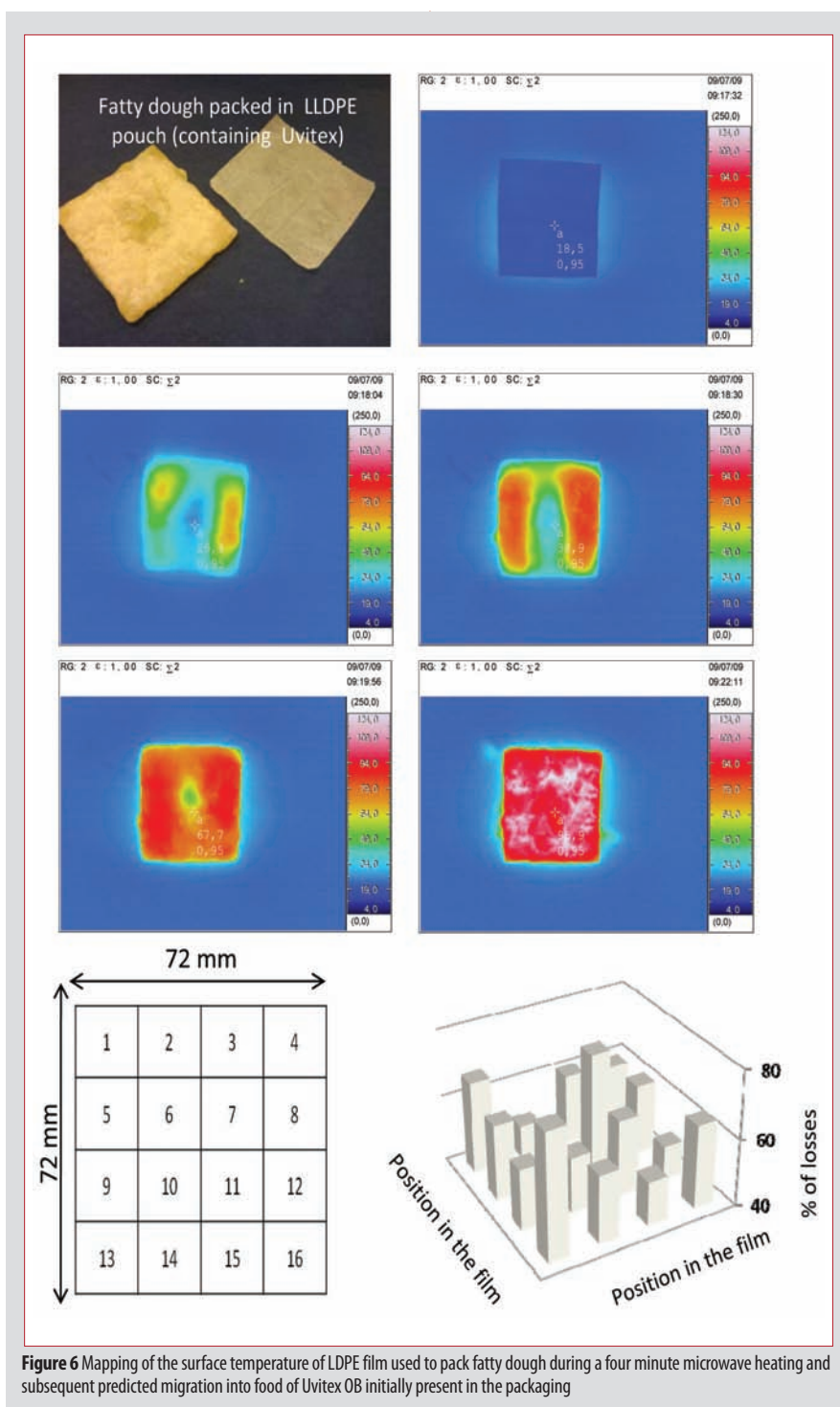
Figure 5 Example of the use of non-destructive FT-IR based methodology to study simultaneous olive oil and Uvitex OB (additive) migration in LDPE (adapted from Mauricio-Iglesias *et al*¹⁰)

Table 1 List of commercial single and multilayer films used for HP Pasteurisation and sterilisation treatments

Material	Abbreviated identification
Single material films	
Linear Low Density Polyethylene	LLDPE
Cast Polypropylene	PP cast
Multilayer laminated films	
Bioriented Polyethyleneterephthalate/Linear Low Density Polyethylene	PET-LLDPE
Bioriented Polyethyleneterephthalate/cast polypropylene	PET-PPcast
Metallized Bioriented PET/ Linear Low Density Polyethylene	PETmet-LLDPE
Bioriented Polyethyleneterephthalate/Polyamide/Aluminum foil/ Linear Low Density Polyethylene	PET/PA/AI/LLDPE
Cast Polypropylene/Bioriented Polyethyleneterephthalate/Polyvinylidene chloride-coated	PP cast/PET/PVDC coated
Cast Polyamide/Cast Polypropylene	PA cast/PP cast
Cast Polyamide/ink/Cast Polypropylene	PA cast/Ink/PP cast
Multilayer coextruded films	
Bioriented Polyamide/Cast Polypropylene	OPA/PP cast
Polypropylene/ Polyethylenevinylalcohol/Polypropylene	PP/EVOH/PP
Polypropylene/Polyamide/Polypropylene	PP/PA/PP

Migration and scalping phenomena in light of food safety

Novel food processing methods, such as high pressure or microwaves, imply that both packaging and foodstuff undergo the stabilisation treatment. During such treatment, the packaging material is involved and exposed to different processing conditions which may alter its structure and consequently its mechanical and mass transfer (barrier and migration) properties. For instance, processing conditions, such as time/temperature couple undergone during conventional or microwave heating, are known to accelerate mass transfer from packaging materials into food. Numerous publications could be thus found on the effect



of temperature on overall and specific migration from plastics materials into food during microwave heating³³. Most of the authors conclude the importance of the high level of temperature that could be reached (specially in the case of susceptor technology) and its heterogeneity in the material^{13,30,31}. Contrary to microwave heating, substantial knowledge was still required about the specific effect of pressure on food/packaging interactions at the beginning of the NovelQ project. As shown in

Figure 4 on page 45, only four studies dealing with this subject were found in literature: two dealing with the global migration^{34,35} and two others with the specific migration of one antioxidant, Irganox 1076³⁶, and a pressure-transmitting fluid, 1,2 propanediol^{36,37}. All these studies were performed on synthetic plastic materials and nothing was known about the expected behaviour of biodegradable materials and 'active' biodegradable materials during and after HPT.

It appeared thus crucial at the beginning of the NovelQ project to (1) understand and clarify the effect of high pressure treatment (especially harder pressure and temperature condition) on the food/packaging interactions of conventional synthetic material but also of biodegradable materials (Figure 4) and (2) to assess the effect of temperature heterogeneities during microwave treatment of a packed fatty food on the migration of a plastic additive.

Effect of high pressure treatment on food packaging interactions

Three materials were studied, namely linear low density polyethylene (LLDPE), polylactide (PLA) and a wheat gluten/montmorillonite (WG/MMT) nanocomposite. Food/packaging interactions were studied after two HP/T treatments intended to perform a pasteurisation (800 MPa, five minutes, 40°C)

» HP treatment is steadily gaining as a food preservation method that maintains the natural sensory and nutritional attributes of food, extending shelf life with minimal quality loss «

and a sterilisation (800 MPa, five minutes, 115°C) treatment, as well as subsequent storage for 10 days. Specific migration of an additive (Uvitex OB) was assessed for LLDPE and PLA, whereas other adapted tests were carried out for WG/MMT, i.e. overall migration, protein migration and nanoparticles migration. HP/T treatments did not significantly modify the migration or scalping in the conditions studied but for the release of nanoparticles from WG^{38,39}. Furthermore, the increase in the melting point of LLDPE allowed the sterilisation of LLDPE whereas it melted when submitted to a conventional thermal sterilisation.

To date the most frequently used methods in migration assessment are based in expensive and time consuming methods based on destruction and quantification. To make easier packaging testing, modelling has recently been approved as a method for migration assessment (directive 2002/72/CE). However, the parameters needed, i.e. diffusivity (D) and the partition coefficient (K) are seldom available. The use of FTIR and Raman spectroscopy to assess migration behaviour and, more importantly, to determine the diffusivity of an additive in LDPE was successfully carried out



allowing a complete characterisation of mass transfer⁴⁰ (Figure 5, page 45).

In the framework of the NovelQ EU project, the French JRU IATE demonstrated that antimicrobial bio-sourced packaging materials may contribute to combine environmental

used alone, even though the quantity of active compound released from the film was lower than the minimal quantity required to obtain inhibition at atmospheric pressure. This combined effect between pressure and antimicrobial agent can be used to reduce the

point of packed fatty dough (worse case for migration of hydrophobic substance such as common additives of LDPE) during the MW heating (Figure 6, page 46). From this mapping, several areas of homogeneous temperature have been selected and the potential migration of Uvitex OB added in the material in contact with food was predicted for each pre-selected area using the mathematical model developed in JRU IATE during the NovelQ project and the diffusivity of Uvitex OB determined using the non destructive FT-IR based method presented above (Mauricio-Iglesias, Guillard et al., 2009). The resulting predicted migration rate show that a large quantity of Uvitex OB could be potentially released in fatty food during less than four minutes of MW heating. It is important to point out that heterogeneities in surface temperature lead to huge differences in level of migration (from less than 50 per cent to more than 70 per cent depending of the temperature). These high levels of migration underlined the roles of both the temperature and the nature of the food in contact (fatty versus aqueous medium) in migration phenomena. Moreover, this experiment illustrated the importance of temperature

Table 2 Combined effect of high pressure processing and antimicrobial activity of allyl isothiocyanate (AITC) and of active PLA-based film containing cyclodextrins encapsulated on the growth of *Botrytis cinerea* during storage (10 days) at 22°C (from Raouche et al. submitted)

	0.1 MPA	300 MPA	600 MPA	800 MPA
CONTROL	+	+	+	+
AITC	+	+	-	-
PLA/CD _{AITC}	+	-	-	-

+ growth of *Botrytis cinerea*; - no growth after 10 days of storage

protection and food quality improvement. PLA-based materials containing AITC previously encapsulated in cyclodextrins was demonstrated to be an efficient optimised antimicrobial system for inhibiting *B. cinerea* growth during at least 10 days at 22°C. Moreover, combining mild HP treatment (around 300 MPa) and use of active film proved to be more efficient for inhibiting *B. cinerea* growth than either a HP pasteurisation-like treatment (800 MPa) or antimicrobial packaging

quantity of active agent used in antimicrobial film and/or to reduce the intensity of the HP treatment (Table 2⁴¹).

Effect of temperature heterogeneities during microwave heating on the food / packaging interactions

The use of IR probe allows the mapping of temperature on food surface during microwave heating (methodology of SIK). This showed the local, surface temperature reached in each

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heterogeneities during MO heating and their impact on migration. These preliminary results need to be experimentally validated and it is worth looking more in-depth in further studies.

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Stéphane Peyron

Stéphane Peyron is Assistant Professor at the University of Montpellier II in the Biochemistry Department. He is a specialist of vibrational spectroscopy (Infrared and Raman spectroscopies) with a particular focus on their application in mass transfer measurement in food-packaging systems. He has been a participant of the European integrated project NovelQ from 2006 to 2010 on the aspect of safety concerns of food contact materials submitted to the novel food technologies and is author of 15 international publications, 10 communications and book chapters.

Valérie Guillard

Dr. Valérie Guillard is a specialist of mass transfer modelling of food/packaging interactions, working at the University of Montpellier II for 10 years. She is the designer of a mathematical model permitting to predict in advance the mass transfer of gas or vapour in a food / packaging system and between the different phases of a food product (e.g. water migration in composite food). This tool requires an accurate knowledge of the parameters describing mass transfer. Dr. Valérie Guillard is thus working on the determination of diffusivity and solubility parameters in food and packaging materials.



Ernesto Di Maio

Ernesto has been Assistant Professor of Materials Science and Technology since 2004 at the Department of Materials and Production Engineering of the University of Naples Federico II. He has achieved a PhD in Materials Technology in 2001 and, since 2001, has been Visiting Research Scholar in the Department of Chemical Engineering, University of Houston, Houston, Advisor Prof. Raimond W. Flumerfelt. The research work is focused on the thermoplasticisation processes of biopolymers and on their processing technologies such as film blowing and extrusion foaming. The aim is to understand the influence of the processing parameters, composition as well as the molecular architecture on the morphology and properties of the film and foam structures.

Miguel Mauricio Iglesias

Miguel Mauricio-Iglesias holds an MSc in Chemical Engineering from the University of Santiago de Compostela and a PhD in Food Technology from the University of Montpellier II. His research deals with mass transfer in polymers applied to membranes and food packaging. Currently, he is a postdoctoral fellow at the Technical University of Denmark.

41. Raouche, S., Mauricio, M., Peyron, S., Guillard, V., Gontard, N., submitted. Antimicrobial activity of bio-sourced material containing Allyl isothiocyanate or Carvacol during HP/T treatments and further storage *Food Microbiology*.



Salvatore Iannace

Salvatore Iannace graduated in Chemical Engineering in 1988 at the University of Naples Federico II, where he gained a PhD in 1991. He is the project head of the department of macromolecular design (Italian National Research Council) on 'Nanostructured, multicomponent polymeric systems and membranes for functional and structural applications' from 2007. He is also adjunct professor of Polymer Technologies at the University of Naples Federico II. His principal research activities include biodegradable polymers, composites and nanocomposites, processing and properties of thermoplastic matrix composites, processing and properties of films and foams. He has published over 100 papers on international scientific journals, books and conference proceedings included invited lectures on foams and nanocomposites. He has been the scientific coordinator of approximately 30 national and international research projects. He organised the first conference on Biofoams (2007) and co-chaired the second Biofoams (2009) in Canada, and the foam symposiums at The Polymer Processing Society 24th Annual Meeting (2008).



Giuseppe Mensitieri

Giuseppe Mensitieri graduated in Chemical Engineering on 1985 at the University of Naples Federico II where he is full professor of Materials Science and Technology and of Macromolecular Materials. He is currently Director of the Bachelor Program in 'Materials Science and Engineering'.

His main field of research is that of polymeric materials with focus on structure – properties relationships. In particular, his research activities concern transport through polymers of low molecular weight molecules, thermodynamics of solute-polymer mixtures, polymer – penetrant chemical-physical interactions, technological applications related to mass transport phenomena.

Giuseppe Mensitieri has been visiting researcher at the Department of Chemical Engineering of the University of Toronto and the Department of Chemical Engineering of the North Carolina State University. He is author of 85 scientific publications on international refereed journals, eight book chapters and of four patents.



Nathalie Gontard

Nathalie Gontard is a Professor at the University of Montpellier and leader of a team working on an integrated approach of the 'packaging / food / processing' systems with a focus on modelling tool, biodegradable, active and nano-engineered materials. She was involved in numerous international (EU and developing countries), national and industrial projects. Currently, she is expert at the EFSA (European Food Safety Authority) and coordinator of the EcoBioCAP EU FP7 project.