Memoria de Tesis Doctoral

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Modelos de innovación en el sector agroalimentario. Ejemplo de desarrollo de un nuevo producto cárnico crudo

Innovation models in the agri-food sector. An example of development of a new raw meat product

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INFORME DE JUSTIFICACIÓN DEL TIPO DE TESIS DOCTORAL PRESENTADA

La presente Tesis Doctoral que lleva el título "*Modelos de innovación en el sector agroalimentario. Ejemplo de desarrollo de un nuevo producto cárnico crudo*" ha sido realizada por la doctoranda **Dña. Ferdaous Zouaghi** bajo la dirección de la **Dra. María Jesús Cantalejo Díez,** Profesora Titular de Universidad en el Departamento de Tecnología de Alimentos y la **Dra. Mercedes Sánchez García,** Profesora Titular de Universidad en el Departamento de Gestión de Empresas.

Esta Tesis está basada en un compendio de tres artículos previamente publicados en revistas internacionales que forman parte del **Journal Citation Reports (JCR)**, con un factor de impacto elevado en el **primer cuartil** y un cuarto artículo que está en proceso de evaluación (tercera revisión) en la revista Technovation (primer cuartil en Management con un factor de impacto de 2.526). Todos estos trabajos están en coautoría con las directoras de esta Tesis Doctoral y de otros miembros del equipo de investigación.

Los artículos que se incluyen en esta tesis doctoral son los siguientes:

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Los primeros trabajos nacen a partir de un proyecto de investigación "Formas de Innovación En El Sistema Agroalimentario y Sus Efectos Sobre Los Resultados

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Además, para la realización de esta tesis doctoral, la doctoranda **Dña. Ferdaous Zouaghi**, obtuvo una ayuda para la formación de personal investigador de la Universidad Pública de Navarra “Modalidad B”. Tanto la doctoranda como las directoras de esta Tesis Doctoral aseguramos que estos artículos incluidos no se han presentado previamente como parte de ninguna otra tesis doctoral.

Dña. Ferdaous Zouaghi

Dra. María Jesús Cantalejo Diez **Dra. Mercedes Sánchez García**

La **Dra. María Jesús Cantalejo Díez,** Profesora Titular de Universidad en el Departamento de Tecnología de Alimentos en la Universidad Pública de Navarra (UPNA) y la **Dra. Mercedes Sánchez García,** Profesora Titular de Universidad en el Departamento de Gestión de Empresas en la Universidad Pública de Navarra (UPNA)

INFORMAN

Que la presente memoria de tesis Doctoral titulada "*Modelos de innovación en el sector agroalimentario. Ejemplo de desarrollo de un nuevo producto cárnico crudo"* elaborada por Dña. Ferdaous Zouaghi ha sido realizada bajo nuestra dirección, y que cumple con las condiciones exigidas por la legislación vigente para optar al grado de Doctor con Mención International. La doctoranda Ferdaous Zouaghi ha realizado dos estancias de investigación en el marco de las Ayudas Predoctorales UPNA, en los siguientes centros: en Kent Bussiness School, University of Kent en Canterbury, Reino Unido (desde el 4 de Junio hasta el 6 de Agosto del 2014) con el objetivo de estudiar la diversidad de las redes tecnológicas sobre el rendimiento innovador de las empresas. Dicha estancia se complementa con una segunda estancia en el Departamento de Chair for Agricultural and Food Market Research en la Universidad de Bonn, Alemania (desde el 4 de Mayo hasta el 3 de Junio del 2015) con el objetivo de analizar los resultados económicos de las empresas agroalimentarias y de los factores que les afectan. Estas estancias en dos centros diferentes se concretó a través de la colaboración entre estos grupos de investigación. Dicha colaboración está relacionada con el tema de Tesis de la doctoranda además de que estas dos estancias de investigación están vinculadas con el objetivo del Proyecto de Plan Nacional AGL2012-39793-C03-01 que lidera el equipo de UPNA (2013-2015).

Y para que así conste, firman la presente en Pamplona, Mayo de 2016.

Dra. María Jesús Cantalejo Diez **Dra. Mercedes Sánchez García**

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DEDICATORIA

Esta tesis está dedicada a mi querido marido Youssef Chahor y mis padres Ali Zouaghi y Latifa Bedda por todo el amor, coraje que me han otorgado durante todos estos años.

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ABSTRACT

In today's highly competitive global environment, a company's ability to introduce innovations is a key success factor for sustaining competitive advantage, particularly in an unstable global macroeconomic environment such as a time of crisis. Changes in consumer preferences over time require faster upgrades in innovation and technology on an industry-wide level. These changes range from basic considerations such as improving food safety, shelf life, and reducing wastage, to demands for increasingly sophisticated foods having special characteristics in terms of nutritional value, palatability, and convenience.

This thesis has two perspectives. One is economic: we study firms' innovation efforts in terms of R&D expenditure and in human capital resources in order to capture the value of its innovation. The other is a practical one: we study the creation of a new high-quality food product through the use of combined food processing technologies. The results of this research are, thus, presented and analyzed from these two perspectives.

The first study begins by looking at which main innovations should be of interest to firms in the sector, then moves to examining how various determinants of innovation (e.g. internal and external sourcing of innovation inputs as well as a specific firm characteristics) allow food firms to improve their innovative performance even in difficult times such as a general economic crisis. In the second study, we try to explain how external R&D cooperation can enhance firms' innovation performance and technological knowledge and the role played by internal capabilities to extract value from those external collaborations. In the third study, the way the development of new products can occur at a pilot-scale level through the introduction of combined food technologies that could lead to improve food quality and provide, at the same time, benefits in terms of a longer shelf life is examined. In the fourth study, we discuss the role of the use of modified atmosphere packaging (MAP) to improve the sensory properties of the new product with regard to the consumer's demands. In this dissertation, we tried to address these questions using a conceptual analysis by developing the applied econometric model explaining the link between different sources of innovation and performance using the Spanish Technological Innovation Panel (PITEC). Furthermore, experimental studies were carried out using combined processing technologies and safety assessment techniques available for meat

applications (i.e. ozonation; freeze-drying and modified atmosphere packaging (MAP)). These technologies offer several benefits, including increased process efficiency, improved product safety, enhanced quality attributes, extended shelf-life and stability of the products.

The major contributions of this thesis to the field of food innovation can be summarized in four outcomes: The first study highlights the importance of innovation in agri-food sectors as a key mechanism for organizational growth and even survival in tough economic times, and a new approach to account for the role exercised by different sources of innovation to improve their innovative performance in the coming years. The second study also contributes to bridging the Resourced-View (RBV) Theory and as a result the significant role of firm absorptive capacities such as knowledge and skills improvement also appear to be key factors for the effective utilization and integration of external knowledge needed for greater innovation performance. The third study also discusses the role of food processing technology for the future application or integration of the food supply chain in achieving safe foods of high quality that represent an alternative, as they would allow extending the retail period in the case of natural catastrophes, military campaigns, export to third countries, scarcity in electricity supply, etc. The fourth study focuses on the benefits of the innovations using modified atmosphere packaging technologies for providing better sensory quality of meat in order to reach more potential markets and satisfy consumer demands.

***Keywords**:* Firm Performance, Innovation, Raw chicken, Meat, Food-processing techniques, Shelf-life.

RESUMEN

Hoy día, en un entorno tan altamente competitivo, las empresas pueden conseguir ventajas competitivas a través de la innovación, en particular en momentos de inestabilidad económica como estos últimos tiempos de crisis. Las empresas se ven obligadas a un continuo cambio o actualización a nivel de la innovación y la tecnología con el fin de mejorar su nivel competitivo. Estos cambios van desde consideraciones básicas, tales como la seguridad alimentaria, ampliar la vida útil de los alimentos, reducir el desperdicio alimentario, hasta la demanda de alimentos cada vez más sofisticados que tienen unas características especiales en cuanto a su valor nutritivo, palatabilidad y conveniencia.

Esta tesis tiene dos perspectivas, una económica, donde estudiamos el esfuerzo innovador de las empresas acumulado en los gastos de I+D y en los recursos humanos para capturar el valor de su innovación. Se complementa con la visión práctica, mediante la elaboración de un nuevo producto de alta calidad a través el uso de tecnologías de procesamiento de alimentos y las combinaciones de varias de ellas. Los resultados de esta investigación se presentan teniendo en cuantas estas dos perspectivas.

En el primer estudio, se trata de analizar los tipos de innovaciones que pueden ser de interés para las empresas agroalimentarias, luego se examina el efecto ejercido por los indicadores de inputs de innovación (los gastos en I+D internos y externos así como las propias características de la empresa) sobre el rendimiento innovador de las empresas, incluso en momentos difíciles, como la última crisis económica. En el segundo estudio, tratamos de explicar cómo la cooperación en I+D puede mejorar el rendimiento innovador de la empresa, así como el papel ejercido por las capacidades internas de las empresas para extraer valor y conocimientos de esas colaboraciones externas. En el tercer estudio, analizamos cómo se produce el proceso de desarrollo de un nuevo producto a nivel industrial a través de la introducción de tecnologías de tratamientos combinados que podrían conducir a mejorar la calidad de los alimentos y proporcionar una larga vida útil de los alimentos. En el cuarto estudio, se discute el papel de la innovación en el envasado en atmósferas modificadas para mejorar las propiedades sensoriales de los nuevos productos. En esta Tesis hemos tratado de responder a estas preguntas mediante un análisis conceptual, utilizando el Panel de Innovación Tecnológica Española (PITEC), donde hemos desarrollado un modelo econométrico que sustenta los factores que afectan a la actividad innovadora y a los resultados de la

innovación. Por otra parte, los estudios experimentales se lograron mediante el uso de tratamientos combinados (ozonización, liofilización y envasado en atmósfera modificada) en alimentos así como la evaluación de la seguridad alimentarias aplicada en el sector cárnico. Dichas tecnologías pueden ofrecer varias ventajas, incluyendo un incremento en la eficiencia de los procesos, así como mejorar la seguridad y los atributos de calidad de los productos durante un almacenamiento prolongado.

Las principales aportaciones de esta Tesis en el campo de la innovación en las industrias alimentarias se resumen en cuatro puntos: El primer estudio muestra la importancia de la innovación en los sectores agroalimentarios como mecanismo clave para su crecimiento e incluso para sobrevivir en tiempos económicos difíciles. Además, supone la influencia ejercida por diferentes fuentes de innovación para mejorar el desempeño innovador de las empresas en los próximos años. El segundo trabajo también contribuye a la Teoría de Recursos y Capacidades, destacando el papel de la capacidad de absorción de la empresa tales como los conocimientos y las habilidades, los cuales parecen ser factores clave para la utilización eficaz y la integración del conocimiento externo necesario para obtener mayores resultados de innovación. El tercer capítulo analiza las implicaciones de las técnicas de procesamiento de alimentos así como sus futuras aplicaciones o integración en la cadena de suministro de alimentos con el objetivo de producir alimentos seguros que respondan a las necesidades del mercado y a las expectativas del consumidor. El cuatro estudio agrega los beneficios de las innovaciones en las tecnologías de envasado en atmosfera modificada para proporcionar una mejor calidad sensorial de los nuevos productos cárnicos para llegar a mercados potenciales y satisfacer las demandas del consumidor.

Palabras clave: Desempeño innovador de las empresas, Innovación, Carne de pollo cruda, Métodos combinados, Vida útil.

CHAPTER I. Introduction

Chapter I. Introduction

1.1. Research motivation

This thesis deals with the challenges of managing innovation processes in the food industry and aims to make a contribution towards understanding how food process innovations can occur at company level in the context of implementing combined food processing technologies that enhance food quality and safety. This thesis has two perspectives: an economic view, through which we study the innovation efforts of firms in terms of R&D expenditure and in R&D human resource investment to capture the value of its innovation. It is complemented by a practical view (technological view), and studies the creation of a new food product through the use of combined food processing technologies to improve the quality and stability of food products.

With regard to the first perspective, this study is motivated by the need to understand the main drivers for innovation performance in the agri-food sector as compared to other non-food companies. It is important to identify which innovation inputs lead to improve agri-food innovation performance despite the recent crisis and thus identify the innovation trends of this sector in the coming years. In this context, this study takes a time frame of 5-8 years, including the current economic crisis period, which will provide insights for food manufacturing managers to define and redirect their strategies of innovation in the future. The second motivation of this study related to the second perspective view of this thesis is the need to understand how food-processing techniques will play a vital role in food quality and security. Combined processing techniques provide the opportunity for the food industry to adapt itself to the changing food market and to consumers' needs, given that the modern societies demand safety and quality.

1.2. Concept of innovation and innovation measurements

Innovation is considered one of the most important business drivers for companies' growth and is also one of the important sources and enabler of competitive advantages (Capitanio et al., 2009). According to the Organization for Economic Cooperation and Development (OECD, 2005) "*Innovation is the implementation of a new or significantly* *improved product (good or service), or process, a new marketing method, or a new organizational method in business practices, workplace organization or external relations*". The OCDE (2005) classifies four types of innovation: product, process, organization and marketing. Product and process innovations are often considered to be technological innovations while marketing and organizational are thought of as non-technological.

A **product innovation** is the introduction of a good or service that is new or significantly improved with respect to its characteristics or intended uses. This includes significant improvements in technical specifications, components and materials, incorporated software, user friendliness or other functional characteristics.

A **process innovation** is the implementation of a new or significantly improved production or delivery method. This includes significant changes in techniques, equipment and/or software.

A **marketing innovation** is the implementation of a new marketing method involving significant changes in product design or packaging, product placement, product promotion or pricing.

An **organizational innovation** is the implementation of a new organizational method in the firm's business practices, workplace organization or external relations. Organizational innovations can be intended to increase a firm's performance by reducing administrative costs, improving workplace satisfaction, or reducing costs of supplies.

Innovation also encompasses both **radical** and **incremental innovation**. Research generally identifies an innovation as either radical or incremental by determining the degree of change associated with it. More precisely, **radical innovations** lead to entirely new products that are new to the market, whereas **incremental innovations** lead to improvements in existing products and are new to the firm (Ettlie et al., 1984).

All above types of innovation can be defined as **outputs** of innovation, which measure the shortest term for success of an innovative activity of the firm that results from inputs. **Input indicators** are those factors, influences or conditions that support the innovation process and are used as a proxy for the level of innovative effort. These indicators include expenditure on R&D (intramural R&D comprises all R&D performed within the enterprise and extramural R&D comprises the acquisition of R&D services), innovation investment expenditure (expenditure on machinery and equipment in order to implement new or significantly improved products or processes), investment in human resources and skills for innovation (e.g. cost of staff training, workshops, upgrading qualifications), among others (OCDE, 2005).

1.3. Innovation in the food sector

The food industry is one of the most important in the European Union and it is highly significant in terms of economic output and employment (Hirsch & Gschwandtner, 2013). In innovation literature, the European food industry has been shown to particularly invest much less in R&D when compared to other industries and radically new products are rare (Costa & Jongen, 2006; Bigliardi & Galati, 2013).

However, food and drink companies both within and outside the EU have continued to show resilience in the economic crisis, maintaining similar levels of R&D investment (Chamorro et al., 2012). In Spain, the percentage of innovative firms in the sector is now similar to the average for industry as a whole, the number of food firms that have invested in R&D in the period 2012–2014 having multiplied by four. According to Spain's INE (2014), 37.78% of food firms introduced a product innovation in the 2010–2014 triennium, and the turnover generated by these innovations was 35.35% of the total of those firms. However, very few new products survive in the long term; about 80% of those new products are expected to fail within the first two years after their launch into the market (Tsimiklis and Mkatsoris, 2015). When investigating the reasons for the low success rates, studies concluded that failed product innovators did not fully understand customer needs, or even that they launched products without taking into consideration the realities of those who will use the product (Dougherty, 1992).

In order to produce and successfully commercialize innovation, firms must synthesize a wide variety of expertise and knowledge produced by different complementary sources (Muscio, 2007). The collaborative approach to innovation, termed "open innovation", can be contrasted with the traditional "closed" approach to innovation, which entails the complete integration of Research and Development (R&D) within the boundaries of a firm (an option not best suited to the strained resources of smaller food companies) (Bigliardi and Galati, 2013; Hudnurkar et al., 2014). Firms' collaboration with external institutions allows the expansion of their range of expertise and can support the development of new products. However, in order to successfully access new knowledge through collaborations with firms and institutions, firms must manage their absorptive capability to ensure the effective utilization and integration of external knowledge needed for development of new products (Haeussler et al., 2012).

Open innovation represents a vital source of knowledge for most foods in order to gain and sustain their competitive advantage; they have to deliver the best customer value at the lowest possible costs (Bigliardi and Galati, 2013; Hudnurkar et al., 2014). The customer is increasingly demanding healthy food products, free of conventional chemical preservatives. For this reason, food industry innovations are often aimed at developing important replacement products, following nutritional directions, or acting upon food additive regulations. Innovations may occur throughout all parts of the food chain and a possible classification of the food innovations is the following: (1) new food ingredients and materials, (2) innovations in fresh foods, (3) new food process techniques, (4) innovations in food quality, (5) new packaging methods, and (6) new distribution or retailing methods (Bigliardi and Galati, 2013). These trends in innovation in foods and drinks are also applicable to the meat industry. Innovation in meat products has become a global necessity given the forecasts of future meat consumption and the resource constraints facing livestock production.

The consumption of meat and meat products, which contain important levels of proteins, vitamins, minerals and essential micronutrients, is growing in developing countries. Meat processing provides the opportunity to add value, reduce prices, improve food safety and extend shelf life. The Livestock in Food Security report (FAO, 2011) estimated a nearly 73% increase in meat consumption from 2010 to 2050. Fresh meat is the most perishable food among all the important foodstuffs due to its nutritive compounds (Jay, 1992). Microbial growth is the main cause of meat spoilage, which results in off-odors and off-flavors, as well as textural defects (Sun and Holley, 2012). The growing concern for health has led the meat industry to introduce new products to meet rapid changes in consumer tastes and demands for healthier food products, safe, natural, free of conventional chemical preservatives and with an extended shelf-life. Another factor influencing the need for innovation has been the series of food crises in recent years and the effect they have had on the legislation affecting the sector and also the consumer confidence: i.e. the so-called “mad cow” disease, avian influenza, and blue-tongue disease (Chamorro et al., 2012). All of them have led the meat industry to search for novel and innovative ways of processing meat for maintaining quality and safety in order to maintain and expand new markets (Troy et al., 2016). The hurdle technology is a combination of two or more different control techniques which have been proven to be effective for controlling spoilage/ pathogenic microorganisms in food products. The principle of this technology can be explained as two or more inhibition and inactivation methods (hurdles) at suboptimal levels being more effective than one (Leistner, 1992). This method is becoming attractive, because a series of hurdles are used to obtain optimum combinations which do not affect the sensory quality, while maintaining the microbial stability and safety of food (Alzamora et al.1993; Leistner, 1992).

1.4. Development of the Research and the Overview of Thesis

This thesis consists of four chapters complemented by this introduction and a concluding chapter. In this section, we provide a brief overview of different papers and the respective research questions they address. The objective of this research is twofold: first, to identify the determinants of different types of innovative inputs (R&D and technological acquisitions) and their relationship with different innovative outputs and to provide some insights that help direct strategies for innovation in the coming years. Second, to explain how the innovation process occurs within the food industry where we take as an example "a new raw meat product from *Broiler* chicken breast".

The first two studies (Chapters 2 and 3) are focused on the innovation managing processes in industry and show what happens to factors (inputs) that likely drove firms' innovative performance. The remaining studies (Chapters 4 and 5) show food processing technologies required to improve shelf life and food safety. In this way, we believe that this dissertation allows us to analyze in detail the complete innovation process.

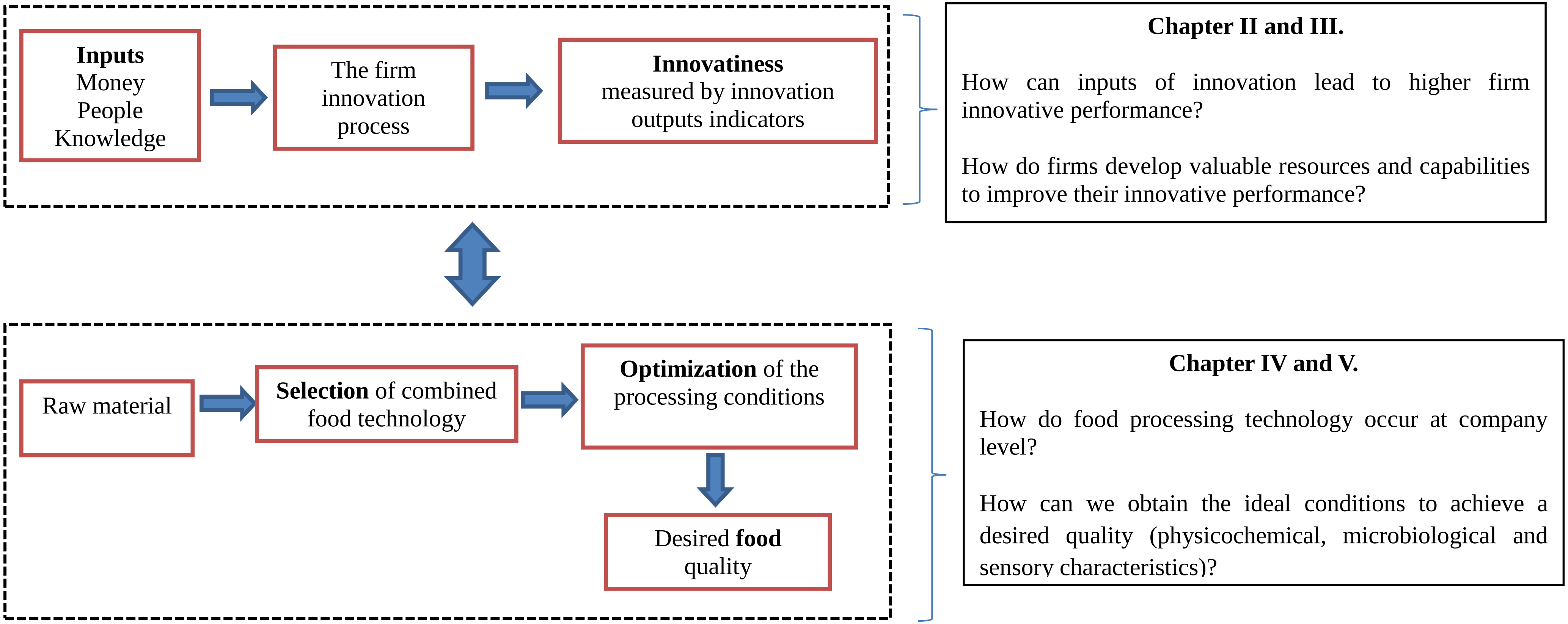

Figure 1. 1. The scope of this thesis

Chapter 2- Has the global financial crisis had different effects on innovation performance in the agri-food sector by comparison to the rest of the economy?

This first study provides an extensive literature review of work relevant to research on agri-food firm performance and innovation and provides a first comprehensive overview on the effect on the global financial crisis on firms' innovative performance and on the effort made by the firms in assigning resources for R&D. Then studies on the factors fostering innovation in firms are reviewed by focusing on various internal and external resources which impact upon the innovation and performance of food industries during a downturn. It outlines the key indicators of innovation, such as R&D expenditure (internal and external R&D), cooperation agreements, propensity to innovate, sales from new products etc., supplemented by firm specific factors. The focal point of this thesis is technological innovation. Furthermore, it is acknowledged that organizational and marketing innovation can facilitate technological innovation processes (OECD, 2005), and is therefore an integral part of the innovation process.

This chapter explores the idea that the economic crisis has had a significant and negative impact on firms' innovative performance and on the effort made by the firms in assigning resources for R&D. A crisis affects technological innovations to a great extent, as well as small companies, those which carry out less internal R&D and cooperation efforts. However, innovating firms have been proved to obtain better results both in economic and productive terms. Further to that, the agri-food sector innovative behavior has been less affected by the crisis than other economic sectors. Our finding also has implications in understanding the role of some innovation inputs in helping firms to manage better innovation strategy during the recent crisis. For instance, engagement in internal R&D activities not only influences the process of innovation but also has a substantial role during a crisis in explaining the counter-cyclical behavior of firms (i.e. persisting in innovation). Our empirical evidence also confirms the importance of cooperation with different partners as an attractive strategy for Spanish businesses in times of crisis to reduce costs and share the risks of innovation. This highlights the importance of the "absorptive capacity" notion observed by Serrano-Bedia et al., (2012) (among others), when it comes to taking advantage from this external knowledge. Firms need to develop absorptive capability by building knowledge stocks through investment in internal R&Dto better benefit from external knowledge sources. A series of firm characteristics may also stimulate firms to innovate; the findings also show the important role of human capital in cushioning the effect of crisis in innovation activities.

Chapter 3- Capturing Value from Alliance Portfolio Diversity: The Moderating Effect of R&D Human Capital

Innovation is essential to success in the food sector. Since most food firms do not have the competencies or the capital needed to innovate on their own, they need to find partners to join forces in open innovation collaborations in search of successful new products and technologies. Access to new knowledge through collaborations with external partners can help firms reduce both their R&D costs and also the total product development time, especially in times of crisis. However, low-technology sectors like food industries often face difficulties in establishing a strategic and efficient network.

This study is a continuation of the previous chapter and has a detailed investigation of the collaboration between supply chain partners of firms (e.g. with other firms, consultants, universities, competitors, and customers) to achieve new products. Research has demonstrated the value of external linkages to increase in-house R&D efforts, but very little is known about how managers can operationally leverage the potential benefits of open collaborative modes of innovation to create an innovative edge. This chapter explores how low technology sectors (e.g. the food sector) use their cooperation networks compared to high-technology sectors for improving their innovation performance.

Additionally, this chapter informed our understanding of how firms develop valuable resources and capabilities to take value from open strategies. In this sense, the moderating effect of R&D human capital–education and skills– on the alliance portfolio diversity-innovation performance relationship is explored. Using data from the Spanish Technological Innovation Panel (PITEC) for the 2005–2012 period, random-effects panel Tobit models support the curvilinear (inverted U-shaped) relationship between alliance diversity and innovation performance reported in studies; however, the value of alliance diversity is more accentuated in high-technology industries, particularly in radical innovation performance given the technological complexity, market uncertainty and divergent skills set required for breakthrough innovations in high-technology sectors. Further, we found evidence that the value of alliance diversity on innovation performance is contingent upon firms' R&D human capital, which emphasizes the importance of internal capabilities to effectively integrate external flows of knowledge into innovation processes. This study provides valuable insights to managers aiming at increasing the effectiveness of their alliance portfolio.

Chapter 4. Combined effects of ozone and freeze-drying on the shelf-life of broiler chicken meat

This paper deals with combined food technologies which have shown potential for meat processing applications. The microbial stability and safety of most traditional and novel foods is based on a combination of several preservation factors (called hurdles), that the microorganisms present in food are unable to overcome. In achieving the desired safety through only one hurdle, great care in processing needs to be applied and generally causes significant damage to the nutritional and sensory quality of foods. For this reason, it is important to have a multi-hurdle approach for developing safe and wholesome food products (Rahman, 2015). The multiple hurdle concept is becoming an attractive technology given that a series of hurdles are used to obtain the optimum combinations which do not affect the sensory quality, while maintaining the microbial stability and safety of the food (Alzamora et al., 1993; Leistner, 1992). Hurdle technology is generally defined as using the simultaneous or the sequential application of factors and/or treatments affecting microbial growth (Turantaş et al., 2015). In this chapter, ozonation and freeze-drying were employed as hurdles to develop a new raw meat product from broiler chicken breasts. Ozone is a powerful antimicrobial agent very effective in destroying a wide range of microorganisms including viruses, bacteria, fungi, protozoa, and bacterial and fungal spores (Khadre and Yousef, 2001). Freeze-drying is the most common form of food preservation to improve the long-term stability of food because the percentage of humidity and the water activity can be reduced if the product is well lyophilized, which retards the growth of microorganisms for a long period. The shelf-life of the chicken meat samples was determined using both microbiological and sensory analyses during eight months of storage. The combined effect of gaseous ozone and lyophilisation in chicken breast meat showed great antimicrobial effectiveness due to the action of ozone as well as the low percentage of humidity (<10%) and water activity below 0.5 of the product. These techniques also allowed for extending the shelf-life of those products over eight months of storage at room temperature without refrigeration. However, the combination of those hurdles were not sufficient to maintain the physicochemical (texture) and sensory qualities of the ozonated dried meat for a long time (Cantalejo et al., 2016).

Chapter 5. Study of modified atmosphere packaging on the quality of ozonated freeze-dried chicken meat

This chapter is a continuation of the previous chapter, and focuses on using modified atmosphere packaging technologies, which also have shown to be an effective way of controlling spoilage/ pathogenic microorganisms in new products by maintaining their quality longer. Modified Atmosphere Packaging (MAP) is a technique for modifying the internal gas atmosphere of the food package in order to to slow deteriorative reactions inside the package and to prolong shelf life of the product (Nair et al., 2015), carbon dioxide, oxygen and nitrogen, being the most commonly used gases in MAP. Therefore, the aim of this study was to evaluate the effects of MAP on the physicochemical and sensory properties of ozonated freeze-dried chicken meat stored at room temperature in order to obtain a new raw high-quality meat product with no preservatives and stable over time at room temperature. This is the first time that these three combined techniques (ozonation, freeze-drying and MAP) have been applied on poultry meats.

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CHAPTER II. Has the global financial crisis had different effects on innovation performance in the agri-food sector by comparison to the rest of the economy?

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Chapter II. Has the global financial crisis had different effects on innovation performance in the agri-food sector by comparison to the rest of the economy?

2.1. Introduction

The agri-food industry is one of the most important sectors in the European Union and it is highly significant in terms of economic output and employment (Hirsch & Gschwandtner, 2013). In addition, it is a leading industrial sector in the Spanish economy and the fifth largest in Europe (Alarcón, Polonio, & Sánchez, 2013); it plays an important role in Spain's economy contributing 7.2% of its GDP and more than 20% of total employment (Spanish Food & Drink Industry Federation, 2014). Traditionally, the agri-food sector is considered a Low-Tech intensive industry and the evidence supports the view that a firm's returns and growth depend on its capacity to innovate (Capitanio, Coppola, & Pascucci, 2009). This is because European food markets are characterized by high market saturation and strong competition (Hirsch & Gschwandtner, 2013) and it allows firms to grow more quickly and be more profitable than non-innovators (Atalay, Anafarta, & Sarvan, 2013).

Nowadays, the globalization and expansion of financial markets and the current economic crisis are changing the rules of the economy. Innovating in times of crisis is seen by many authors as an opportunity to grow, survive and succeed and as the attempt to maintain or develop competitiveness in today's global markets (Kühne, Vanhonacker, Gellynck, & Verbeke, 2010; Mohezar & Nor, 2014; Peters, Shane, & Torgerson, 2009). Despite the importance of innovation during crisis, most of the empirical literature dealing with the impact of an economic crisis on innovation has focused only on firms' innovation investment (Paunov, 2012) or on customer behaviour (Ásgeirsdóttir, Corman, & Noonan, 2012; Mansoor & Jalal, 2011).

However, this study focuses on analyzing the overall effects of an economic crisis, both in terms of innovation inputs and innovation performance. On this background, the overall objective of this work is to examine the impact of the economic crisis on the probability of Spanish firms to introduce innovations and on innovative sales opened up by a new product. In this sense, we studied firms' decisions to engage in innovation taking into account all the types of innovation described by the Oslo Manual (OCDE, 2005) i.e. technological and non-technological innovations. Additionally, we measured performance in terms of the market success of firms' innovations according to the share of turnover generated by new products.

We chose the Spanish case because it is one of the countries which have suffered most seriously from the financial crisis in the EU (Sinitsky, 2013).

Finally, this paper developed a conceptual model highlighting different innovation indicators which impact on the innovative performance of firms related to the past literature like business factors (in-house R&D; external R&D; domestic and foreign cooperation in innovation) and the international strategy of the firm measured by export operations.

2.2. Literature review

2.2.1. Source of innovation in the agri-food sector

Agri-food industries are traditionally regarded as a sector with low levels of R&D intensity (Capitanio et al., 2009; Grunert et al., 2008), which has been confirmed to be true in the case of Spain (Garcia Martinez & Briz, 2000). Despite relatively low R&D investments, innovation for this sector has become an important instrument in the turbulent environment that increasing globalization creates, which includes changing quality demands and price discount fights among retailers (Batterink, Wubben, & Omta, 2006). Food firms are mainly process-innovation oriented (Batterink et al., 2006) and both product and process innovation are to a large extent characterized by incremental rather than radical changes (Bayona et al., 2013; Fortuin & Omta, 2009; Hervas-Oliver et al., 2014). The importance of incremental innovation is associated with constraints on the demand side (including retailer behaviour) and conservative consumer behaviour (Capitanio et al., 2009; Filippaios, Papanastassiou, Pearce, & Rama, 2009).

Regarding the origin of agri-food innovations, a large part of them seem to start from customer and retailer demands, marketing strategies, consumer perception of quality and safety and environmental pressure[1]. Vanhonacker et al. (2013) indicate that few innovations are widely accepted by consumers in this sector, where 50% of new products launched on the market fail (Ronteltap, van Trijp, Renes, & Frewer, 2007). Consumer acceptance is crucial to the adoption and dissemination of new technologies in food production and to the success of any new product launched on the market (Garcia Martinez & Briz, 2000). Additional detailed

[1] The implementation of food safety management systems has grown significantly in the food production chain in order to improve food security. European food safety regulation covers a broad range of regulatory techniques and standards including the GlobalGAP, IFS, Marks & Spencer's Field-to-Fork, Tesco Nurture, (Kirezieva et al., 2015).

knowledge of consumer preferences in terms of food technology innovations can help minimize innovation failure rates (Chen, Anders, & An, 2013). In this context, marketing innovation plays an important role in the food sector apart from product and process innovation when it comes to creating information exchange between producers and consumers and to the success of new food products in the market.

Particularly in times of crisis, when consumers' confidence and overall consumer expenditures are greatly affected, both the demand and the supply side pay great attention to the price trends of food products (Koutsimanis, Getter, Behe, Harte, & Almenar, 2012). The foregoing considerations are based on the literature and indicate the importance of all types of innovation in the agri-food industry. Firms in this sector tend to innovate so as to stand out from their competitors at all times and fulfil the needs and expectations of their customers, particularly in times of crisis, and also to sustain prosperity, attain long term goals and develop competitiveness in today's global markets (Kühne et al., 2010; Mansoor & Jalal, 2011).

2.2.2. Determinants of firm innovative performance

This section describes the conceptual framework built on the basis of the Resource-Based View (RBV) (Berney, 1991) and the Dynamic Capabilities Theory (Teece et al., 1997) to analyze how firms may adapt, assimilate and deploy their behavior, resources and capabilities within a changing environment. Using the Resource-Based View (RBV) of the firm as a theoretical backdrop; we aim to find out the relative impact of different activities beyond formal R&D (internal and external), sources of innovation outside firms' boundaries (domestic and foreign cooperation in innovation) and firms' internal characteristics (firm size, business sector and productivity) on their short- and long-term competitive position. Extending the RBV theory, we build on the Dynamic Capabilities Theory to examine why and how some firms have handled the current crisis better than others and how factors (inputs) allow firms to effectively face the crisis by improving their innovative performance during such periods. We argue that managers of firms that want to achieve competitive advantage need to adapt, integrate and reconfigure resources and competences to match the changing market (Makkonen, Pohjola, Olkkonen, & Koponen, 2014; Teece et al., 1997). We summarize our arguments in a set of hypotheses listed below.

Firm factors

The first determinant on firm innovative performance is Research and Development activities (R&D). R&D is considered to be one of the key drivers for innovation (Bascavusoglu-Moreau & Tether, 2012). R&D has a particularly successful impact on innovation efforts when firms carry it out in a continuing way (Köhler, Sofka, & Grimpe, 2012). Moreover, a strong set of internal competencies in R&D not only increases firms' innovative outputs but also allows them to use and exploit knowledge acquired outside the firm (Artz, Norman, Hatfield, & Cardinal, 2010). In this regard, some authors find that the different options for using innovation inputs (internal or external) affect innovation performance more than the R&D effort in general terms (López Rodríguez & García Rodríguez, 2005).

However, the rapid advance of technological knowledge, the growing costs of R&D and shorter product life cycles make it impossible for any firm to sustain all the abilities and knowledge required for production in-house (Berchicci, 2013). In this line, Koschatzky (2001) suggests that firms which do not exchange knowledge in innovation reduce their knowledge base on a long-term basis and lose the capability to enter into exchange relations with other firms and organizations (Avermaete, Viaene, Morgan, & Crawford, 2003). According to this agreement, firms should open their R&D activities to external sources as the externalization of R&D activities allows firms to search for new external knowledge sources outside their environment to benefit from complementary sets of knowledge from external agents and improve their performance and innovate successfully. There is agreement in the literature that the agri-food industries are slightly more open than other Spanish firms in this regard (Bayona et al., 2013). Furthermore, it is crucial for firms to be able to identify and exploit the significant value of external knowledge from other sources of innovation. This capability enhances the firm's absorptive capacity introduced by Cohen and Levinthal (1989), who argue that internal R&D investments are necessary for firms not only to increase innovative outputs but to enhance their capability to assimilate and exploit better sources of knowledge generated outside its boundaries effectively. Firms that depend totally on external partners sometimes lack internal R&D processes themselves and the ability to fully capture and assimilate external knowledge (Chesbrough & Teece, 1996), which suggests that external knowledge should be used to complement rather than substitute for internal R&D (Vega-Jurado, Gutiérrez-Gracia, & Fernández-De-Lucio, 2009). However, previous studies have found empirical evidence that firms with international R&D are more likely to generate innovative products and achieve higher sales growth due to these new products as compared to firms that innovate domestically only (Peters & Schmiele, 2010). This suggests that the internationalization of R&D increases the chances of firms participating in international knowledge sharing. Foreign knowledge will increase firms' innovativeness and market success with innovations when they possess the necessary abilities to make use of their knowledge base. A key reason for firms to go abroad with R&D activities is getting access to new knowledge not available in their home country (Dachs, Borowiecki, Kinkel, & Schmall, 2012). In line with this, we put forward the following hypotheses:

H1.a. Firms that carry out internal R&D will see a positive impact on firm performance in relation to firms that do not.

H1.b National or international external technology acquisition positively correlates with firms' innovative performance.

H1.c. The effect of international R&D can be expected to be stronger than national R&D.

As a consequence of the recent financial crisis, many companies have been forced to reduce their investment in innovation. Milić (2013) suggests that investments in innovations and future growth are at risk during an economic crisis, when most organizations cut their R&D budgets. Paunov (2012) shows that in Latin American countries the current crisis has led many firms to put a halt to ongoing innovation projects. Moreover, Filippetti and Archibugi (2011) note that in certain countries in Europe the percentage of firms reducing investments in innovation is higher than those increasing their innovation expenditure. Similarly, Cincera, Cozza, Tübke, and Voigt (2012) highlight the fact that a large percentage of companies in Europe have reduced R&D activities as a result of the crisis. Given the decrease of R&D efforts during a crisis, we hypothesize that:

H1.d. It is to be expected that the positive effect of internal R&D on firms' innovative performance will be lower during economic crisis.

H1.e. It is to be expected that the positive effect of external R&D on firms' innovative performance will be lower during economic crisis.

The second determinant of innovation performance is cooperation agreements, they is one of the dimensions of open innovation and an additional knowledge sourcing strategy. Cooperation with external partners has proved to be essential in the case of SMEs, where the cost of innovation is more significant as compared to other sectors due to their limited labor, financial and material resources (Laforet, 2013). Bayona et al., (2013) found that cooperation in Spanish agri-food firms has a positive effect on innovation performance. However, firms have opportunities to cooperate with different kinds of partners, namely national, international, industrial and institutional partners. Cooperation with a specific type of partner is generally more likely to be chosen if that type of partner is seen as an essential source of knowledge for innovation success. Belderbos, Carree, and Lokshin (2004) used Dutch data on innovating firms and found that competitor and supplier cooperation is associated with incremental innovations, whereas customers and universities are important sources of knowledge for firms pursuing radical innovations. Similarly, Harhoff, Mueller, and Van Reenen (2014) highlights the fact that collaborations with customers are intended to adapt existing products to new markets and can boost sales of products abroad. Due to international economy integration, R&D cooperation is not limited by national borders. Some studies have found a positive impact of international R&D cooperation on innovation performance. Arvanitis and Bolli (2013) analyzed the differences between national and international innovation cooperation in five European countries: Belgium, Germany, Norway, Portugal and Switzerland, and found that innovation performance of firms improves with international cooperation but remains unaffected by national cooperation. Miotti and Sachwald (2003) studied French manufacturing firms and showed that innovation performance is not affected by innovation cooperation agreements with national partners but is positively influenced by cooperation with foreign partners. However, Jaklic, Damijan, and Rojec (2008) find positive effects of national but not of international innovation cooperation in Slovenian firms. During the latest years of crisis, cooperation has become a more attractive strategy to cope with it for Spanish business; this is particularly the case with SME firms, which have considerably increased cooperation. Given the double aim of the collaborative strategy; pooling knowledge and sharing development costs, this strategy should increase in periods of economic downturns (Laperche, Lefebvre, & Langlet, 2011) so as to preserve the innovation capacity of firms. In line with the empirical studies above, we expect that an economic crisis will lead to the development of collaborative strategies (Laperche et al., 2011). Hence, the following hypotheses are proposed:

H2.a. Cooperation agreements with different national partners will have a positive effect on the innovative performance of the firms.

H2.b. Cooperation agreements with international partners will have a positive effect on the innovative performance of the firms.

H2.c. It is to be expected that this positive effect of cooperation agreements on firms' innovative performance will be easier to be perceived during economic crisis.

Numerous studies have shown that the export variable is important in a firm's ability to innovate. Firms competing in international markets are under intense innovation pressure in general, which reveals itself in a constant need to provide innovative products to remain competitive (Kirner, Kinkel, & Jaeger, 2009). Almeida and Fernandes (2008) found that firms that export are more likely to innovate than firms selling only to the domestic market. Nieto and Santamaría (2007) also showed that export intensity has a positive and significant effect on the likelihood of achieving incremental innovations. However, in the current crisis exporting has become an attractive and sustainable route to survive and get out of recession not only for large companies but also for many SMEs. Peters et al., (2009) argue that a weaker dollar would be beneficial for the American agricultural sector since it would result in higher export earnings, higher commodity prices, and an increase in production. Monreal-Pérez, Aragón-Sánchez, and Sánchez-Marín (2012) suggest that the economic crisis has driven firms to sell their goods and services abroad. Because of the decrease in domestic demand, firms have found that their products are more difficult to sell in their local markets. In most cases, the motivation of firms to expand their markets seems to respond to the need to survive a global market and to achieve a more stable competitive position (Filipescu, Rialp, & Rialp, 2009). Hence, we propose the following hypothesis:

H3.a. The export variable is positively related to innovative firm performance.

H3.b. It is to be expected that his positive effect will be higher during economic crisis.

Firm internal characteristics

Although business factors are key drivers of innovation performance, the role of firm internal characteristics cannot be neglect. Firm size, business sector and productivity, have a considerable impact on innovation performance. Productivity is considered to be the most reliable indicator for evaluating the economic performance of a firm. Crucini, Kose, and Otrok, (2011) suggested that total factor productivity shocks have been a primordial source of fluctuations in global economic activity. Empirical findings suggest that the relationship between firm productivity and innovation activities is positive. Doraszelski and Jaumandreu (2007) found that R&D spending is highly positively associated with the probability of introducing a new product and process innovations, investments which in turn increase firms' productivity. The same authors highlight that innovative firms have higher labor productivity and are bigger than firms that do not innovate. In terms of type of innovation, Parisi, Schiantarelli, and Sembenelli (2006) analyzed Italian firms and found that the introduction of process innovation has a sizeable effect on productivity. Cassiman and Martinez-Ros (2007) suggest that product innovation rather than process innovation affects firm productivity. Moreover, Antonioli, Mazzanti, and Pini (2011) find a positive impact of organizational and technological innovations on labour productivity. Hence, the following hypothesis is proposed:

H4. There is a positive relationship between firm productivity and innovative performance.

Recent empirical evidence generally shows a positive relation between firm size and the likelihood of innovation (Alarcón et al., 2013), but some studies show a non-significant (Lööf, Heshmati, Asplund, & Nåås, 2001) or even a negative relationship between firm size and probability of innovation (Pavitt, Robson, & Townsend, 1987). Bayona et al., (2013) detected a positive relation between larger firms and innovation because of improved access to human and financial resources and profit persistence. Damanpour (2010), on his part, suggests that size has a more positive association with process than with product innovations. The recent downturn will negatively impact not only investment and production but it has also revealed employment problems related to higher unemployment rates (Ashford, Hall, & Ashford, 2012). Spain is one of the countries that witnessed the most marked expansion with a sharp fall in employment (Ortega & Peñalosa, 2012). Therefore we hypothesize that:

H5.a. Size has a positive impact on the innovative performance of firms.

H5.b. This positive effect is expected to be lower during economic crisis.

2.2.3. Impact of an economic crisis on innovation performance

Many studies show the various changes which occurred when the global crisis hit. Some of the effects of the current economic crisis on consumers are employment uncertainty and a growing unemployment rate and an income fall, all of which in turn affect customer purchase behaviour, mostly negatively (Dave & Kelly, 2012; Mansoor & Jalal, 2011). Consumers tend to be more careful, planning their expenditure and focusing on spending efficiency, reducing consumption level in different ways according to each product category (Mansoor & Jalal, 2011). Dave and Kelly (2012) note a link between low-income households and unhealthy food consumption; they found a countercyclical effect for unhealthy foods and significant procyclical effects for healthy food. That is, lower incomes caused by an increasing unemployment rate and/or reduction in working hours during a period of recession tend to raise the substitution of healthy food (e.g. fruits and vegetables) for unhealthy food consumption (e.g. snacks, cheap fast food or limited service restaurants) in both old and young adults. Chang, Gunnell, Sterne, Lu, and Cheng (2009) also note that young and middle-aged adults are more affected by a change in economic circumstances (such as an increase in unemployment and lower income) than older people.

Another study by Ásgeirsdóttir et al., (2012) analyzed the effects of a macroeconomic downturn in Ireland on a range of health behaviors. Based on a longitudinal health and lifestyle survey from 2007 to 2009, they concluded that the crisis in Iceland resulted in the adoption of less healthy lifestyles such as a reduction in the consumption of fruits, vegetables, vitamins and supplements and an increased use of fish oil, food with little nutritional value and smoking as a response to stress. Furthermore, the same authors confirmed that the effect of a crisis was greater on the working-age population in relation to the adult population. Blanchard (1993) found that the 1990-1991 recessions in the USA was largely the result of a "consumption shock" This fact suggests that changes in consumption can predict changes in output. Consumer confidence was much weaker than that which could be accounted for by its usual correlation with an exogenous shock to the economy, including future income, unemployment rate, and inflation.

Under these conditions, innovative businesses suffered the lower demand for their products and hence foresaw substantial uncertainties over future trends in consumption (OECD, 2012). Filippetti and Archibugi (2011) suggest that the drop in demand played a substantial role in firms' decisions regarding innovation investments. Moreover, not all sectors and categories of products have been affected in the same way by these environment changes. For example, due to the importance of the food sector as a necessary element to human survival, the impact of the recent crisis has been lower than in any other sector of the economy in Spain (Baamonde, 2009). Food will continue to represent a significant percentage of consumer expenditure in Spain (AAFC, 2012). Katchova and Enlow (2013) analyze the financial performance of publicly traded agribusinesses when compared to all firms over the 1961-2011 period. They show that agribusinesses had a strong financial performance and outperform the sample of all firms based on a series of financial ratios. These findings are important for investors considering adding agribusinesses to their investment portfolios particularly during the recent economic recession. Schiefer, Hirsch, Hartmann, & Gschwandtner (2013) focused on the EU food sector and also found evidence of weak economic fluctuations which explained the difference in firm performance when compared to firm-specification characteristics. In line with this, we put forward following hypotheses:

H6.a. The economic crisis had a negative impact on firms' innovative performance

H6.b. It is to be expected that the effect of the economic crisis will be lower in the agri-food sector than in any other sector of the economy.

Following the extant literature, a theoretical model of the case study was developed. In this framework we have studied the factors selected for our model of analysis and the hypothesized relationships between them in depth (Figure 2.1).

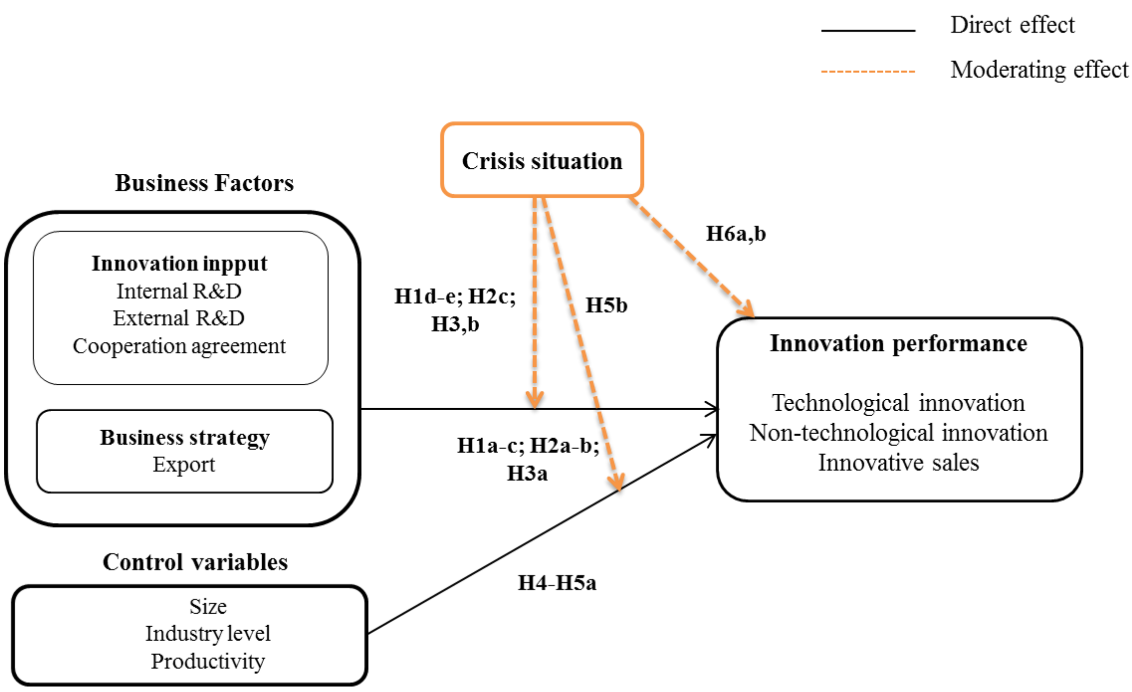

Figure 2. 1. Theoretical model and hypotheses

2.3. Models used

The econometric models used are random effects logit model and random-effects Tobit models[2]. Those frameworks take into account the existence of multiple observations of each

[2] We are considering a sample of the whole population of Spanish firms; the random effects model would be more appropriate for a large population (Henderson & Ullah, 2005).

firm in different periods of time and compute a different intercept for each of the observations in each period of time (Un, Cuervo-Cazurra, & Asakawa, 2010).

The logit with panel-level random effects for firm *i* in period *t* can equivalently be written as:

$$Y_{it}^{*} = \alpha + \beta\, X_{it-1} + \mu_i + \varepsilon_{it} \qquad (\mathrm{i} = 1; \dots; \mathrm{n}\,;\, \mathrm{t} = 1; \dots; \mathrm{T})$$

Where $Y_{it} = 1$ if $Y^{*}_{it} > 0$

0 0therwise

Y^{*}_{it} denote the unobservable propensity to innovate, α is the constant term, β is a vector of parameters and X_{it-1} is the vector of explanatory variables[3]. The random effect model decomposed the error term into two components ($u_i + \varepsilon_{it}$) in order to take account of unobserved heterogeneity; one of which is specific to each firm's i (u_i), and a component ε_{it} stands for other unobserved variables (random error). The random-effects Tobit model is obtained such that:

$$Y_{it}^{*} = X_{it-1}\beta + \alpha_i + \varepsilon_{it} \qquad (\mathrm{i} = 1; \dots; \mathrm{n}\,;\, \mathrm{t} = 1; \dots; \mathrm{T})$$

While $y_{it} = y^{*}_{it}$ if $y^{*}_{it} > 0$

$y_{it} = 0$ if $y^{*}_{it} \leq 0$

We make the usual random effects assumption that α_i and ε_{it} are independent and identically distributed of $x_{i1},\dots,x_{iT}$, with zero means and variances σ^2_α and σ^2_ε, respectively.

In order to test our hypothesis (H2-6) cited above about the effect of a crisis on firm performance, a set of interaction terms between each explanatory variable and the time dummy (D_2010-2012) is included in both the Logit and Tobit models.

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[3] We lagged all independent and control variables (except sector dummies which do not vary across panel waves) by one period with respect to innovation output variables. This approach allows us to minimize endogeneity and to justify the inclusion of this variable as an ex-ante explaining variable (Bradley, Wiklund, & Shepherd, 2011).

2.4. Data set Description

This section illustrates the dataset analysis and variables description. The database used for our empirical analysis has been taken from the Spanish Technological Innovation Panel (PITEC)[4], which is carried out on a yearly basis by the Spanish National Statistics Institute (INE) in collaboration with the Spanish Science and Technology Foundation (FECYT) and the Foundation for Technological Innovation (COTEC). The data are collected annually, gathering data since 2003, the latest available year at the present time being 2012.

For the purposes of this present paper, we used information from PITEC for the 2008–2012 period[5] and we studied all the Spanish sectors available in PITEC. Then, we organized them separately under three principal sectors: agriculture, cattle, forestry, fishing–(NACE-2009 code 0000-), food, beverages, and tobacco (-NACE-2009 code 0003-) and the rest of Spanish firms. According to OECD (2005), the concept of innovation performance encloses multidimensional measures in terms of technological innovation, non-technological innovation and the percentages of sales generated by new products. In this study we use categorical and numerical indicators of innovative performance. The first categorical indicator output is measured by dichotomous variables, which indicate whether or not the firm introduced an innovation during the last 2 years (from t-2 to t). We distinguish between four types of innovation described in the Oslo Manual (OCDE, 2005): product, process, organizational and marketing innovations. The second output is the quantitative indicators of innovation performance based on the share of turnover derived from new or improved products during the last 2 years (from t-2 to t). These variables can be used to provide important information on the impact of product innovation on turnover and on the degree of innovativeness of the firm.

As explanatory variables, we introduce binary variables indicating whether the firm undertakes R&D development activities and cooperation agreements, and if firm operates in international markets for developing innovation. Furthermore, we include a set of control

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[4] The Database is located free on the FECYT site: http://icono.fecyt.es/PITEC .

[5] Due to the particularities of this survey, some of the output variables of interest such as organizational and marketing innovations are available only for years 2004 and 2005 and then disappear again until 2008.

variables related to firms' characteristics: Firm size and firm productivity. In addition, as the innovation behavior of firms depends on the sector in which it operates, we also controlled a firm's sector on the two digit NACE codes by using dummy variables coded '1' if the firm belongs to the respective two-digit sector, and '0' otherwise. We created dummies for the agricultural and food sectors. The rest of the sector was used as the control group.

Finally, a time dummy D_2010-2012 which corresponds to years 2010 and 2011 was added to the econometric model in order to control for the long-term effect of the crisis on the innovation performance of firms. The baseline will be years 2008 and 2009[6]. According to Ghemawat (1993), during general business downturns, this investment has tended to decline two to four times faster than output. Based on this work, we assume that the effect of a crisis on firm performance is seen not at the beginning of the crisis but later on, and thus we consider two periods: (a) 2008-2009: the "beginning of the crisis", (2) 2010-2012: "during the crisis". Table 2.1 lists the description of all the variables used in detail.

Table 2. 1. Description of the variables

Variables	Definitions	Mean	Std.Dev.
Dependent Variables			
INN_Product	1 if the firm introduced product innovation, 0 otherwise	0.561	0.496
INN_Process	1 if the firm introduced process innovation, 0 otherwise	0.590	0.492
INN_Organizational	1 if the firm introduced organizational innovation, 0 otherwise	0.457	0.498
INN_Marketing	1 if the firm introduced marketing innovation, 0 otherwise	0.300	0.458
INN_Radical	The percentage of the firm's sales from products new to the market	9.892	22.828
INN_Incremental	The percentage of the firm's sales from products new to the firm	46.187	45.992

[6] The 2008-2012 period was characterized by a significant decrease in both demand for innovative products and in the share of firms achieving innovations in all Spanish sectors. We noted that the effect of the crisis began to show a negative impact on almost all innovation inputs and outputs from the year 2010 onwards. The number of companies carrying out exporting operations has increased significantly (approximately 5.4% for the food industry and 4.9 % for the total sector).

Independent Variables			
Innovation sources			
InternalR&D_continuous*	1 if the firm engaged in-house R&D activities continuously	0.422	0.494
Internal R&D_occasional*	1 if the firm engaged in-house R&D activities occasionally	0.104	0.306
External R&D_Nat	1 if the firm engaged in national external R&D activities	0.209	0.406
External R&D_Inter	1 if the firm engaged in international external R&D activities	0.012	0.111
Cooperation partners			
COOP_Ind_NAT	1 if the firm cooperated in innovation with national industrial agents (customers, suppliers, competitors and firms belonging to the same business group), 0 otherwise	0.212	0.409
COOP_Ind_INTER	1 if the firm cooperated in innovation with international industrial agents (universities, public research organizations, technologic centers and commercial laboratories/R&D enterprises), 0 otherwise	0.227	0.419
COOP_Instit_NAT	1 if the firm cooperated in innovation with national institutional agents, 0 otherwise	0.100	0.300
COOP_Instit_INTER	1 if the firm cooperated in innovation with international institutional agents, 0 otherwise	0.051	0.220
EXPORT	1 if firms that operate outside Spain, 0 otherwise		
Firm variables			
SIZE	Ln (total number of employees)	4.047	1.696
Productivity per employee	Ln (ratio of firm sales to the total firm employees)	11.772	1.054
Sectoral dummies			
Food_SEC**	1 if the firm belongs to food, beverages sector, 0 otherwise	0.073	0.258
Agri_SEC**	1 if the firm belongs to agricultural sector, 0 otherwise	0.013	0.113
Dummy time			
D_2010-2012	Time dummy, 1 if the observation corresponds to the period 2010-2012, 0 if the period is 2008-2009.	0.579	0.494

*The firm not engaged in in-house R&D activities was used as reference category; ** The rest of the sector was used as the baseline category

Figure 2.2 shows changes in macroeconomic indicators (GDP rate growth per capita, unemployment rate) in Spain as a response to the crisis. As Figure 2.2 shows, the greatest impact of the economic crisis in Spain was suffered from 2009 on. Like many developed countries affected by a crisis (Peters et al., 2009 on the USA), the global crisis had a prompt and significant impact on Spain; the unemployment rate went from 8.5 percent in 2006 to

26.1 percent in 2013. Spain's gross domestic product also saw a negative growth rate from 2009 onwards. GDP fell 6.8 percent in 2009 vs. 2.9 percent in the previous year.

The crisis also had a negative impact on household consumption patterns in Spain–food, restaurant and hotels and housing, each accounting for around 17%, 16% and 22% of consumption expenditure respectively–had the largest weightings (Eurostat, 2015). Trends in consumption in Spain during the crisis decreased by 2 percent in non-food items, the sectors more affected being clothing, household equipment, transport and recreation/culture. Food, alcohol and tobacco consumption remained stable from 2006 to 2011, growing by nearly 0.5% percent in 2013. This provides some initial evidence to the fact that crises have a lesser effect on this sector as compared to the whole sector.

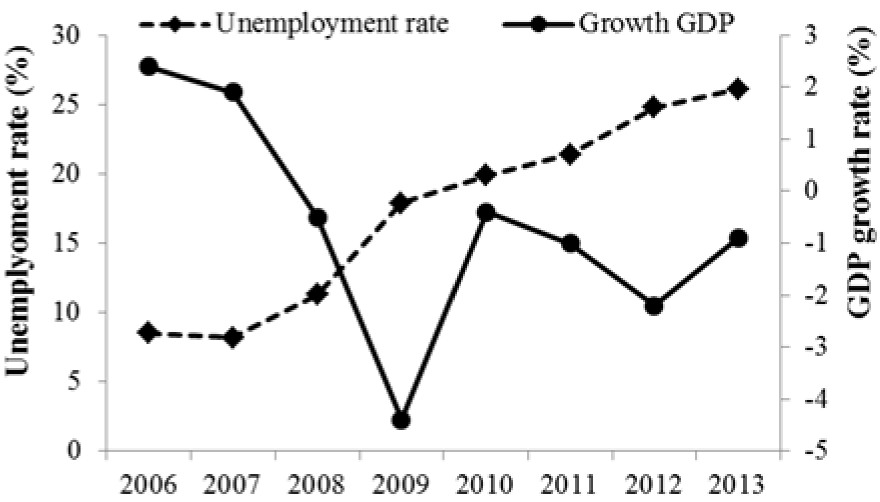

Figure 2. 2. Trends in gross domestic product (GDP) growth rate per capita and unemployment rate as the percent of total labor force in Spain during 2008-2012 (Source: Eurostat and the Word bank)

2.4.1. Main outcomes of the dataset

Figure 2.3 shows the growth rate of sales and employment in different types of firms (innovative versus non-innovative firms). We define firms which implemented an innovation during the period under review (OCDE, 2005) as innovating firms. We can see that the effects of the financial crisis differed considerably across sectors. The agriculture and food sectors are less affected in terms of sales and employment. The unemployment rate increased and reached 7.7%, 4.5% and 3.9% in 2012, while sales dropped by -7.6%, -0.6% and -3% for all firms, agriculture and food industry, respectively.

The difference across innovative and non-innovative firms shows that innovating firms maintained employment and sales rates better than their non-innovating counterparts. It is interesting to note that both the food and agriculture industries were able to derive better shares of sales from innovation than the total Spanish sector; innovating firms show a significantly positive sales growth in agriculture sector while non-innovative ones have a negative sales growth, which confirms the importance of innovation in this sector.

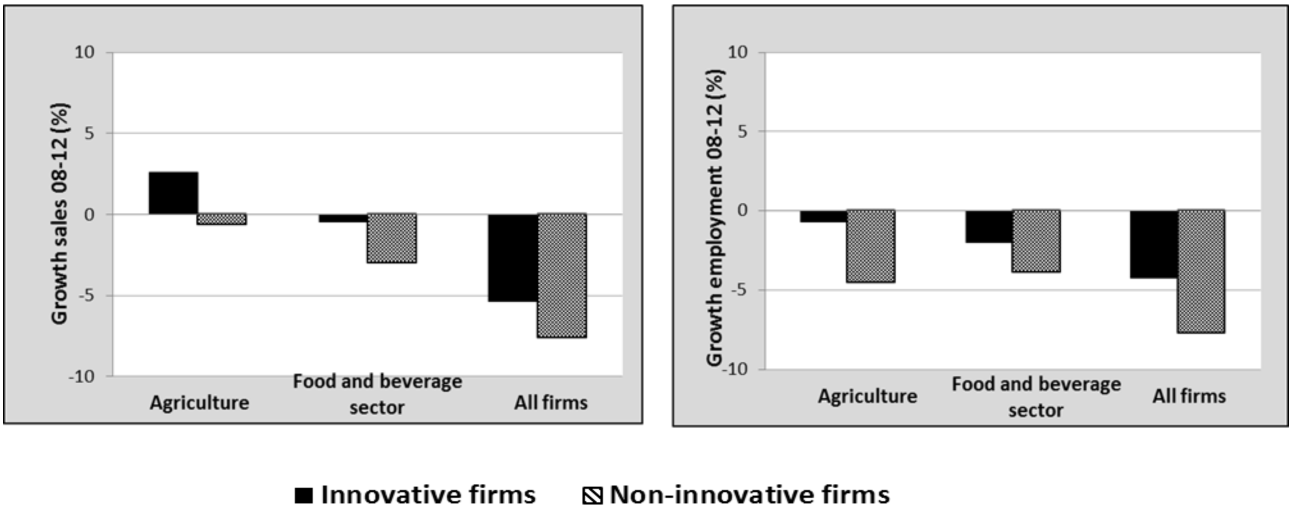

Figure 2. 3. Sales and employment growth rates for innovative and non-innovative firms over the 2008-2012 period.

2.4.2. Measurement model test and discussions

The results of random-effects Logit model and Random-Tobit estimations[7] are reported in Table 2.2 and Table 2.3, respectively. In order to test our proposed hypotheses, we estimated various models. In the Table 2.2, models (1), (3), (5) and (7) are the base models present the estimation's results for each innovation output (product, process, marketing and organizational innovation) and models (2), (4), (6) and (8) introduce the interactions between

[7] The models were tested for multicollinearity and the correlation values among all variables are quite low; a maximum of 0.483 was obtained. This value is below 0.56, the maximum value recommended for the multicollinearity test. Therefore, we calculated variance inflation factors (VIFs) for each correlation and obtained a maximum of 1.69. This level is well below the rule of thumb cut-off of 10 (Neter, Kutner, Nachtsheim, & Wasserman, 1996), which indicates that multicollinearity does not pose a problem to our estimation models.

each explanatory variable and the time dummy (D_2010-2012). In Table 2.3, models (1) and (3) show the relationship between explanatory variables and innovative product sales. Interactions between the each explanatory variable with the time dummy (D_2010-2012) are included in models (2) and (4).

Table 2. 2. Factors influencing the decision to innovate: Random-effects logit model estimation

	Technological innovations				Non-technological innovations			
	Product innovation		Process innovation		Organizational innovation		Marketing innovation	
	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
Continuous_Internal R&D$_{t-1}$	2.598***	2.578***	1.290***	1.266***	1.241***	1.229***	1.377***	1.363***
	(0.057)	(0.057)	(0.052)	(0.052)	(0.053)	(0.053)	(0.059)	(0.059)
Occasional_Internal R&D$_{t-1}$	1.739***	1.733***	1.194***	1.174***	0.831***	0.810***	0.874***	0.856***
	(0.065)	(0.065)	(0.063)	(0.064)	(0.063)	(0.064)	(0.070)	(0.070)
External R&D_Nat$_{t-1}$	0.252***	0.250***	0.361***	0.352***	0.264***	0.259***	0.198***	0.198***
	(0.054)	(0.054)	(0.051)	(0.051)	(0.050)	(0.050)	(0.052)	(0.053)
External R&D_Inter$_{t-1}$	0.819***	0.829***	0.857***	0.864***	0.675***	0.674***	0.282	0.288
	(0.202)	(0.203)	(0.193)	(0.193)	(0.184)	(0.184)	(0.189)	(0.190)
COOP_Ind_NAT$_{t-1}$	0.541***	0.549***	0.568***	0.577***	0.438***	0.443***	0.262***	0.259***
	(0.062)	(0.062)	(0.059)	(0.059)	(0.058)	(0.058)	(0.062)	(0.062)
COOP_Instit_NAT$_{t-1}$	0.511***	0.495***	0.342***	0.328***	0.230***	0.223***	0.135**	0.138**
	(0.064)	(0.064)	(0.060)	(0.061)	(0.060)	(0.060)	(0.064)	(0.064)
COOP_Ind_INTER$_{t-1}$	0.426***	0.424***	0.288***	0.294***	0.297***	0.294***	0.186**	0.183**
	(0.092)	(0.093)	(0.086)	(0.087)	(0.082)	(0.082)	(0.083)	(0.083)
COOP_Instit_INTER$_{t-1}$	0.124	0.120	0.113	0.149	0.315**	0.317**	0.209*	0.199*
	(0.126)	(0.126)	(0.118)	(0.120)	(0.113)	(0.113)	(0.112)	(0.112)
SIZE$_{t-1}$	0.074***	0.067***	0.461***	0.458***	0.441***	0.445***	0.132***	0.132***
	(0.018)	(0.018)	(0.018)	(0.018)	(0.019)	(0.019)	(0.021)	(0.021)
Export$_{t-1}$	0.463***	0.477***	-0.063	-0.047	0.057	0.067	0.273***	0.277***
	(0.049)	(0.050)	(0.046)	(0.046)	(0.048)	(0.048)	(0.052)	(0.052)
Productivity$_{t-1}$	0.139***	0.140***	0.156***	0.158***	0.065**	0.067**	0.077**	0.079**
	(0.026)	(0.026)	(0.025)	(0.025)	(0.026)	(0.026)	(0.030)	(0.030)
FOOD_SEC	-0.235*	-0.239*	0.664***	0.676***	0.019	0.013	1.010***	1.007***
	(0.123)	(0.124)	(0.116)	(0.117)	(0.124)	(0.124)	(0.137)	(0.137)
AGRI_SEC	-1.067***	-1.0715***	0.484*	0.508**	-0.821**	-0.829**	-0.733**	-0.769**
	(0.265)	(0.267)	(0.254)	(0.257)	(0.283)	(0.285)	(0.325)	(0.328)
D_2010-2012	-0.753***	-0.789***	-0.721***	-0.737***	-0.355***	-0.369***	-0.038	-0.061
	(0.032)	(0.036)	(0.030)	(0.033)	(0.030)	(0.033)	(0.032)	(0.037)
Interactions terms								

D_2010-2012*Food_SEC		0.052		-0.344***		-0.218*		-0.147
		(0.127)		(0.121)		(0.115)		(0.119)
D_2010-2012*Agri_SEC		-0.374		-0.282		-0.115		-0.548
		(0.273)		(0.265)		(0.282)		(0.313)
D_2010-2012*Continuous_Internal R&D$_{t-1}$		0.297***		0.231***		-0.029		-0.042
		(0.079)		(0.074)		(0.075)		(0.088)
D_2010-2012*Occasional_Internal R&D$_{t-1}$		0.471***		0.217**		-0.182*		-0.082
		(0.108)		(0.106)		(0.107)		(0.117)
D_2010-2012*External R&D_NAT$_{t-1}$		-0.027		-0.004		0.004		-0.042
		(0.093)		(0.087)		(0.084)		(0.088)
D_2010-2012*External R&D_INTER$_{t-1}$		0.682**		0.234		0.274		0.152
		(0.328)		(0.319)		(0.295)		(0.302)
D_2010-2012*COOP_Ind_NAT$_{t-1}$		-0.223**		-0.065		-0.087		-0.253**
		(0.108)		(0.102)		(0.098)		(0.102)
D_2010-2012*COOP_Instit_NAT$_{t-1}$		0.108		-0.075		-0.149		0.209**
		(0.105)		(0.099)		(0.097)		(0.102)
D_2010-2012*COOP_Ind_INTER$_{t-1}$		-0.071		-0.034		0.068		0.209
		(0.159)		(0.148)		(0.136)		(0.135)
D_2010-2012*COOP_Instit_INTER$_{t-1}$		0.203		-0.394*		0.079		0.063
		(0.214)		(0.206)		(0.188)		(0.180)
D_2010-2012*SIZE$_{t-1}$		0.196***		0.206***		0.077***		-0.071***
		(0.020)		(0.019)		(0.019)		(0.021)
D_2010-2012*Export$_{t-1}$		-0.202**		-0.173**		0.081		-0.052
		(0.072)		(0.066)		(0.067)		(0.072)
D_2010-2012*Productivity$_{t-1}$		-0.074**		-0.043		0.036		-0.003
		(0.033)		(0.030)		(0.031)		(0.034)
Constant	-2.822***	0.558***	-3.652***	0.800***	-3.659***	-0.378***	-4.408***	-1.948***
	(0.317)	(0.035)	(0.301)	(0.033)	(0.323)	(0.036)	(0.364)	(0.044)
Wald χ2	3911.88***	3970.92***	2722.93***	2793.69***	1917.01***	1936.00***	1062.25***	1085.08***
AIC	39404.67	39299.65	43837.63	43720.42	42809.55	42797.39	37802.63	37799.28
BIC	39543.31	39550.95	43976.27	43971.71	42978.2	43048.69	37941.27	38050.58

Standard errors are reported in parentheses. * $p < 0.1$; ** $p < 0.05$; *** $p < 0.01$.

Table 2. 3. Estimation results for innovation output: Sales of new products

	Sales due to Incremental Innovations		Sales due to Radical Innovations	
	(1)	(2)	(3)	(4)
Continuous_Internal R&D$_{t-1}$	3.626***	3.568***	2.227***	2.193***
	(0.088)	(0.088)	(0.061)	(0.061)
Occasional_Internal R&D$_{t-1}$	2.933***	2.876***	1.600***	1.575***
	(0.103)	(0.103)	(0.071)	(0.072)
External R&D_NAT$_{t-1}$	0.159**	0.164**	0.232***	0.231***
	(0.075)	(0.075)	(0.049)	(0.049)
External R&D_INTER$_{t-1}$	0.716***	0.706***	0.449***	0.452***
	(0.261)	(0.260)	(0.170)	(0.169)
COOP_Ind_NAT$_{t-1}$	0.620***	0.607***	0.256***	0.251***
	(0.089)	(0.088)	(0.058)	(0.058)
COOP_Instit_NAT$_{t-1}$	0.378***	0.359***	0.564***	0.556***
	(0.091)	(0.096)	(0.060)	(0.060)
COOP_Ind_INTER$_{t-1}$	0.232*	0.218*	0.246***	0.236***
	(0.119)	(0.118)	(0.076)	(0.076)
COOP_Instit_INTER$_{t-1}$	0.062	0.046	0.351***	0.338***
	(0.157)	(0.157)	(0.099)	(0.099)
SIZE $_{t-1}$	0.187***	0.173***	-0.057**	-0.060**
	(0.032)	(0.036)	(0.022)	(0.022)
Export $_{t-1}$	0.546***	0.549***	0.340***	0.347***
	(0.076)	(0.076)	(0.051)	(0.051)
Productivity $_{t-1}$	0.296***	0.296***	-0.003	-0.002
	(0.044)	(0.044)	(0.030)	(0.030)
FOOD_SEC	-0.307	-0.320	-0.392***	-0.384**
	(0.212)	(0.213)	(0.149)	(0.149)
AGRI_SEC	-1.438***	-1.437***	-0.642**	-0.700**
	(0.466)	(0.468)	(0.325)	(0.327)
D_2010-2012	-0.848***	-0.975***	-0.544***	-0.670***
	(0.046)	(0.052)	(0.031)	(0.037)
Interactions terms				
D_2010-2012*Food_SEC		-0.241		0.256**
		(0.180)		(0.123)
D_2010-2012*Agri_SEC		-0.478		-0.671**
		(0.429)		(0.294)
D_2010-2012*Continuous_Internal R&D$_{t-1}$		1.111***		0.528***
		(0.118)		(0.083)
D_2010-2012*Occasional_Internal R&D$_{t-1}$		1.093***		0.531***
		(0.169)		(0.118)
D_2010-2012*External R&D_NAT$_{t-1}$		0.043		-0.009
		(0.123)		(0.079)
D_2010-2012*External R&D_INTER$_{t-1}$		1.093***		-0.122
		(0.414)		(0.269)
D_2010-2012*COOP_Ind_NAT$_{t-1}$		-0.221		0.058
		(0.143)		(0.093)
D_2010-2012*COOP_Instit_NAT$_{t-1}$		0.336**		0.042
		(0.142)		(0.092)
D_2010-2012*COOP_Ind_INTER$_{t-1}$		-0.055		-0.020
		(0.185)		(0.117)
D_2010-2012*COOP_Instit_INTER$_{t-1}$		0.045		-0.025
		(0.240)		(0.150)
D_2010-2012*SIZE$_{t-1}$		0.228***		0.152***

		(0.029)		(0.020)
D_2010-2012*Export$_{t-1}$		-0.051		-0.127*
		(0.102)		(0.068)
D_2010-2012*Productivity$_{t-1}$		-0.115**		-0.031
		(0.050)		(0.034)
Constant	-5.376***	0.796***	-2.874***	-1.853***
	(0.534)	(0.062)	(0.369)	(0.050)
Wald χ2	3102.08***	3240.00***	2507.55***	2585.90***
AIC	114257.5	114073.9	88320.56	88217.13
BIC	114404.8	114333.9	88467.87	88477.09

Standard errors are reported in parentheses. * p < 0.1;** p < 0.05;*** p < 0.01

As predicted in H1.a, in-house R&D on both continuous and occasional basis were found positive and significant in all models showed in table 2.2 (models 1, 3, 5 and 7) and in table 2.3 (models 1 and 3). The results indicate that firms that carry out internal R&D have a better innovative performance in relation to firms that do not.

Our results also support H1.b and H1.c, the estimations display that the acquisition of both national and international extramural R&D has a positive impact on a firm's decision to engage in innovation (models 1, 3, 5 and 7, table 2.2) and on innovative product sales (models 1 and 3, table 2.3). The effects of international extramural R&D exceed the impact of national extramural R&D on all innovation output measures (Tables 2.2 and 2.3). This implies that the internalization of R&D activities can be beneficial for companies to achieve more innovation. This is in line with most other studies (Peters & Schmiele, 2010), which tend to find that firms that have international R&D activities are more likely to launch new products than firms with home-based R&D only. However, the coefficients of interaction term between dummy time (D_2010-2012) and internal R&D (D_2010-2012*continuous_Internal R&D and D_2010-2012*occasional_Internal R&D) are positive and statistically significant in model 2-4 (Tables 2.2 and 2.3), suggesting that internal R&D not only has a positive impact on firm's innovation performance as revealed by H.1.a, but it also keeps playing an important role during crisis as determinants of product (β=0.297 and β=0.471; $p<0.01$) and process (β=0.231 and β=0.217; $p<0.01$) innovations and innovative sales performance (β=1.111 and β=1.093; $p<0.01$ in radical innovation; β=0.528 and β=0.531; $p<0.01$ in incremental innovation). However, in model 6-8 (Table 2.2), continuous in-house R&D drop its significance as a determinants of non-technological innovations which the interaction term between dummy time (D_2010-2012) and internal R&D is negative and non-significant. Whereas, the interaction between occasional in-house R&D and crisis variable (D_2010-2012) is negatively signed and significant in model 6 (Table 2.2), the results can be explained by a decrease in the number of firms carrying out R&D investment in innovation in times of crisis.-Therefore, H.1.d is partly confirmed.

Likewise, the positive and significant interaction terms (D_2010-2012*External R&D_INTER) observed in Table 2.2 (β=0.682, p<0.01; model 2) and in Table 2.3 (β=1.093, p<0.01; model 2) showed the importance of internalization of R&D activities on firm's decision to engage in product innovation and to increase the percentage of innovative sales due to the variety of knowledge shared abroad, particularly in times of crisis. However, domestic R&D activities lose significance as a determining factor on the commercial success of product innovation. Our H1.e is partially supported.

Regarding cooperation agreements, the effect of the different types of partner in cooperation on a firm's innovation performance varies and mainly depends on the type of innovation, as well as on the degree of novelty of the innovations. Cooperation agreements with national partners show positive and significant effects on firm's decisions to innovate and on firm innovativeness, both incremental and radical, thus supporting H2.a. For international partners, cooperation with industrial agents had a positive impact on achieving all innovations types and innovative product sales, collaboration with international institutional partners shows a positive and significant effect only for non-technological innovations and radical innovation. We can see that the effect of national cooperation is stronger than international cooperation on the achievement of all kind of innovations, which contradicts H2.b. During crisis, the significant and negative coefficients of the interactive terms of (D_2010-2012*COOP_Ind_NAT) shown in models 2 and 8 (Table 2.2), implying a decrease in the effect exercised by cooperation on achieving both product and marketing innovations during a crisis. However, contrary results showed when innovative sales is concerned, models (2) indicate that the interactive terms (D_2010-2012*COOP_Instit_NAT) have a positive and significant sign, illustrating that cooperation can help firms to improves their innovative sales during crisis, although, the other types of cooperation loses its significance, this effect may be related to the decrease of internal R&D efforts made by firms during a crisis seem to reduce the exploitation of external knowledge sources derived from innovation cooperation agreements to increase innovative sales. These results contradict H2.c. The export variable has positive impact for product and marketing innovations (models 1 and 7; Table 2.2), and it has the expected positive sign in Table 2.3 (models 1 and 3). Thus, H3.a is supported. Turning to the interaction terms, the results do not support H3.b, the coefficient of interaction term between the dummy time (D_2010-2012) and export variable is negative and significant in both product and process innovation (β=-0.202 and β=-0.173; p<0.05, Table 2.2), similar results revealed in Table 2.3 with radical innovation (β=-0.127; p<0.1). The negative export-innovation link displayed in crisis period maybe associated to decline of internal R&D efforts made by firms in such period. Prior studies argues that greater R&D investment in time of crisis enlarge a firm' flexibility and enhance its export intensity (Lee, Beamish, Lee, & Park, 2009).

As far as the control variables are concerned, our results indicate a positive relationship between firm's productivity and all innovation outputs (models 1, 3, 5 and 7; Table 2.2). In Table 2.3, a positive relationship between a firm's productivity and sales due to incremental innovation (β=0.296; p<0.001) is well showed, giving support to H4. In Models 2 (Table 2.2) as well as in model 2 (Table 2.3), the significant and negative interaction (D_2010-2012*Productivity) showed a negative relation between firm productivity and firm innovative performance. Two possible justifications for this latter result are that a decrease in R&D spending and innovation investment by firms during a crisis adversely affects firms' productivity; the literature argues that investing in innovation and more specifically in internal R&D activities increases firms' productivity (Cassiman & Martinez-Ros, 2007; Doraszelski & Jaumandreu, 2007; Parisi et al., 2006). Another possible justification would be that in a recession period many firms opt for cutting costs through manpower adjustments and freezing pay rates, increasing job insecurity and consequently decreasing productivity (Pappas, 2014) .

Regarding firm size, size has a positive impact on the decision of firms to innovate (Table 2.2) and on sales of products new to firms (Model 1, table 2.3) whereas its effect is significantly negative on sales of products new to the market (Model 3, table 2.3). Our H5.a is partially supported. Testing the H5.b, the positive and significant coefficients of (D_2010-2012*SIZE) in Table 2.2 (models 2, 4 and 6) and in Table 2.3 (models 2 and 4) contributes to a better understanding the important role of the human capital during a crisis in the process of innovation as well as in the successful of innovative sales. Thus, H5.b is not supported.

As regards to crisis variable, H6.a proposed that the economic crisis had a negative impact on firms' innovative performance. Our results showed that the effect of crisis is more pronounced for technological innovation than non-technological innovation, firms become less likely to generate product (β=-0.753; p<0.01) and process innovation (β=-0.721; p<0.01) to a great extent and in organizational innovation to a less extent (β=-0.355; p<0.01). These results are expected given the drop in R&D investments in innovation during a crisis as already stated above. Paunov (2012) highlights three principal aspects that drive a business to put a halt to innovation or innovation investments during a crisis: the first one is uncertainty regarding the outcomes of such investments. Second, initial costs of innovation are high and require firms to have important financial resources and these costs may or may not be recovered. Third, a handsome share of the investment is directed at skilled workers and if the innovation project is abandoned or left unfinished workers will be dismissed and knowledge capital will be lost. However, we find a non-significant effect of crisis on marketing innovation. This relates that all industries still innovate in marketing innovation during crisis in order to creating information exchange between producers and consumers and to fulfil the needs and the expectations of customers for the success of new products in the market. Juříková, Jurášková, and Kocourek (2012) found that companies that increased their marketing budgets during a recession gained market share three times as quickly as those that had cut them. Similarly, in Table 2.3; we showed that the economic crisis negatively affects the turnover of innovative sales; this decrease is not surprising and is probably the result of consumers' frugality in times of crisis and the drop of innovative product demand, supporting H6.a.

Concerning the variables related to the sector, as can be noted from Table 2.2, the food industry is significantly more likely to introduce process (β=0.664; p<0.01) than other Spanish sectors, but have a lower probability of achieving product (β=-0.235; p<0.05) when compared to the other Spanish firms. Even though the food industry is oriented to process innovation as revealed by different studies (Batterink et al., 2006), our study has shown that marketing innovation was also considered important in the food industry. The model (7) in Table 2.2 shows that food firms are significantly more likely to introduce marketing innovation (β=1.101; p<0.001) than other Spanish sectors. This has to do with the particularity of this sector, which is focused on market possibilities and the needs of end users. Regarding the agriculture sector, we found that this sector is more focused on process innovation than other types of innovations. The model (3) in Table 2.2 shows that agricultural firms are significantly more likely to introduce process innovation (β=0.603; p<0.01) than other Spanish firms, but have a lower probability of achieving product (β=-1.067; p<0.001), marketing (β=-0.821; p<0.05) and organizational (β=-0.733; p<0.05) innovations when compared to the other Spanish firms (Models 1, 5 and 7; Table 2.2). This result is interesting because it shows that agricultural firms keep engaging specifically in process innovation rather than on diverse types of innovation to reduce exposure to risk and thus to attain higher survival odds. Regarding sales of new products, our findings suggest that agricultural firms are less innovative in terms of both incremental and radical innovations than the rest of Spanish firms, while the food industry shows the same behaviour as the rest of Spanish firms in terms of incremental innovation. These results are in line with those in Garcia Martinez and Briz (2000), who found that the food industry is characterized by incremental rather than radical changes due to demand-side constraints and consumers' conservative behavior.

Finally, the results partially supported the H 6.b, which provide that the economic crisis will be lower in the agri-food sector than in any other sector of the economy in Spain. The interactive term between crisis variable and food sector in table 2.2 (D_2010-2012*Food_SEC) is significant and has negative coefficients (β=-0.344; p<0.05) in models (4) and (β=-0.218; p<0.05) in model (6), implying that this sector decrease their efforts to make process and organizational innovations during crisis period respect to the other sector, but still innovate in product and marketing innovation at the same level (non-significant coefficients). This result is interesting because it shows that food firms keep engaging specifically in product and marketing innovation rather than on others types of innovation to still competitive by differentiated its products and even explore new markets. Besides, the agriculture sector shows the same behaviour as at the beginning of the crisis in all types of innovation in order to get competitive. In Table 2.3, the interaction term between food sector and dummy time (D_2010-2012*Food_SEC) is statistically significant and positive (β= 0.256; p<0.01, model 4), which indicates that the food sector is more likely to increase sales due to radical innovations during the crisis than at the beginning of the 2008-2009 crisis. These results show that the impact of the recent crisis has been lower in this sector. Hence, our H 6.b partially supported. Table 2.4 includes a summary of the final confirmed or rejected status of the different hypotheses proposed in the study.

Table 2. 4. Overview of hypotheses and findings

Hypothesis	Results
Effect of R&D activities	
H1.a. Firms that carry out internal R&D will see a positive impact on firm performance in relation to firms that do not	✓
H1.b. National or international external technology acquisition positively correlates with firms' innovative performance	✓
H1.c. The effect of international R&D can be expected to be stronger than national R&D	✓
H1.d. It is to be expected that the positive effect of internal R&D on firms' innovative performance will be lower during an economic crisis	partially supported
H1.e. It is to be expected that the positive effect of external R&D on firms' innovative performance will be lower in an economic crisis	partially supported
Effect of cooperation	
H2.a. Cooperation agreements with different national partners will have a positive effect on the innovative performance of the firms	✓
H2.b. Cooperation agreements with international partners will have a positive effect on the innovative performance of the firms	✗
H2.c. The positive effect of cooperation agreements on firms' innovative performance will be easier to be perceived in times of crisis	✗
Effect of export	
H3.a. The export variable is positively related to innovative firm performance	✓
H3.b. It is to be expected that his positive effect will be higher in an economic crisis	✗
Effect of productivity	
H4. There is a positive relationship between firm productivity and innovative performance	✓
Effect of firm size	
H5.a. Size has a positive impact on the innovative performance of firms	partially supported
H5.b. This positive effect is expected to be lower in an economic crisis	✗
Effect of crisis	
H6.a. The economic crisis had a negative impact on firms' innovative performance	✓
H6.b. The effect of the economic crisis will be lower in the agri-food sector than in any other sector of the economy in Spain	partially supported

2.5. Conclusions and implications

Companies are affected in many different ways by economic crises. Some have been forced to reduce their investment in R&D and others put a halt to innovation as a result of uncertainty regarding the market success of innovations and the fear of not recovering production costs. In the Spanish case study employed, the findings provide several important implications for theory and practice. First, while innovation as a driver of firm performance has been well established in the literature (Kühne et al., 2010), our paper provides the importance of innovation during recession periods as key mechanism for organizational growth and even survive in tough economic times, especially in the food and agriculture sectors. The results reveal that agri-food firms' profits and growth depend on their ability to innovate.

The food industry tends to engage in product and marketing innovations at the same level rather more frequently than in other types of innovations during a time of crisis and is more likely to increase its sales due to radical innovations than other Spanish sectors. On the other hand, the agricultural sector continues to invest in all type of innovation at the same level in order to stay competitive and to attain long-term viability and even survive in tough economic crisis. Second, increasing innovative performance should be a goal for many firms, especially in difficult time to cope better and hence survive in tough economic times. The current paper has confirmed that engagement in internal R&D activities not only influences the firms' innovative performance, which is quite shown in literature (Bayona et al., 2013; Vega-Jurado et al., 2009) but also has an important role during crisis as determinants of product and process innovations and on the success of the innovations.

Additionally, opening up R&D activities to external knowledge by means of the acquisition of external R&D as well as by cooperation agreements allows firms to have access to more knowledge, which helps their innovation process and improves innovative sales. In order to take advantage of this expansion of knowledge access base through acquisition of external R&D and cooperation, companies have to make more efforts in continuous in-house R&D investment. Senior managers should be encouraged to persist in their investment in in-house R&D activities which do not depend solely on the acquisition of knowledge outside their environment and the exploitation of relevant external knowledge should also be set as a priority (Tsai & Hsieh, 2009).

Third, the results provide evidence that business managers should be aware of the importance of innovation in times of crisis and of the need to invest more in R&D in a continuing rather than occasional fashion, which would lead to better productivity levels and to the international competitiveness of their firms. The relationship between a firm's innovative performance and productivity and export intensity becomes more negative during the crisis period than at the beginning of the crisis as a consequence of the fall in R&D efforts seen in firms over the course of the crisis. As Dabla-Norris, Kersting, & Verdier (2010) pointed out that innovation is crucial to firm performance as it increases productivity in a direct and measurable way.

Fourth, our findings highlight the importance of the human capital in the process of innovation; firm size keeps playing a significant role in explaining innovation outputs during a crisis. This should be taken into account by company managers, who should keep a staff of skilled workers and persist in investment in innovation, which promotes higher levels of employment and job creation.

2.6. Perspectives for future works

This study faced some limitations and these could suggest lines of future studies. Our paper is limited in terms of years due the particularity of the PITEC database cited above, which provides information until 2012 with some output variables of interest available only as from 2008. Therefore, the effect of the crisis is not yet clear enough so as to confirm some of our hypotheses; we need more post-2012 years to prove the whole set of hypotheses. Furthermore, it would be interesting for future study to compare the innovative behaviour of firms pre- and post-crisis[8]. Another limitation of the PITEC database is the lack of both information about agri-food sub-sectors and financial indicators, which can help to capture the effect of crisis in several productive agri-food sectors and also to boost other financial ratios (i.e. total assets of firm, return on assets, return on sales). A promising future study path would be to carry out a comparative study of the innovative behaviour of Spanish firms during an economic crisis in relation to other countries using a similar database, when they

[8] In this regard, Bowden and Zhu (2008) point out the advantages of carrying out the analysis of this sector with long time series. Further to that, the special nature of the agricultural sector cycles should be taken into account in the analysis (Jianfei & Xiaorong, 2012)

are available, for pre- and post-crisis years combined with the use of models which take into account both individual innovation capabilities of firms and their environmental and contextual role (industry, GDP, market power, among others).

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CHAPTER III. Capturing Value from Alliance Portfolio Diversity: The Moderating Effect of R&D Human Capital

(*Third revision in Technovation)*

Chapter III. Capturing Value from Alliance Portfolio Diversity: The Moderating Effect of R&D Human Capital

3.1. Introduction

Today's fast paced business environment and shortening product life cycles require firms to consider externally generated scientific knowledge and technology to augment in-house R&D efforts (Huston and Sakkab, 2006). Open innovation research has underscored the value of external sources of knowledge and the use of cooperation networks to boost firms' innovation performance and meet new business challenges (Chesbrough, 2012, Enkel et al., 2009, Laursen and Salter, 2006). Heterogeneity of external partners enables firms to access diverse markets and technological knowledge (Lin, 2014, Zhou and Li, 2012) and facilitates the process of innovation by allowing firms to make new linkages and associations (Cohen and Levinthal, 1990).

However, too much diversity of external sources could adversely impact innovation performance due to increased organisational and managerial complexity (Duysters and Lokshin, 2011, Bader and Enkel, 2014, Foss et al., 2011). Studies report a curvilinear (inverted U-shaped) relationship between R&D strategic alliances and innovation performance, suggesting that collaborative diversity is beneficial to a specific inflexion point, after which further increasing diversity has a negative effect on innovation performance (Chen et al., 2011, Duysters and Lokshin, 2011, Oerlemans et al., 2013, de Leeuw et al., 2014). Limited research, however, has focused on a systematic investigation of the impact on product innovation performance of external channels of knowledge and technology transfers from business ecosystems. Particularly, the role exerted by internal capabilities to extract value from external collaborations remains largely under-researched (Lazzarotti et al., 2015). Absorptive capacity (Cohen and Levinthal, 1990), defined as the ability of a firm to recognize and utilize new external knowledge, is essential for the effective exploitation of collaborative innovation. A firm's absorptive capacity depends on its existing stock of knowledge, much of which is embedded in its products, processes and people (Escribano et al., 2009). Specifically, we contend that human capital, defined as the knowledge, skills and abilities residing and used by individuals (Subramaniam and Youndt, 2005), enables firms to benefit from a much wider partner diversity.

Responding to call for more research on how to manage business ecosystem (Biemans and Langerak, 2015), this study examines the role of R&D human capital to capture value from diversity in cooperation networks. We draw on the resourced-view (RBV) premise that dynamic capabilities are sources of competitive advantages (Barney, 1991, Barney et al., 2011, Teece et al., 1997) and the theory of human capital (Becker, 1964) to develop a framework that positions R&D human capital as a critical enabler of firms' open innovation strategy. Human capital enables firms to expand their technological boundaries and successfully absorb and deploy new and substantially different knowledge domains (Subramaniam and Youndt, 2005, Faems and Subramanian, 2013). Our hypothesising suggests that R&D human capital can mitigate the diminishing returns in product innovation performance from the integration of high levels of partner diversity.

This paper contributes to the literature in two important ways. First, we contribute to innovation management theory by proposing and testing the moderating role of R&D human capital to identify, assimilate, transform and exploit externally generated knowledge for greater innovation performance. Open innovation research has largely focused on the environmental context of the firm (e.g., type of industry) (Chesbrough and Crowther, 2006) and organisational factors (e.g., structures, systems and procedures) (Petroni et al., 2011, Ritala et al., 2009) while the role of human and social capital in cooperation networks remains largely under-explored. Human capital is a source of competitive advantage that activates firms' capacity to monitor externally generated knowledge and technology and evaluate its relevance (Narula, 2004) for the adoption of productive innovations and new technologies (Nelson and Phelps, 1966).

Second, we demonstrate the contingent nature of human capital in open innovation, indicating when and where R&D education and skills offer the greatest benefit to extract value from partner diversity. The heterogeneity of technological intensity in manufacturing sectors leads to differing knowledge needs and internal capabilities to identify and integrate external knowledge flows into internal innovation processes (Denicolai et al. 2014). Our study demonstrates the need for firms to assess and develop R&D human capital strategies based on the type of innovation activity pursued as its dimensions of education ('general' human capital) and skills ('specific' human capital) (Becker, 1964, Kriechel and Pfann, 2005) impact firms' ability to benefit from open innovation differently. Specifically, our study highlights the importance of R&D skills intensity, particularly in low-tech sectors, compared to R&D education intensity to capture value from more open sourcing strategies.

The paper is structured as follows. Following this introduction, in section two we provide an overview of the relevant literature on APD and R&D human capital and present the research hypotheses. Section three details the research design and methods and section four presents the results. We discuss our findings in section five together with the theoretical and managerial implications of our findings, and a direction for future research and practice in external collaboration.

3.2. Theoretical background and hypotheses development

3.2.1. Alliance portfolio diversity and innovation performance

Increasing global competition, rapid technological advances and shortening product life cycles put firms under unprecedented pressure to introduce new products and services to survive and remain competitive (Teirlinck and Spithoven, 2013, van Beers and Zand, 2014). Breakthrough innovation requires a wider-knowledge base and organisations increasingly rely on external knowledge assets for the successful realisation of their innovative endeavours (Garcia Martinez, 2013, Chiaroni et al., 2010). Sustainable superior innovation performance can be attained by combining diverse market and technological knowledge sources in the alliance portfolio (Lin, 2014) and exploiting possible complementarities and synergies (de Leeuw et al., 2014). External cooperation networks are an ideal platform for learning as external partners bring diverse knowledge and resources that firms can integrate into new products and services (Doz, 1996, Hamel, 1991, Cohen and Levinthal, 1990). In general, the larger and more diverse the business ecosystems, the higher the innovation performance of a firm (Caloghirou et al., 2004, Laursen and Salter, 2006).

However, managing coordinated innovation by network partners requires management attention (Foss et al., 2011). The role of R&D management changes completely and new sets of skills and competencies are required (Witzeman et al., 2006, Mortara and Minshall, 2014). As noted by Christensen (2006, p. 35), ‘Open innovation can be considered an organisational innovation’. It requires firms to implement core processes and develop knowledge capacities (Lichtenthaler and Lichtenthaler, 2009) to apply the open innovation approach effectively (Gassmann and Enkel, 2004). The integration of high levels of partner diversity could lead to high coordination, monitoring and communication costs (Combs and Ketchen, 1999), resulting in an unsuccessful transfer of tacit knowledge by firms to their internal innovation activities (Grimpe & Kaiser 2010), negatively affecting as a result innovation performance (Katila and Ahuja, 2002, Laursen and Salter, 2006).

Thus, we hypothesise a positive but non-linear relationship between APD and product innovation performance. We expect that if the number of external partners exceeds a certain threshold, organizational tension, complexity and coordination begin to hamper a firm's ability to leverage the benefits of external collaboration for innovation. Consequently, innovation search across diverse partners will face diminishing returns.

Hypothesis 1. Alliance portfolio diversity has a positive, curvilinear (inverted U-shaped) impact on product innovation performance.

3.2.2. Intersectoral differences in optimal levels of APD

The present study hypothesises that high-tech and low-tech manufacturing sectors create different contexts for knowledge creation and sharing, hence benefiting from different levels of APD. High-tech sectors are characterised by high levels of technological sophistication and extensive R&D activities (Covin et al., 1990). These industries require a broad range of external partners to remain competitive in their rapidly changing business environments (Ili et al., 2010, Martín de Castro, 2015). In contrast, firms in low-technology sectors require less levels of external search breadth (Laursen & Salter 2006). Innovation in low-tech sectors is driven by customer-related and practical knowledge (Hirsch-Kreinsen, 2008, Von Tunzelmann and Acha, 2005, Heidenreich, 2009). Low-tech innovation is usually not an outcome of the latest scientific or technological knowledge. Empirical studies demonstrate that low-tech industries acquire externally developed mature and well-established technologies, modify these or apply them in a new context (Bender, 2008). Thus, we hypothesise that high-tech industries focus on science-based modes of innovation and engage in more open sourcing strategies whereas low-tech industries target the exploitation of practical and user-driven stocks of external knowledge by collaborating with a smaller number of external partners.

Hypothesis 2. Different levels of APD are beneficial for different levels of technological intensity. For HMHT manufacturing sectors, the optimum will be at a higher level of APD compared to LMLT manufacturing industries.

3.2.3. The moderating effect of R&D human capital

Human capital theory affirms that individual skills, knowledge and capabilities are valuable resources and an important source of economic productivity, and that those skills can be built through education and experience (Becker, 1964). Effectively managing external knowledge flows requires the development of complementary internal capabilities (Teece et al., 1997, Chiaroni et al., 2010). A firm's ability to learn new knowledge through its interaction with external partners requires sufficient technical understanding to capitalize on that knowledge (Huang et al. 2015). This internal capability, referred to as absorptive capacity (Cohen and Levinthal, 1990, Cohen and Levinthal, 1989), reflects a firm's ability to identify, assimilate and exploit external knowledge flows successfully. Thus, firms' presenting high levels of internal R&D capabilities are expected to effectively utilise external knowledge (Arora and Gambardella, 1994, Laursen and Salter, 2004) and engage in more open knowledge search strategies (Cassiman and Veugelers, 2006). Such open sourcing strategies require high levels of human capital (Teixeira and Tavares-Lehmann, 2014, Fukugawa, 2013).

Empirical research highlights the importance of a highly skilled workforce to assimilate and integrate external knowledge into internal innovation processes (Teirlinck and Spithoven, 2013, Huang et al., 2015). Particularly, high task specific (skills) human capital is required to integrate external knowledge with high degree of tacitness associated with highly sophisticated, complex technological processes (Gibbons and Waldman, 2004). Further, Veugelers (1997), Caloghirou et al. (2004) and Spithoven and Teirlinck (2010) argue that highly educated human resources are critical dimensions in the firm's internal bundle of resources and capabilities. However, the complementarity between in-house R&D efforts and external knowledge flows is non-linear (Grimpe and Kaiser, 2010, Berchicci, 2013) and our premise is that R&D human capital can mitigate the diminishing returns in product innovation performance from the integration of high levels of partner diversity.

Hypothesis 3a. Education positively moderates the inverted U-shaped relationship between APD and product innovation performance, such that the curvilinear relationship will be flatter in firms with high R&D education compared to firms with low R&D education.

Hypothesis 3b. Skills positively moderate the inverted U-shaped relationship between APD and product innovation performance, such that the curvilinear relationship will be flatter under in firms with high R&D skills compared to firms with low R&D skills.

Our hypothesised model is depicted in Figure 3.1.

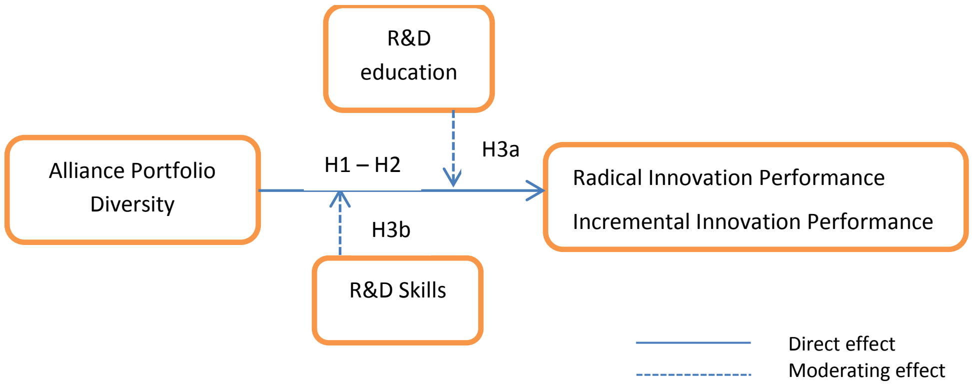


Figure 3. 1. Research framework

3.3. Methodology

3.3.1. Data and sample

The data for the quantitative analysis has been drawn from the Spanish Technological Innovation Panel (PITEC), which is a statistical instrument for studying innovation activities of Spanish companies over time. The database[9] is compiled by the Spanish National Statistics Institute (INE), in collaboration with the Spanish Science and Technology Foundation (FECYT) and the Foundation for Technological Innovation (COTEC). The PITEC dataset contains panel data for more than 12,000 firms since 2003. The study was conducted using information on firms' innovation performance and R&D employment characteristics for the years 2005-2012[10]. For the purposes of this research, the dataset was confined to manufacturing firms that have introduced radical or/and incremental innovations over the studied period. Our final sample contained 32836 observations, 14740 for HMHT industries and 18096 for LMLT industries.

3.3.2. Measures

Dependent variable

Innovation Performance: The literature in organisational innovation distinguishes between incremental and radical innovation (Damanpour 1991; Damanpour et al. 2009). *Radical*

[9] The data base is placed at the disposal of researches on the FECYT site: http://icono.fecyt.es/contenido.asp?dir=05%29Publi/AA%29panel
[10] R&D education and skills data is only available from 2005.

innovation is measured as the percentage of the firm's total sales from innovations new to the market in the last 2 years. *Incremental innovation* is defined as the percentage of the firm's total sales from innovations new to the firm in the last 2 years.

Independent variables

Alliance Portfolio Diversity. To measure APD we consider survey information on cooperation agreements for innovation in the previous three years. Collaborative alliances are distinguished by means of eight partner types: 1) customers, 2) suppliers, 3) competitors, 4) firms belonging to the same enterprise group, 5) universities, 6) public research organizations, 7) technological centres, and 8) commercial laboratories/R&D enterprises. For each type of partners, information is further categorized by their geographical location: Spain, EU and Other Countries. Thus, 24 binary variables are generated, representing all possible combinations between partner type and geographical location. Following de Leeuw et al.'s (2014) approach, APD is calculated by dividing the number of different partner types of a firm by the maximum possible number of partner types (24 in our case) and then squaring the result. The result of this calculation is a diversity score with values between 0 (least diverse – all partners belong to the same category) and 1 (highest diversity- balanced distribution of partners across a larger number of different categories).

R&D human capital intensity: our study uses the traditional measures of human capital: education and skills, employed in empirical research to capture the 'general' and 'specific' dimensions of human capital, respectively (Kriechel and Pfann, 2005). Education intensity is a continuous variable capturing the percentage of R&D staff with third level education or higher (Xia, 2013, Teixeira and Tavares-Lehmann, 2014). Top educated staff increase a firm's capacity to absorb and apply new knowledge into their innovation processes (Rothwell and Dodgson, 1991) and facilitate knowledge sharing within the organisation (Schmidt, 2010). Skills intensity is also a continuous variable accounting for the percentage of top skilled R&D workers (researchers and technicians) (Teixeira and Tavares-Lehmann, 2014). Skilled workers offer greater ability to find, integrate and use new tacit knowledge and later developmental opportunities (Yang et al., 2009).

Control variables

Firm size is measured by (the natural logarithm of) the number of employees (Damanpour, 1996). Further, we account for non-linear effects of firm size by computing a squared term

(SizeSq) (Cassiman & Veugelers 2002). We expect firm size to have a positive effect since larger firms have the necessary internal capabilities to engage in R&D partnerships.

Prior experience: we include a dummy variable to capture a firm's prior experience in external collaboration since experienced firms are more likely to effectively manage their alliance activities than those without (Sampson 2007).

R&D intensity, defined as firm R&D expenditure as a proportion of firm total sales (Laursen and Salter, 2004, Huang et al., 2015), contributes to the internal knowledge base of the firms, so-called absorptive capacity (Cohen and Levinthal, 1990, Zahra and George, 2002), necessary to efficiently absorb and deploy external knowledge (Griffith et al., 2003, Arora and Gambardella, 1990). R&D intensity is expected to complement (rather than substitute) external knowledge search and have a positive impact on innovation outputs (Veugelers, 1997b).

Export intensity is measured by (the natural logarithm of) the ratio of export sales to total sales. Firms competing in international markets are under intense innovation pressure to remain competitive (Kirner et al., 2009). Hence, export intensity might act as an incentive to improve innovation performance through collaborative innovation.

Industry effects. As the innovation behaviour of firms is closely linked to their respective industry sector (Malerba et al., 1997, Audretsch, 1997), we also controlled for the firm's industry affiliation based on the classification proposed by van Beers and Zand (2014). We created two industry dummy variables identifying HMHT and LMLT industries.

Year effects. We use firm-level innovation performance data from 2005 to 2012. Eight year dummy variables are included to control unobservable factors that change over time but remain relatively constant across industries (Lin 2014). Table 3.1 summarises variable names and definitions.

Table 3. 1. Variables' Description

Variables	Type	Definitions
Dependent Variables		
Radical Innovation	Continuous	Percentage of the firm´s sales from products new to the market in the last 2 years
Incremental Innovation	Continuous	Percentage of the firm´s sales from products new to the firm in the last 2 years
Independent Variables		
APD	Continuous	Alliance Portfolio Diversity
ADP^2	Continuous	Alliance Portfolio Diversity squared
Moderator variables		
R&D education	Continuous	Percentage of R&D staff with third level education or higher
R&D Skills	Continuous	Percentage of R&D top skilled workers
Control variables		
Firm Size	Continuous	Number of employees (Ln)
Firm SizeSq	Continuous	Number of employees (Ln) squared
Prior experience	Binary	Firm's prior experience in external collaboration
R&D intensity	Continuous	R&D expenditure as a proportion of total sales
Export intensity	Continuous	Ratio of export sales to total sales
Industry	Binary	Dummy variables indicating the sector where the firm operates
Year	Binary	Dummy variables indicating the year to which observations belong to (2005-2012)

Model and estimation

We use random-effects panel Tobit models to test our hypotheses since our dependent variable is the percentage of sales from innovative products (radical and incremental), a variable that is truncated at zero and 100. The model is specified as:

$$Y_{it}^{*} = X_{it}\beta + \alpha_i + \varepsilon_{it} \qquad (i = 1; \ldots; n; t = 1; \ldots; T)$$

$$\text{While} \quad \begin{cases} y_{it} = y^*_{it} & \text{if } y^*_{it} > 0 \\ y_{it} = 0 & \text{if } y^*_{it} \leq 0 \end{cases}$$

where *i* refers to the firm and *t* refers to the time period. We make the usual random effects assumption that α_i and ε_{it} are independent and identically distributed of $x_{i1},\ldots,x_{iT}$, with zero means and variances $\sigma_{2\alpha}$ and $\sigma_{2\varepsilon}$, respectively (Mátyás and Sevestre, 2008). A log-transformation of both radical and incremental innovations variables is used to reduce the problem of non-normality of the residuals (Laursen and Salter, 2006). In order to observe inter-sectoral differences, estimations are reported for two industry groups: HMHT and LMLT industries. Standard one-tailed z-test is used to compare regression coefficients between the two groups (Paternoster et al., 1998, van Beers and Zand, 2014):

$$Z = \frac{|b_1 - b_2|}{\sqrt{\sigma_{b_1}^2 + \sigma_{b_2}^2}}$$

where b_1 and b_2 are the estimated coefficients associated with the two subsamples, and σ_{b1} and σ_{b2} are the standard errors.

3.4. Results

Table 3.2 provides descriptive statistics for each of the variables. Correlation values among all variables are generally low to moderate, suggesting there is a low risk of facing collinearity issues or redundancies with this set of variables. The highest correlation is 0.58, far less than the problematic level. The general rule of thumb is that correlation values should not exceed 0.75 (Tsui et al., 1995). This is confirmed by the analysis of Variance of Inflation (Vif). The maximum Vif value is 1.49, well below the rule of thumb cut-off of 10, which again indicates that there are no serious multicollinearity problems in the models (Neter et al., 1996).

Table 3. 2. Correlation and descriptive statistics

Variable	**Mean**	**S.D.**	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
1.Radical Innovation	10.14	22.68	1								
2.Incremental Innovation	50.47	45.77	-0.13*	1							
3.APD	0.04	0.09	0.08*	0.15*	1						
4.R&D education	29.74	33.77	0.14*	0.24*	0.24*	1					
5.R&D skills	50.12	43.09	0.16*	0.29*	0.24*	0.58*	1				
6.R&D intensity	0.04	0.21	0.10*	0.02*	0.09*	0.15*	0.14*	1			
7. Export intensity	0.12	0.20	0.05*	0.07*	0.13*	0.16*	0.15*	0.01	1		
8.Prior experience	0.27	0.44	0.07*	0.13*	0.50*	0.18*	0.21*	0.08*	0.04*	1	
9. Firm size (Ln)	4.02	1.39	-0.01*	0.14*	0.28*	0.20*	0.16*	-0.13*	0.14*	0.16*	1
Vif			1.48	1.46	1.40	1.19	1.18	1.48	1.49	1.42	1.46

N = 32836
*p < 0.01; S.D = standard deviation; Vif= Variance Inflation Factor

Table 3.3 and 3.4 present the random-effects Tobit estimations regarding the probability of introducing radial and incremental innovations, respectively. For each subsample (HMHT and LMLT industries), we estimate six model specifications. Model 1 is the baseline model, including only the control variables. In Model 2, we augment our baseline specification by adding APD and its squared term (APD^2) to test Hypotheses 1 and 2. Model 3 adds both linear and squared terms of R&D education. Similarly, Model 4 includes the other dimension of R&D human capital – skills and skills squared. In order to test Hypothesis 3a, the interaction effects of APD and APD^2 with R&D education are introduced in Model 5. Finally, Model 6 includes the interaction effects of APD and APD^2 with the R&D skills to test Hypothesis 3b. To avoid potential multicollinearity problems of interaction terms, we have mean-centered all the independents variables before calculating the interaction terms (Aiken and West, 1991), and subsequently checked to ensure that all Vif values were below 10 (Neter et al., 1996). The random-effect models show an overall adequate level of validity according to various statistics commonly used for interpretation (Hair et al., 2010): highly significant model $\chi 2$, and the smaller values of the Akaike information criterion (AIC) and the Bayesian information criterion (BIC) in models compared with each previous model indicate that the relative goodness of fit in each model improved significantly on the previous one.

3.4.1. Direct effects

Hypothesis 1 suggests a curvilinear relationship between APD and product innovation performance. Model 2 (Tables 3.3 and 3.4) shows that the linear APD term has a significant positive coefficient ($p<0.01$), while APD^2 has a significant negative coefficient ($p<0.01$), suggesting an inverted U-shaped relationship between APD and product innovation performance. Hypothesis 1 is supported. Hypothesis 2 posits that different levels of APD are beneficial for different levels of technological intensity. According to the non-linear specification of APD (Model 2 in Table 3.3), the numbers of partner types at the tipping point[11] is 17.03 for HMHT and 15.4 for LMLT industries for radical innovation performance. The difference between the two subsamples is statistically significant ($z=1.63$, $p<0.05$). For incremental innovation performance, Model 2 (Table 3.4) shows that the optimal APD level is also higher for HMHT (16.58) than for LMLT industries (15.69). However, the difference between both sectors is not significant ($z= 0.89$, ns). Therefore, Hypothesis 2 is partially supported. These results suggest that the impact of partner diversity on product innovation performance is contingent upon the industry's technological intensity and type of innovation

[11] We follow de Leeuw et al. (2014) to calculate the tipping points and the corresponding optimal number of partner types.

activity pursued by companies (Figure 3.2). Greater product complexity, market uncertainty and the divergent set of skills needed to achieve explorative performance objectives in HMHT industries require greater diversity of partners (van Beers and Zand, 2014).

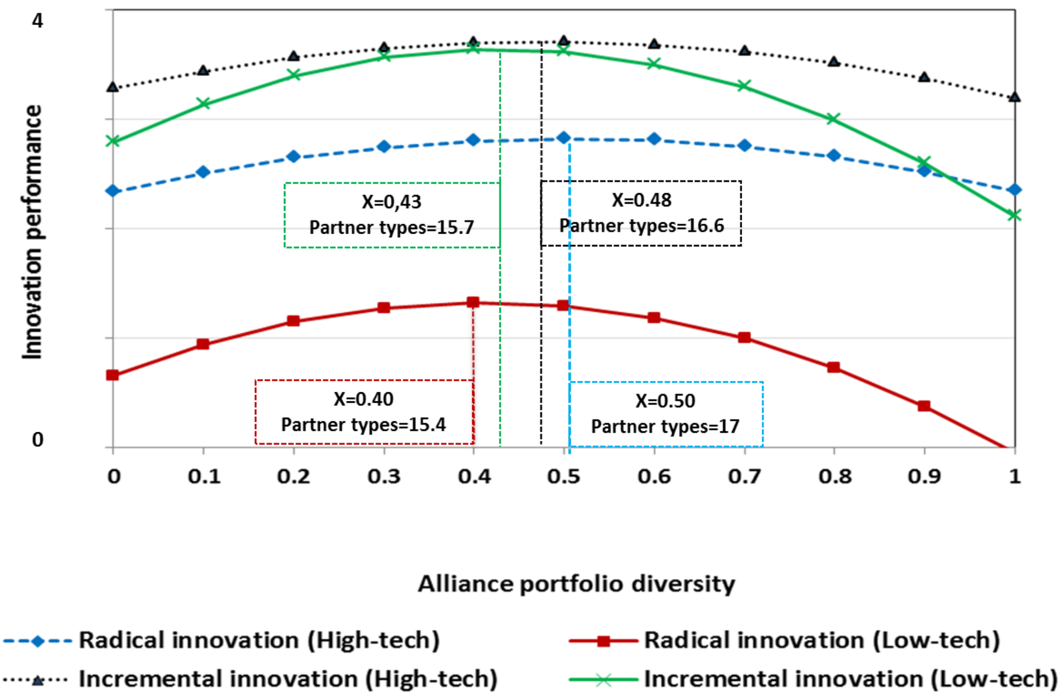


Figure 3. 2. Relationship between APD and product innovation performance – Industry Differences

3.4.2. Moderating effects of R&D human capital

R&D education

Hypothesis 3a stated that R&D education moderates the relationship between APD and product innovation performance. Model 5 (Tables 3.3 and 3.4) shows that the interaction coefficients are significant and negative in linear terms (APD*R&D education) ($p<0.01$) and significant and positive in quadratic terms (APD^2*R&D education) ($p<0.01$). Hence, Hypothesis 3a is supported. Figures 3.3 and 3.4 show the curvilinear relationship between APD and radical and incremental innovation performance for three levels of R&D education: low (minus one standard deviation from the mean), moderate (mean value) and high (plus one standard deviation from the mean) (Aiken and West, 1991). Findings indicate that firms with low levels of R&D education intensity (as a proxy of internal absorptive capacity) exhibit lower innovation performance compared to firms with moderate and high R&D education intensity.

However, results suggest differences in the moderating impact of R&D education depending on firms' technological intensity. For HMHT industries (Figure 3.3.a), a simple slope analysis shows that the relation between APD^2 and radical innovative performance is negative and significant when R&D education is low (b=-10.924, p<0.01) and less significantly negative for moderate R&D education (b=-6.408, p<0.01). Interestingly, the effect of APD^2 is not significant for high R&D education (b=−1.892, ns), suggesting that high levels of R&D education intensity enables HMHT industries to capture value from more open sourcing strategies. In contrast, the moderating effects obtained for LMLT industries (Figure 3.3.b) show that the effect of APD^2 on radical innovation performance is negative and significant for low R&D education (b=-19.90, p<0.01) while less negative and significant for moderate (b =-13.41, p<0.01) and high R&D education intensity (b =−6.92, p<0.01). These results demonstrate that LMLT industries' low absorptive capacity significantly hinders their ability to recognise and access external innovation knowledge (Cohen and Levinthal, 1990, Hervas-Oliver et al., 2011).

Figure 3.4 shows similar results for incremental innovation performance. A simple slope analysis shows that the relationship between APD^2 and incremental innovation performance is negative and significant for low R&D education (b=-18.52 and -30.16, p< 0.01, for HMHT and LMLT, respectively) and moderate R&D education (b =-8.69 and b=-18.35, p< 0.01 for HMHT and LMLT, respectively). However, for high levels of R&D education intensity, the effect of APD^2 is positive but not significant for HMHT (b =1.15, n.s) (Figure 3.4.a), whereas less negative and significant for LMLT (b =-6.55, p< 0.01) (Figure 3.4.b). Overall, these estimates support the absorptive capacity argument that high levels of 'general' human capital intensity are required to increase the effective utilization of external science-based knowledge in HMHT sectors.

R&D skills

Hypothesis 3b posits that R&D skills moderate the relationship between APD and product innovation performance. Model 6 (Tables 3.3 and 3.4) shows that the interaction coefficients are significant and negative in linear terms (APD*R&D skills) (p<0.01) and positive and significant in quadratic terms (APD^2*R&D skills) (p<0.01). Hence, Hypothesis 3b is supported. Figures 3.5 and 3.6 show the curvilinear relationship between APD and radical and incremental innovation for three levels of R&D skills: high, medium and low (Aiken and West, 1991). Results are similar to those presented above for R&D education, suggesting that higher levels of ‘general’ and ‘specific’ human capital intensity enhance firms’ ability to effectively utilise external knowledge (Arora and Gambardella, 1990, Laursen and Salter, 2004). The arc of the APD curve becomes flatter when firms possess moderate and high levels of R&D education and skills intensity.

In terms of industry differences, the results of a simple slope test show that the effect of APD on radical innovation performance (Figure 3.5) is negative and significant for low R&D skills (b=-16.21 and b=-26.99, p<0.01 for HMHT and LMLT, respectively) and moderate R&D skills (b=-8.21 and b=-15.24, p<0.01 for HMHT and LMLT, respectively) but no significant for high R&D skills (b=-0.22 and b=-3.48, ns for HMHT and LMLT, respectively). Significantly, these findings highlight the importance of high levels of ‘specific’ human capital intensity for both sectors to capture value from more open sourcing strategies.

Regarding incremental innovation performance, Figure 3.6 shows a strong moderating effect of R&D skills intensity: for high levels of R&D skills, the relationship between APD and incremental innovation performance turns positive and significant for HMHT industries (b=5.27, p<0.01) and not significant for LMLT sectors (b=-0.61, ns). Taken together, these results stress the importance of ‘specific’ human capital in open innovation, particularly in LMLT sectors, compared to ‘general’ human capital. Hence, skills which can be acquired by performing the work activities themselves (i.e., learning by doing) become critical for building high absorptive capacity to effectively utilize external knowledge. High levels of R&D skills intensity lead to a linear relationship between partner diversity and incremental innovation performance in HMHT industries whereas in LMLT sectors it makes the curvilinear relationship non-significant.

Table 3. 3. Random-effects Tobit models for radical innovation performance

	HMHT						LMLT						z-test
	(1)	(2)	(3)	(4)	(5)	(6)	(1)	(2)	(3)	(4)	(5)	(6)	
Main effects													
APD		5.80***	4.88***	4.59***	6.08***	6.01***		9.84***	8.04***	7.27***	10.51***	10.69***	4.04***
		(0.73)	(0.72)	(0.72)	(0.81)	(0.81)		(0.92)	(0.91)	(0.91)	(1.05)	(1.03)	(1.17)
H1. APD²		-5.76***	-4.67***	-4.20***	-6.96***	-6.19***		-11.94***	-9.63***	-8.27***	-13.41***	-11.26***	6.18**
		(1.50)	(1.48)	(1.48)	(1.89)	(1.72)		(2.22)	(2.19)	(2.18)	(2.73)	(2.42)	(2.68)
H2. N. of Partner at Tipping Point		17.03***						15.40***					1.63***
		(0.39)						(0.27)					(0.47)
R&D education			0.04***		0.04***				0.05***		0.05***		0.01**
			(0.00)		(0.00)				(0.00)		(0.00)		(0.00)
R&D education²			-0.00***		-0.00***				-0.00***		-0.00***		0.00**
			(0.00)		(0.00)				(0.00)		(0.00)		(0.00)
R&D Skills				0.05***		0.04***				0.06***		0.06***	0.01**
				(0.00)		(0.00)				(0.00)		(0.00)	(0.00)
R&D Skills²				-0.00***		-0.00***				-0.00***		-0.00***	0.00**
				(0.00)		(0.00)				(0.00)		(0.00)	(0.00)
Interaction effects													
APD*R&D education					-0.09***						-0.14***		0.05*
					(0.02)						(0.02)		(0.03)
H3a.APD2*R&D education					0.14***						0.21***		0.07
					(0.05)						(0.07)		(0.08)
APD*R&D Skills						-0.10***						-0.17***	
						(0.02)						(0.02)	
H3b.APD²*R&D Skills						0.20***						0.27***	
						(0.06)						(0.07)	
Controls													
R&D intensity	0.73***	0.66***	0.40***	0.36***	0.41***	0.38***	0.74***	0.63***	0.280	0.19	0.30*	0.22	0.01
	(0.14)	(0.14)	(0.14)	(0.14)	(0.14)	(0.14)	(0.18)	(0.18)	(0.179)	(0.18)	(0.18)	(0.18)	(0.22)
Export intensity	0.59***	0.52***	0.39**	0.40***	0.38**	0.41***	0.48**	0.40*	0.312	0.26	0.31	0.26	0.11
	(0.16)	(0.16)	(0.16)	(0.15)	(0.16)	(0.15)	(0.22)	(0.22)	(0.212)	(0.21)	(0.21)	(0.21)	(0.27)
Prior experience	0.63***	0.41***	0.37***	0.36***	0.36***	0.35***	0.82***	0.49***	0.422***	0.40***	0.39***	0.37***	0.19
	(0.07)	(0.08)	(0.07)	(0.07)	(0.07)	(0.07)	(0.08)	(0.09)	(0.084)	(0.08)	(0.08)	(0.08)	(0.11)
Firm size (Ln)	0.33**	0.35**	0.09	0.02	0.07	0.00	0.94***	0.90***	0.591***	0.41**	0.55***	0.38**	0.61***
	(0.15)	(0.15)	(0.14)	(0.14)	(0.14)	(0.14)	(0.18)	(0.18)	(0.172)	(0.17)	(0.17)	(0.17)	(0.23)
Firm size Sq	-0.01	-0.02	-0.00	0.00	-0.00	0.00	-0.07***	-0.07***	-0.051**	-0.03	-0.05**	-0.03	0.06**
	(0.02)	(0.02)	(0.01)	(0.01)	(0.01)	(0.01)	(0.02)	(0.02)	(0.020)	(0.02)	(0.02)	(0.02)	(0.03)
Log-likelihood	-19063.26	-19011.64	-18898.22	-18847.39	-18889.25	-18838.90	-18617.44	-18534.45	-18380.11	-18298.81	-18357.83	-18264.57	
Wald χ2	426.50***	526.51***	743.71***	829.93***	758.49***	842.32***	486.85***	641.81***	926.42***	1059.28***	961.52***	1108.95***	
AIC	38182.53	38083.28	37860.43	37758.79	37846.51	37745.81	37308.87	37146.9	36842.23	36679.62	36801.66	36615.14	
BIC	38395.28	38311.23	38103.58	38001.93	38104.85	38004.15	37597.6	37451.23	37162.17	36999.56	37137.21	36950.68	

Standard error in parentheses. *Significance at 1%;**significance at 5%;***significance at 10%. Year and sector dummy variables were included in the analysis but results are omitted here.

Table 3. 4. Random-effects Tobit models for incremental innovation performance

	HMHT						LMLT						z-test
	(1)	(2)	(3)	(4)	(5)	(6)	(1)	(2)	(3)	(4)	(5)	(6)	
Main effects													
APD		5.35***	3.91***	3.52***	6.03***	6.26***		11.97***	9.32***	8.04***	12.72***	11.80***	6.62***
		(0.89)	(0.88)	(0.88)	(0.98)	(0.97)		(1.39)	(1.38)	(1.37)	(1.56)	(1.53)	(1.65)
H1. APD^2		-5.60***	-3.91**	-3.29*	-8.69***	-6.98***		-13.99***	-10.74***	-8.61**	-18.35***	-14.36***	8.39***
		(1.86)	(1.84)	(1.83)	(2.34)	(2.07)		(3.42)	(3.37)	(3.35)	(4.16)	(3.70)	(3.89)
H2. N. of Partner Types at Tipping Point		16.58***						15.69***					0.89
		(1.70)						(1.24)					(2.10)
R&D education			0.06***		0.06***				0.08***		0.08***		0.02***
			(0.00)		(0.00)				(0.00)		(0.00)		(0.00)
R&D education^2			-0.00***		-0.00***				-0.00***		-0.00***		0.00***
			(0.00)		(0.00)				(0.00)		(0.00)		(0.00)
R&D Skills				0.06***		0.06***				0.08***		0.07***	0.02**
				(0.00)		(0.00)				(0.00)		(0.00)	(0.00)
R&D Skills^2				-0.00***		-0.00***				-0.00***		-0.00	0.00**
				(0.00)		(0.00)				(0.00)		(0.00)	(0.00)
Interaction effects													
APD*R&D education					-0.15***						-0.19***		0.04
					(0.03)						(0.04)		(0.05)
H3a.APD^2*R&D education					0.28***						0.38***		0.10
					(0.07)						(0.10)		(0.12)
APD*R&D Skills						-0.20***						-0.02***	0.00
						(0.03)						(0.023)	(0.03)
H3b.APD^2*R&D Skills						0.40***						0.48***	0.00
						(0.08)						(0.10)	(0.00)
Controls													
R&D intensity	-0.08	-0.14	-0.57***	-0.64***	-0.54***	-0.60***	1.16***	1.00***	0.49*	0.32	0.52*	0.36	1.24***
	(0.17)	(0.18)	(0.18)	(0.18)	(0.18)	(0.18)	(0.29)	(0.28)	(0.27)	(0.27)	(0.27)	(0.27)	(0.33)
Export intensity	0.22	0.15	-0.04	-0.03	-0.05	-0.02	0.09	-0.01	-0.14	-0.23	-0.14	-0.24	0.13
	(0.19)	(0.19)	(0.19)	(0.18)	(0.19)	(0.18)	(0.31)	(0.31)	(0.31)	(0.31)	(0.31)	(0.30)	(0.36)
Prior experience	0.65***	0.45***	0.38***	0.37***	0.36***	0.35***	1.17***	0.77***	0.68***	0.64***	0.64***	0.61***	0.52***
	(0.09)	(0.09)	(0.09)	(0.09)	(0.09)	(0.09)	(0.12)	(0.12)	(0.12)	(0.12)	(0.12)	(0.12)	(0.15)
Firm size (Ln)	1.34***	1.36***	1.00***	0.90***	0.98***	0.86***	2.44***	2.42***	2.05***	1.79***	2.02***	1.78***	1.10***
	(0.17)	(0.17)	(0.17)	(0.16)	(0.17)	(0.16)	(0.25)	(0.25)	(0.24)	(0.24)	(0.24)	(0.24)	(0.30)
Firm size Sq	-0.11***	-0.12***	-0.09***	-0.08***	-0.09***	-0.08***	-0.21***	-0.22***	-0.20***	-0.17***	-0.20***	-0.17***	0.10***
	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)	(0.03)
N. observations	14740	14740	14740	14740	14740	14740	18096	18096	18096	18096	18096	18096	
Log-likelihood	-22804.25	-22776.65	-22618.59	-22546.11	-22602.31	-22519.79	-24305.00	-24248.52	-24113.79	-24028.57	-24100.24	-24013.03	
Wald $\chi 2$	2117.87***	2171.29***	2429.03***	2540.56***	2455.18***	2579.99***	1866.46***	1954.76***	2153.36***	2269.90***	2169.43***	2288.94***	
AIC	45664.49	45613.31	45301.17	45156.23	45272.62	45107.59	48684	48575.03	48309.58	48139.14	48286.48	48112.05	
BIC	45877.25	45841.25	45544.32	45399.37	45530.96	45365.94	48972.73	48879.37	48629.52	48459.09	48622.02	48447.6	

Standard error in parentheses. *Significance at 1%;**significance at 5%;***significance at 10%. Year and sector dummy variables were included in the analysis but results are omitted here.

Figure 3. 3. Moderating effect of R&D education on radical innovation performance: (a) HMHT and (b) LMLT

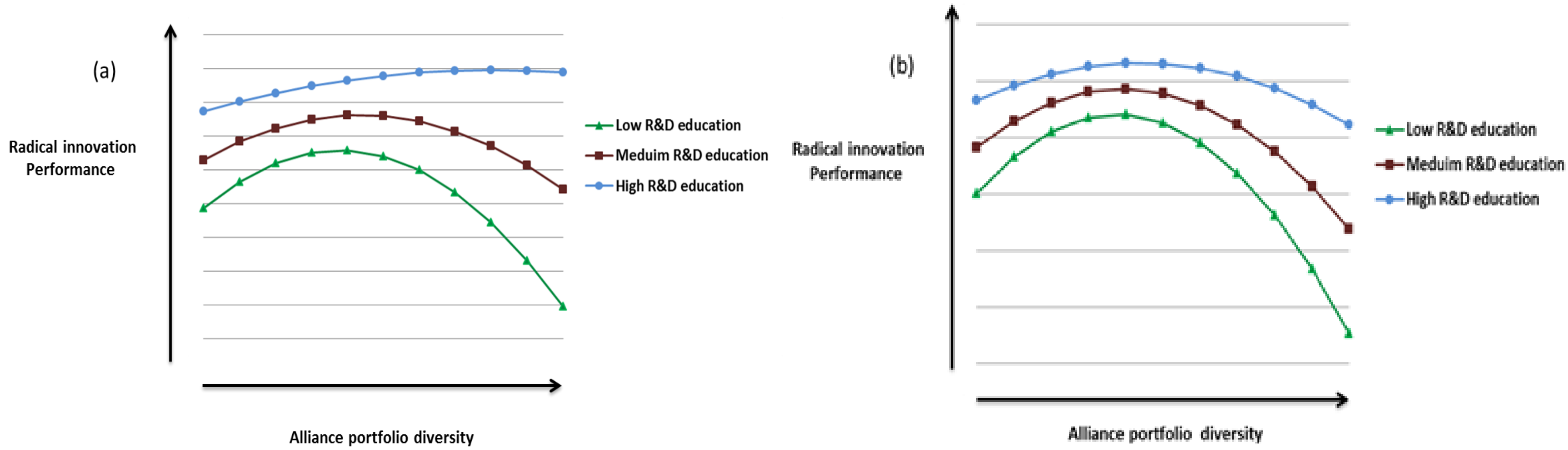


Figure 3. 4. Moderating effect of R&D education on incremental innovation performance: (a) HMHT and (b) LMLT

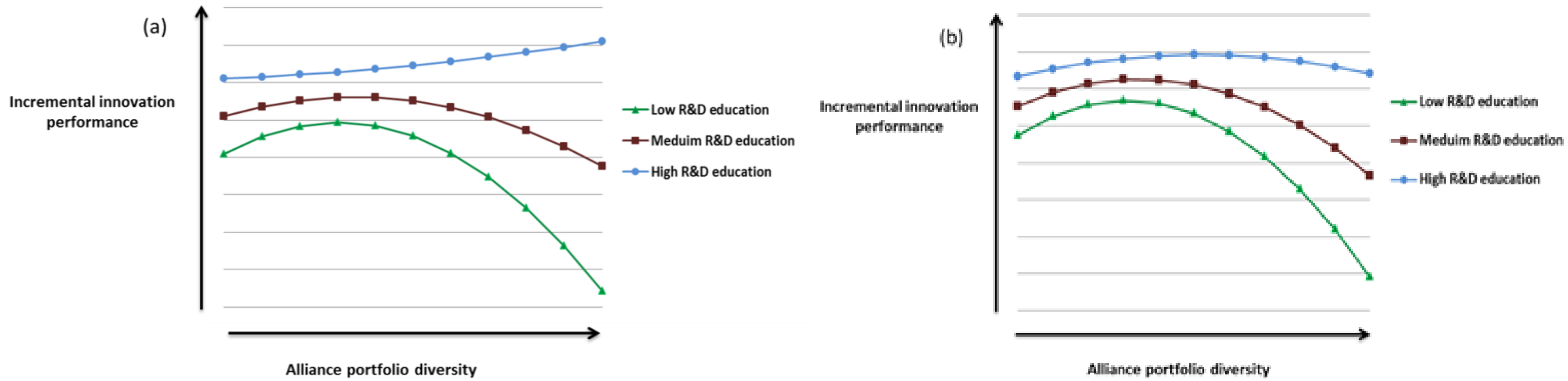


Figure 3. 5. Moderating effect of R&D skills on radical innovation performance: (a) HMHT and (b) LMLT

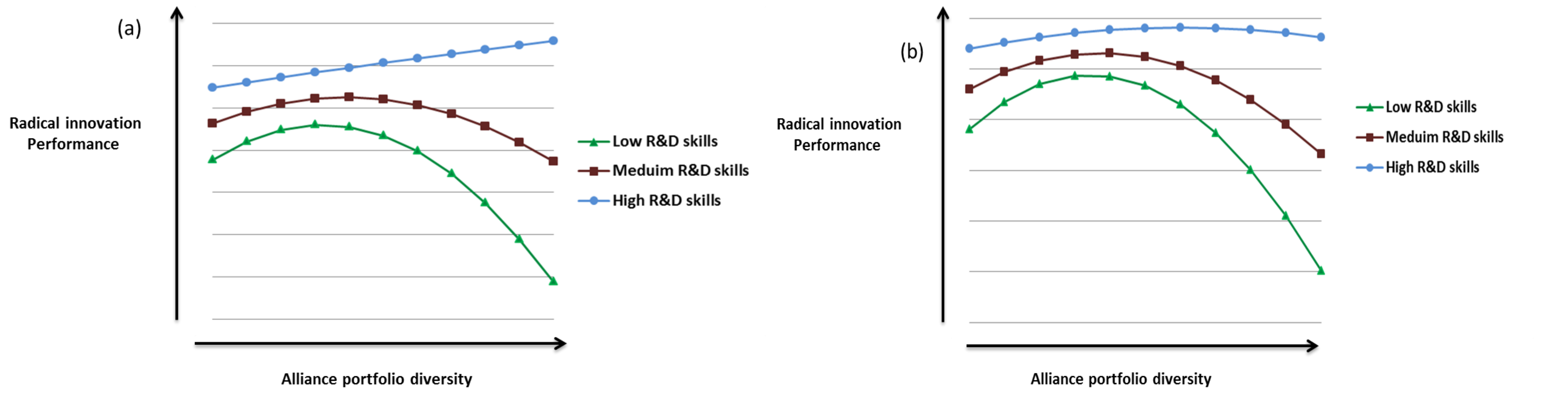
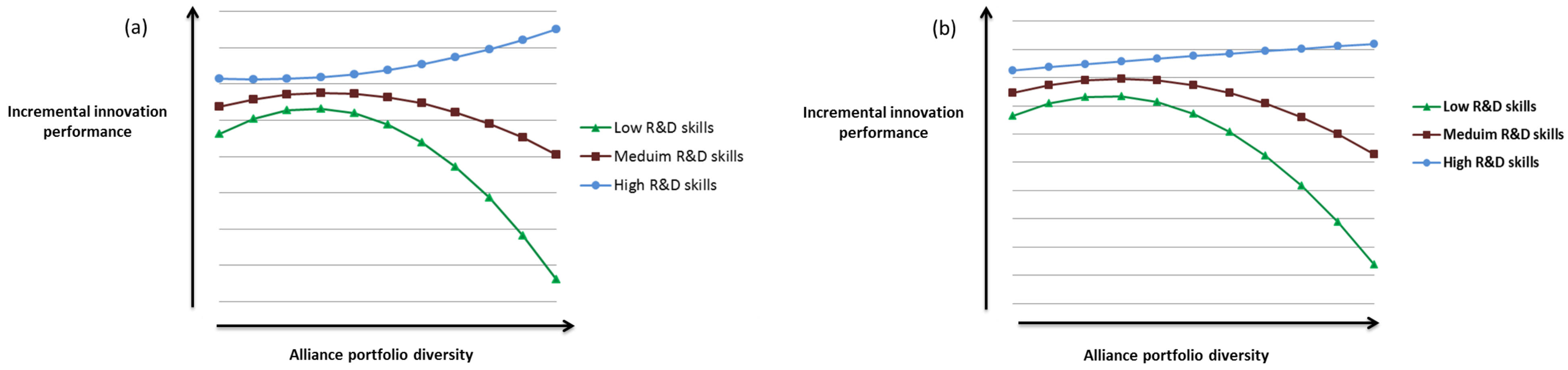


Figure 3. 6. Moderating effect of R&D skills on incremental innovation performance: (a) HMHT and (b) LMLT



3.5. Discussion and Conclusions

Our aim in this research has been to add to our understanding of the relationship between partner diversity and innovation performance. Alliance partner portfolio has attracted significant interest among organisations and policy makers as collaboration becomes a key vector of innovation-related knowledge flows (OECD, 2010). In line with previous work (de Leeuw et al., 2014, Lin, 2014), our results show a positive, curvilinear relationship between APD and product innovation performance, suggesting that firms that collaborate with different external partners exhibit a superior innovation performance, but only up to a point. Beyond this threshold, the increase of partner diversity would be detrimental to firms' innovative performance. Openness towards external knowledge sources enables firms to access diverse markets and technological knowledge (Lin, 2014); however too much partner diversity beyond the optimal point could lead to high management costs and the probability of opportunism (Combs and Ketchen, 1999) and appropriation concerns (Mol, 2005), negatively affecting as a result the transfer of external knowledge by firms into their innovation processes (Katila and Ahuja, 2002, Laursen and Salter, 2006). Thus, an optimal level of partner diversity exists for companies to maximise innovation performance; however, a significant difference is found in the optimal level depending on the level of product novelty (radical vs incremental) and industry's technological intensity (high vs low). Our findings indicate that high-tech industries characterised by rapid technology changes require a broader set of external partners to maximise radical innovation performance than low-tech industries. Interestingly, we did not find significant industry differences for incremental innovation performance.

Two important conclusions can be drawn for these findings. First, our results corroborate the view that high-tech industries need a broad business ecosystem to remain competitive in their rapidly changing business environment (Ili et al., 2010). Second, both sectors require similar partner diversity to maximise incremental innovation performance, thus emphasising the effect of partner diversity in high-tech industries to achieve explorative performance objectives (van Beers and Zand, 2014).

Interaction effects indicate a statistically significant moderating impact of R&D human capital on the relationship between alliance diversity and product innovation performance (Hypotheses 3a and 3b). These findings support previous work concerning the importance of human capital to assimilate and integrate external knowledge into internal innovation activities (McGuirk et al., 2015, Caloghirou et al., 2004, Spithoven and Teirlinck, 2010). The influence of human capital is evident when firms exhibit high levels of R&D education and skills intensity leading to higher product innovation performance. We suggest that top educated and highly skilled R&D staff, by enabling internal capabilities, act as a facilitating mechanism to explore and deploy external knowledge flows successfully, prompting the optimal level of partner diversity to increase (Figures 3.3 to 3.6). This result confirms our hypothesising that 'general' and 'specific' human capital can mitigate the decline in innovation performance at higher level of APD. In contrast, firms with low levels of R&D human capital fail to leverage the potential benefits of external knowledge to create an innovative edge (Lin, 2014). In summary, human capital plays a critical role in supporting firms to overcome challenges in cross-border knowledge transfer at high levels of partner diversity. Our analysis supports the arguments by Kotabe (1990) that firms with high levels of internal R&D capabilities avoids the loss of relevant process knowledge in manufacturing and engineering which help them exploit external knowledge.

Regarding industry differences found for human capital as a moderating variable, we argue that certain dimensions of human capital are more successful in maximising the benefits of partner diversity. Our finding that R&D employee's education intensity is more helpful for high-tech innovative performance than for low-tech when industries adopt higher partner diversity supports the argument that the more complex and tacit knowledge is involved in cooperation agreements the higher the need for greater absorptive capacity, which can be linked to the presence of top educated R&D staff (Cohen and Levinthal, 1990). Absorptive capacity enables high-tech firms to effectively integrate external knowledge flows in their internal innovation processes.

In contrast, the slopes for low-tech industries are significant and negative suggesting clear difficulties for companies in low-tech sectors to extract value from diverse cooperation networks, and thereby the need to invest in internal R&D capabilities to benefit from external collaboration. This sector generally possess limited internal capacity and recourses (Spithoven et al., 2011) to take advantage from a wide range of external source of knowledge and to manage it effectively. Our finding supports the view regarding the complementarity between internal R&D and external knowledge flows (Veugelers, 1997a, Grimpe and Kaiser, 2010, Mol, 2005) and the need to invest in in-house R&D to benefit from external ideas and technology.

A key finding emerging from our study is the importance of high levels of 'specific' human capital to capture value from more open sourcing strategies, particularly in low-tech industries, compared to 'general' human capital. Specifically, R&D skills exerts a strong moderating impact on incremental innovation performance stressing the merits of investing in the training of R&D teams in the specific job skills involved in exploitative innovation as opposed to providing general knowledge. Top skilled R&D staff would give firms broader interfaces to engage with a multitude of potential external partners to achieve exploitative performance objectives (Ketata et al., 2015).

3.5.1. Contributions and managerial implications

Several managerial implications follow from this discussion and should be of interest to managers. First, this study contributes to a better understanding of how manufacturing firms should configure their alliance portfolio depending on their knowledge needs by prioritizing their objectives in terms of the type of innovation they seek to develop. Since the levels of APD are optimal at different levels depending on the type of industry and innovation novelty, managers should design their alliance portfolio accordingly (de Leeuw et al., 2014). Our findings demonstrate that to maximize radical innovation performance in high-tech industries, a larger set of external partners would be required compared to incremental innovation. Contrary, for low-tech industries, diversity in R&D collaborations represents an equal vital source of knowledge for both innovation outcomes.

Second, R&D education and skills are valuable assets, influencing a firm's capability to extract value from partner diversity. Our findings highlight the need to invest in internal research capabilities by upskilling and training R&D staff to tap into innovation knowledge sources and develop absorptive capacity (Lin, 2014; Muscio, 2007). By investing in the acquisition of new skills, R&D employees could more effectively absorb and deploy local or distant knowledge relevant to future innovation (Huang et al., 2015).

Our focus on manufacturing firms offers an important contribution to the open innovation literature, as we demonstrate how 'general' and 'specific' human capital can maximize partner diversity to ensure sustainable competitive advantage through increased innovative performance. Managing partner diversity is especially important for high-tech firms, which require a wider knowledge base to remain competitive in their complex and dynamic business environments. These industries strongly require specific knowledge and skills to ensure cross-fertilization and combination of new streams of knowledge (Covin et al., 1990).

Finally, our findings suggest why firms differ in their internal ability to actively search for external knowledge. Overall, R&D education and skills intensity act as an internal mechanism to capture value from more open sourcing strategies. Therefore, manager should develop high internal capabilities to integrate external knowledge beyond established industry boundaries and enhance potential absorptive capacity for future knowledge transfer and knowledge sharing (Enkel and Heil, 2014). Managers should consider their external relationships structure as a capability enhancing process (Xia, 2013) that will allow firms‘ employees to develop broader skills in the future. This is particularly relevant for low-tech firms which are constrained in their ability to collaborate with different types of external partners due to their limited absorptive capabilities. Hence, we argue that this needs to be reflected in a firm's investments in absorptive capacity. External collaboration does not substitute lacking or insufficient internal innovation capabilities; rather it increases complexity for firms. Thus, dealing with increasing complexity requires building stronger internal capabilities.

3.5.2. Limitations and future research

We acknowledge several limitations in our paper and suggest related opportunities for future research. First, the focus of this study is specifically on firms' abilities, embodied in their educated and skilled human resources to absorb external knowledge for innovation. Future research could be extended by examining the key role of strategic HR management practices; such knowledge management knowledge, training programs and integration of knowledge of the member of firm, those practices is usually linked with higher adaptability, flexibility and competitive advantage. Second, we use data from Spain so evidence from other countries on the differential impact of absorptive capacity dimensions, such as education, skills and training on innovation performance might help to develop more general empirical evidence in future research direction.

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CHAPTER IV. Combined effects of ozone and freeze-drying on the shelf-life of Broiler chicken meat

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Chapter IV. Combined effects of ozone and freeze-drying on the shelf-life of Broiler chicken meat

4.1. Introduction

Chicken meat is one of the most popular food commodities in Europe and the second most preferred meat by European Union consumers after pork meat (FAO, 2014). Some of the reasons for the popularity of this kind of meat are the relatively low price, low fat content and the high nutritional value. Generally, poultry meats are highly perishable to bacterial contaminants due to large amounts of variable nutrients, a high water activity (a_w) and a higher final pH limiting the shelf-life of the product (Lawrie, 1998). In the case of meat and meat products, enzymatic and chemical reactions are responsible for the initial loss of freshness, while microbial activity is responsible for subsequent spoilage. The contamination by several pathogenic microorganisms can cause severe foodborne diseases in consumers (Jayasena et al., 2015).

However, the manufacturing of meat products is constantly challenged to meet rapid changes in consumer tastes and demands for healthier food products, safe, natural, free of conventional chemical preservatives with an extended shelf-life. Consumer acceptance is the key success factor for the development of successful meat products (De Barcellos et al., 2010) and meat safety is considered to be a prerequisite by consumers (Van Wezemael, Verbeke, Kügler, de Barcellos, & Grunert, 2010). For this purpose, the multiple hurdle concept is an integrated basic approach in food preservation and the hurdle technology is generally defined as using the simultaneous or the sequential application of factors and/or treatments affecting microbial growth (Turantaş, Kılıç, & Kılıç, 2015). The principle of this concept can be explained as two or more inhibition and inactivation methods (hurdles) at suboptimal levels are more effective than one (Leistner, 1992). This method is becoming attractive, because several hurdles are used to obtain the optimum combinations which do not affect the sensory quality, while maintaining the microbial stability and safety of the food (Alzamora, Tapia, Argaíz, & Welli, 1993; Leistner, 1992). In fact, ozonation and freeze-drying were employed as hurdles in the present study to develop a new raw meat product from *Broiler* chicken breasts. Ozone is a powerful antimicrobial agent very effective in destroying a wide range of microorganisms including viruses, bacteria, fungi, protozoa, and bacterial and fungal spores (Khadre & Yousef, 2001). This agent inactivates bacteria by disrupting the cell membrane and cell wall, leading to cell lysis (Muhlisin, Cho, Choi, Hahn, & Lee, 2015). Ozone is used in an extensive range of agricultural products, such as vegetables, fruits, fish (Manousaridis et al., 2005) and meat products (Muhlisin et al., 2015; Sekhon et al., 2010; Stivarius, Pohlman, McElyea, & Apple, 2002). The bactericidal effect of ozone depends on several factors, such as temperature, relative humidity, pH and the presence of organic matter (Kim, Yousef, & Chism, 1999).

Freeze-drying is the most common form of food preservation to improve the long-term stability of food because the percentage of humidity and the water activity can be reduced, if the product is well lyophilized, which retards the growth of microorganisms for a long period. This process applies only for high added-value products (Abdelwahed, Degobert, Stainmesse, & Fessi, 2006). Freeze-drying has many applications on food products, such as chicken meat, raw beef, mushrooms, fruits, carrots, tomato, eggs, etc. (Babić, Cantalejo, & Arroqui, 2009; Chang, Lin, Chang, & Liu, 2006; Hammami & René, 1997; Litvin, Mannheim, & Miltz, 1998). The many advantages of lyophilisation make it one of the technologies attracting the attention of the food industry, including: (i) the conservation of the primary physical and chemical characteristics of the product, (ii) a low residual humidity (<10%) providing easy handling during shipping and storage of the lyophilized product and, (iii) long-term stability.

The aim of this research was to study the combined effects of ozone and lyophilisation on the shelf-life extension of *Broiler* chicken meat fillets, stored at room temperature by evaluating microbiological load and sensory characteristics, in order to develop new high-quality raw meat products from fresh chicken meat, safe, with a high nutritional value, with no additives added and long-lasting at room temperature. Therefore, these meat products can be preserved and transported with no refrigeration, due to the relative reduction of moisture content and water activity (energy saving, as no freezing is required). Furthermore, this type of food product would allow a long shelf-life in the case of natural catastrophes (earthquakes, floods,…), export to third countries, military campaigns, mountain climbers and scarcity in electricity supply.

4.2. Materials and methods

4.2.1. Raw matter and sample preparation

Broiler chicken breast meat was obtained from U.V.E., S.A. (Tudela, Navarre, Spain). Chickens were 42 days old before slaughtering with approximately 2 kg of weight. All breasts were stored in a refrigerated room (2-4°C) for the time of reception until used. The samples were trimmed of visible fat and nerves. They were cut into pieces (approximately 3 x 3 cm^2 of section and of 0.7 cm in thickness), before the analyses. Then, they were divided into three batches. The first batch was vacuum-packed, refrigerated and stored at 4±0.5 °C (P Selecta, Pharmalow, Tarre, Navarra, Spain). To characterize the fresh meat, physical-chemical measurements (pH, colour, water activity, humidity and texture) were performed. After characterization, the same batch was vacuum-packed, deep-frozen, and stored at -40±1 °C (Climas, Barcelona, Spain) and used as an external reference of raw meat for sensory and microbiological analyses. The second batch of meat samples was subjected to freeze-drying only, and vacuum packed and stored in a dark place at room temperature (21±1 ºC) and used as an internal control. The third batch of meat samples was treated with ozone, freeze-dried, vacuum-packed and stored in a dark place at 21±1 °C.

4.2.2. Ozone treatments

Ozonation assays were carried out in a 3 m^3 volume refrigerated chamber (Eurozon, Ecologyc 2000, Sestao, Vizcaya, Spain) to a continuous flow of ozone gas at 4±0.5°C and 90±1% relative humidity. These conditions are important for the efficiency of the bactericidal effect of ozone (Kim et al., 1999). Ozone was generated *in situ*, utilizing a UV radiation using an ozone generator (Rilize, model 3060 Eurozon, Sestao, Spain). Ozone concentrations inside the chamber were monitored continuously by circulating air from the chamber through an ultraviolet absorption ozone gas analyzer (Ozomat MP, Anseros, Germany). The different treatments are shown in Table 4.1. Treatment combinations for this study included three ozone concentrations (0.72, 0.6 and 0.4 ppm) and four exposure times (120, 60, 30 and 10 min).

Table 4. 1. Concentrations and exposure times of gas ozone on meat samples

Treatments	Ozone concentrations (ppm)	Exposure times (min)
(0) Trt-0 (Internal control)	-	-
(1) Trt-0.4/30	0.4	30
(2) Trt-0.4/60	0.4	60
(3) Trt-0.4/120	0.4	120
(4) Trt-0.6/10	0.6	10
(5) Trt-0.6/30	0.6	30
(6) Trt-0.72/10	0.72	10
(7) Trt-0.72/30	0.72	30

4.2.3. Freeze-drying process and packaging of samples

After ozone treatments, samples were dehydrated in a pilot scale freeze-dryer (Model Lyobeta 25, Telstar Industrial, S.L., Barcelona, Spain). The different parameters of the freeze-drying process assayed in this study were the same in all treatments and were the best conditions described in the research work of Babić et al. (2009). Briefly, slow freezing, 20.5 h of primary drying (12 h at 0˚C and 8.5 h at 10˚C) at 30 Pa.

All the samples were vacuum-packed, using a vacuum packaging machine (Model SAMMIC V-640, Gipuzkoa, Spain), in impermeable plastic trays type polyamide/polyethylene PA/PE 20/70 200x300 (Ilpra, Barcelona, Spain). The double-layer of the trays resulted in a strong and relatively impenetrable bag for both air and moisture and had an oxygen transfer rate of less than 50 $cm^3m^{-2}d^{-1}bar^{-1}$, permeability to CO_2 less than 150 $cm^3m^{-2}d^{-1}bar^{-1}$ and a water vapor permeability of less than 2.8 g m^{-2} d^{-1}.

Two meat controls were used in this study: (1) Lyophilized chicken samples (trt-0), that were not exposed to ozone treatment and were used as an internal control in order to analyse the efficacy of the combination of ozone and lyophilisation on the self-life of meat. (2) Frozen meat used as an external reference of raw meat (due to the similarity of those samples with the ozonated freeze-dried samples) for sensory and microbiological analyses.

4.2.4. Analyses of samples

Physical and chemical analyses

Physical and chemical analyses (pH, water activity (a_w), humidity, percentage of rehydration, colour and the texture) were carried out during the first day of storage for characterising the fresh meat and all treated samples.

The pH was measured using a pH-meter (Crison PH 25, S.A, Barcelona, Spain) with a combined electrode, which penetrates the meat samples. Water activity (a_w) was measured by means of a hygrometer (Novasina RS-232, LabMaster, Switzerland). Humidity of fresh meat was determined in a stove (P Selecta, Digitronic, Barcelona, Spain) at 102 ± 2ºC until constant weight, according to the ISO R-1442 regulation (ISO, 1973) and the Spanish Official Method for the Analysis of Meat Products (B.O.E., 29/8/79). Humidity of dried meat was determined following the ISO R-1442 method (AOAC, 1975), by using a gravimetric infrared stove (Gram, ST-H 50, Barcelona, Spain).

In order to know how much water was absorbed by freeze-dried chicken meat and their fully rehydration characteristics, the samples were rehydrated in trays filled with distilled water at 21-22 ºC. The change in mass of freeze-dried chicken meat was measured each half an hour, when all meat samples were taken out and dried with a blotting paper, then each sample was weighed. This procedure was repeated until obtaining constant weight of the samples. The percentage of rehydration was calculated using the following expression, proposed by Babić et al. (2009):

$$\text{Rehydration (\%)} = (W_r - W_l)/(W_0 - W_l)x100$$

Where,

W_r: weight of rehydrated sample (g)

W_l: weight of lyophilized sample (g)

W_o: weight of fresh sample (g)

The maximum force (N) was determined using a TA.XT Plus Texture Analyser (Stable Micro Systems Ltd, Aname S.L, England), all the samples being cut perpendicularly to the muscle fibre direction at a crosshead speed of 10 mm/s. Prior to the analysis, samples were packaged in impermeable plastic bags and introduced in a water bath (P Selecta, Precisterm, Barcelona, Spain) at 80±1 °C for 2 min.

The measurement of meat colour was studied by means of a Minolta Chrome Meter CM-2500d (Minolta Co. Ltd. Osaka, Japan), using CIELAB colour space (CIE, 1976) with the D65 Standard illuminate and the 10° Standard Observer. The colour was expressed as the colour coordinates L* (lightness), a* (redness), and b* (yellowness).

Microbiological analyses

The total aerobic mesophilic bacteria (TAMB) lactic acid bacteria (LAB), *Escherichia coli* and *Salmonella spp.* were determined in frozen, only freeze-dried and combined treated samples after 0, 2, 4, 6 and 8 months of storage. 25 g of chicken breast samples from each treatment were previously weighed, transferred to a sterile bag with 225 ml sterile peptone water (Oxoid, CM0009, Hampshire, UK), and homogenised for 30 min using a stomacher (Stomacher 400 Circulator Seward, Colworth, UK). For each sample, appropriate serial decimal dilutions were prepared in the same sterile peptone water solution. Duplicate plates were made for each dilution. TAMB were determined according to ISO norm 4833 (05/2003), by using Plate Count Agar (PCA) (Biomérieux, Marcy-l'Etoile, France) after incubation at 35±1°C for 48±2 h. LAB were determined according to the technique ISO 15214 (1998) on Man, Rogosa and Sharpe agar (MRS, Oxoid, UK), incubated at 30 ºC during 3 days. Catalase test was done on presumptive lactic acid bacteria. *E. coli* was determined according to the ISO 16140 (ISO, 2003) and was incubated at 44±1 °C for 18-24 h by using Coli ID (Biomérieux, Marcy-l'Etoile, France). *Salmonella* was detected qualitatively (presence or absence) by the Enzyme Linked Fluorescent Assay (ELFA) performed by the mini-VIDAS instrument system (bioMerieux, Marcy l'Etoile, France). A pre-enrichment process was performed in broth buffered peptone water (BPW CM1049, Oxoid) for plate incubation at 37±1 °C for 24-26 h. After incubation periods, the procedure DIN 10121 (2000) was followed. Thus, 0.1 ml of pre-enriched samples was introduced into 10 ml of Xylose Lysine Deoxycholate Agar plates (XLD-agar, bioMerieux, Marcy l'Etoile, France) and incubated at 41.5± 1 ºC for 24-26 h. After incubation, 1-2 ml of each XLD broth culture were combined and heated in a boiling water bath at 95-100 ˚C for 15± 1 min. After being cooled down to room temperature, 0.5 ml was transferred into a Vidas *Salmonella* strip (SLM), which was analysed in the mini-VIDAS. Results were available after 45 min. Suspicious *Salmonella* colonies were inoculated onto XLD-agar, incubated at 37±

1°C for 24 h and then biochemically and serologically identified using *Salmonella* Latex test (Oxoid, Basingstoke, UK). All microbiological tests were carried out in duplicate, and the results expressed as log cfu/g.

Descriptive sensory analyses

The descriptive sensory evaluation was performed by 5 trained panellists and the method of Hunt et al. (1991) was adopted to describe the sensory characteristics of the rehydrated treated chicken meat in five attributes: appearance, percentage of surface discoloration, chicken odour, odour characteristics and overall impression. Samples were evaluated for each attribute using a 7-point scale, in which 1 indicates the lowest score and 7 represents the highest score. For the evaluation of the texture profile attributes (TPA), the panel evaluated the rehydrated-cooked treated chicken meat for the three following sensory attributes: hardness, juiciness and chewiness (Lyon & Lyon, 1990). Each attribute was rated on a seven-point scale, with a score 1 equivalent to the lowest intensity of the attribute and the score 7 to the highest intensity of the attribute. In both evaluations, visual and TPA, the limit of acceptability was 4.

4.2.5. Statistical analyses

The analyses of variance (ANOVA) were carried out using the statistical package SPSS 11.0 software (SPSS Inc. Chicago, IL, USA). Pearson´s correlation analyses and mean comparison were analysed according to Tukey's test, the significance being assigned at $P < 0.05$ level.

4.3. Results and discussion

Firstly, a characterization of raw meat and all treated meat samples was carried out during the first day of storage. Secondly, the shelf-life of treated meat was studied during months 0, 2, 4, 6 and 8.

4.3.1. Physical and chemical characteristics of chicken breast meat under different combined treatments

The values of pH, water activity (a_w), humidity (%), rehydration (%), and texture (N) of treated and untreated meat samples are presented in Table 4.2. The mean pH was 5.88±0.21 for fresh meat and 6.05±0.15 for freeze-dried meat (trt-0). The combination of ozone and lyophilisation reduced slightly the pH values in almost all combined treated samples, but the differences were statistically significant (P<0.05) only between the treated samples at 0.6 ppm of ozone for 10 min (trt-0.6/10) and the untreated samples (trt-0). Similarly, Clavijo (2005) reported lower pH values for ozonated dried chicken breast fillets, as compared to control (non-ozonated dried) chicken breast fillets. This decrease of pH in combined treatment tends to inhibit microbial growth and survival (Stivarius et al., 2002). Alonso-Calleja, Martínez-Fernández, Prieto, and Capita (2004) found a high positive correlation between pH and microbial counts, indicating that high pH values favorably influences microbial growth.

However, water activity and humidity may be considered the most important factors in predicting the survival of microorganisms in food due to their direct influence on product quality and stability. The initial a_w and moisture content of fresh chicken meat were about 0.984±0.002 and 73.88±0.06%, respectively. After lyophilisation, a significant decrease (P<0.05) in those values was observed for the samples treated with lyophilisation (trt-0) (0.131±0.002 for a_w and 2.93±0.06% for humidity). The significant decrease in levels of a_w and humidity in meat during lyophilisation might inhibit microorganisms´ growth in meat. Likewise, the reduction of a_w and humidity values were similar to those found by Babić et al. (2009) in freeze-dried chicken meat with the same lyophilisation conditions.

For the samples with combined treatment (ozone and lyophilisation), a_w values were significantly affected by both factors concentration of ozone and its time of exposure. Those values increased significantly (P<0.05) when ozone concentration and exposure time increased. The water activity (a_w) of the combined samples ranged between 0.162±0.005 and 0.268±0.009. It is important to note that all samples had a_w values lower than 0.6. This value is considered as the limit of growth for microorganisms in food (Leistner, 1992), as all bacterial species fail to grow at a_w of less than 0.6 (Barreiro & Sandoval, 2006). Nevertheless, the moisture content increased when contact time of ozone increased, but it was found not to be significantly influenced by ozone concentration. Thus, samples from treatment trt-0.4/120 were noted to have a higher (P<0.05) moisture content than samples from treatments trt-0.4/60 and trt-0.6/30. No significant differences were observed in samples that were ozonated for 10 and 30 min.

On other hand, the mean rehydration percentage for the freeze-dried samples (trt-0) was 72.88±1.28%. These results were similar to those reported by Babić et al. (2009)

who found the highest rehydration percentages of 74.45 ± 8.95 % in freeze-dried *Broiler* chicken meat.

In the case of combined treated samples, the contact time of ozone had a significant effect on the percentage of rehydration (P<0.05), and no significant effect was observed for ozone concentration. Our study suggests that the use of longer exposure time of ozone above 30 min caused significant decrease (P<0.05) of the percentages of rehydration of the samples for both treatments (trt-0.4/60 and trt-0.4/120). These percentages were around 50 %, such products not having economic interest, as half of the product is not suitable to be eaten after rehydration. On the contrary, 30 min ozonation or less did not affect the percentages of rehydration.

Table 4. 2. Determination for pH, aw, humidity (%), percentages of rehydratation (%) and maximum force values (N) for different treatments in chicken meat fillets

Treatments	pH	Aw	Humidity (%)	Rehydratation (%)	Texture (N)
Controls					
Fresh meat	5.88±0.21	0.984±0.002	73.88±0.06	-	30.42±1.41
trt-0	6.05±0.15^{y}	0.131±0.002^{x}	2.93±0.06 ^{x}	72.88±1.28^{x}	40.05±0.93^{x}
Combined treatments: freeze-drying and ozonization (trt-[O_3] in ppm/time of exposure in minutes)					
trt-0.4/30	6.04±0.09^{Aay}	0.162±0.005^{Aay}	2.93±0.08^{Aax}	75.03±1.76^{Cax}	40.91±0.53^{Aax}
trt-0.4/60	6.10±0.08^{Ay}	0.219±0.007^{By}	3.78±0.10^{By}	52.66±1.52^{By}	93.22±0.70^{By}
trt-0.4/120	5.97±0.06^{Ay}	0.238±0.002^{Cy}	8.29±0.19^{Cy}	45.20±0.81^{Ay}	138.60±2.09^{Cy}
trt-0.6/10	5.81±0.03^{Aax}	0.189±0.003^{Aay}	2.96±0.03^{Aax}	73.26±1.97^{Aax}	41.15±0.87^{Aax}
trt-0.6/30	6.10±0.05^{Bay}	0.268±0.009^{Bcy}	2.93±0.04^{Aax}	74.16±1.41^{Aax}	39.67±0.81^{Aax}
trt-0.72/10	5.95±0.09^{Aby}	0.216±0.005^{Bby}	2.97±0.08^{Aax}	71.65±1.30^{Aax}	39.85±0.82^{Aax}
trt-0.72/30	6.10±0.06^{Aay}	0.204±0.003^{Aby}	2.96±0.07^{Aax}	74.27±1.45^{Aax}	40.97±0.18^{Aax}

Data are expressed as means ± standard deviation (n=10); Trt-0 (freeze-dried meat, no treated with ozone)

^{A,B,C} Different capital letters in the same column indicate that means are significantly different (P<0.05) between samples treated with different exposure time of ozone

^{a,b,c} Different lowercase letters in the same column indicate that means are significantly different (P<0.05) between samples treated with different concentration of ozone

^{x,y} Different letters in the same column indicate that means are significantly different (P<0.05) between combined samples and freeze-dried samples.

Likewise, the maximum force was measured to indicate the force required to compress the meat. The maximum force value was approximately 30.42±1.41 N for fresh chicken meat, whereas a significant increase in the force value was observed in the freeze-dried meat (trt-0) 40.05±0.93 N. Similar results were reported by Babíc et al. (2009), who observed an increase in maximum force values for freeze-dried chicken meat when compared with those of fresh meat. The high values of maximum force (N) reported for freeze-dried meats are probably explained by the application of slow freezing. Ciurzyńska and Lenart (2011) justified the change of texture and the final morphological characteristics of freeze-dried products by the growth of the ice crystals formed during slow-freezing process.

In the case of treated samples with ozone and lyophilisation, no significant differences (P>0.05) were found among maximum force values at different concentration levels of ozone. However, the maximum force values were significantly (P<0.05) increased by exposure time, when samples were exposed for a longer time (>30 min) in the treated samples (trt-0.4/60 and trt-0.4/120) compared with the control ones (trt-0). Nevertheless, there were no significant differences between the control (trt-0) and the rest of treated samples. A negative correlation (r =-0.865; P<0.01) between maximum forces and percentage of rehydration was observed, which indicates that when rehydration percentage decreased, maximum force values increased. These results suggest that the increase in maximum forces values of the samples (trt-0.4/60 and trt-0.4/120) may be caused by the lower percentages of rehydration of those samples. Based on these results, the significant decrease in percentages of rehydration and the increase in maximum force values after 60 and 120 min exposure to O_3 imply that ozonation time should be limited to less than 30 min.

Related to changes in color, lightness (L*), redness (a*) and yellowness (b*) values are presented in Table 4.3. The mean L*, a* and b* values of the fresh meat were 43.92±1.85, 2.11±0.09, 6.32±0.21, respectively. Lyophilisation caused a significant increase (P<0.05) in L* (62.45±0.16), a* (2.47±0.06) and b* (13.91±0.05) values. In previous studies, an increase in L*, a* and b*values of freeze-dried meat was observed when compared to raw meat (Babić et al., 2009; Bengtsson & Bengtsson, 1968). The combination of ozone with lyophilisation caused slighter increase in the L* and b* values in most treated samples compared with the non-ozonated control samples (trt-0). Our findings are not in agreement with those of Clavijo (2005), who reported a decrease in L* values in ozonated partially-dehydrated chicken meat. Muhlisin et al. (2015) also observed that exposure to gaseous ozone during 3-day storage did not affect L* and b* values of chicken breast meat.

Table 4. 3. Color parameters (L*, a* and b*) of different treatments in chicken meat fillets

Treatments	L*	a*	b*
Controls			
Fresh meat	43.92±1.85	2.11±0.09	6.32±0.21
trt-0	62.45±0.16^{x}	2.47±0.06^{x}	13.91±0.05^{x}
Combined treatments: freeze-drying and ozonization (trt-[O_3] in ppm/time of exposure in minutes)			
trt-0.4/30	63.36±1.09^{Aax}	2.24±0.08^{Bay}	14.47±0.43^{Aax}
trt-0.4/60	63.50±0.74^{Ax}	2.03±0.03^{Ay}	15.24±0.18^{By}
trt-0.4/120	62.91±0.15^{Ax}	2.43±0.01^{Cx}	13.99±0.17^{Ax}
trt-0.6/10	66.51±0.21^{Aay}	2.55±0.09^{Aax}	13.97±0.23^{Aax}
trt-0.6/30	66.69±0.55^{Aby}	2.56±0.07^{Abx}	14.62±0.37^{Aay}
Trt-0.72/10	68.18±0.22^{Bby}	2.79±0.06^{Bby}	15.36±0.39^{Bby}
trt-0.72/30	66.38±0.41^{Aby}	2.34±0.07^{Aax}	14.41±0.19^{Aax}

Data are expressed as means± standard deviation (n=10); Trt-0 (freeze-dried meat, no treated with ozone)

^{A,B,C} Different capital letters in the same column indicate that means are significantly different (P<0.05) between samples treated with different exposure time of ozone

^{a,b,c} Different lowercase letters in the same column indicate that means are significantly different (P<0.05) between samples treated with different ozone concentrations

^{x,y} Different letters in the same column indicate that means are significantly different (P<0.05) between combined samples and freeze-dried samples

4.3.2. Shelf-life and sensory quality

Microbiological analyses

Changes in microbial populations (TAMB, LAB, and *E.coli*) for frozen chicken meat (FM), freeze-dried (trt-0) and combined treated meat are shown in Table 4.4 throughout eight months of storage. The initial load of the TAMB of the frozen chicken fillets (FM) was about 4.57 log cfu/g in the first month of storage. These counts began to increase in those samples from the 2nd month of storage and exceeded the estimated microbial limit of acceptability (7 log cfu/g) for poultry meat (EC Regulation No. 2073/2005 amended by EC regulation 1086/2011) at the end of storage (7.88 log cfu/g). Whereas, freeze-dried (trt-0) and combined treated samples did not reach this value during the 8 months of storage period (TAMB counts were always less than 5 log units), a significant decrease (P<0.05) of mesophilic bacteria counts was observed during storage time for freeze-dried samples (trt-0) from the second month of storage onwards. The highest reduction in the TAMB counts in those samples was observed in the 6th month of storage, as the initial level of the counts dropped from 4.63 log cfu/g (month 0) to 1.98 log cfu/g on month 6. The combination of ozone and lyophilisation significantly reduced the total aerobic mesophilic bacteria compared with those treated only with lyophilisation (trt-0). This fact may be attributed to the antimicrobial effects of ozone to destroy wide bacterial populations in food (Guzel-Seydim, Greene, & Seydim, 2004). A previous research work carried out by Wu and Doan (2005) showed that ozone (23.09 mg/L) applied for 8 min inactivated 99% of the aerobic bacteria loads on red meat. Muhlisin et al. (2015) reported a reduction about 1.01 log cfu/g and 1.07 log cfu/g in total aerobic and anaerobic bacterial counts, respectively for ozone-treated chicken breast compared to the non-treated samples. Nevertheless, the aerobic mesophilic counts decreased significantly with increase of ozone concentration and exposure time. High ozone concentration of 0.6 ppm or more had a considerable effect to increase the bacterial kill. It is noteworthy that mesophilic counts were significantly lower (P<0.05) in almost all months (0, 2, 4 and 8) with samples treated at 0.6 and 0.72 ppm ozone for 30 min (trt-0,6/30 and trt-0,72/30) compared with samples treated at 0.4 ppm for 30 min (trt-0,4/30).

Moreover, a slight decreasing trend in mesophilic counts was observed when the time of ozonation increased. The number of total aerobic mesophilic bacteria in the treatments trt-0.4/60 and trt-0.4/120 was significantly lower (P<0.05) than in samples treated during 30 min (trt-0.4/30). There were no significant differences between the mesophilic counts of the samples treated with 10 and 30 min. Similar findings were reported by Stivarius et al. (2002), who indicated that the application of 1% ozonated water at 7.2 °C for 15 min diminished all bacterial types compared with those treated for 7 min. At the end of storage, the mesophilic counts were significantly reduced until 6.8 log cfu/g and 3.26 log cfu/g in the ozone treated samples with respect to the control ones, i.e. frozen meat and freeze-dried meat (trt-0), respectively.

Table 4. 4. Microbiological changes (log cfu/g) of treated and untreated samples during eight months of storage

	Month 0	**Month 2**	**Month 4**	**Month 6**	**Month 8**
Treatments	*(A) Total aerobic mesophilic bacteria counts (log cfu/g)*				
FM	4,57±0.05	6,26±0,00	6,78±0,28	7,19±0,25	7,88±0,05
trt-0	4,63±0.06	3,03±0.06	3,45±0.05	1,98±0.03	4,26±0.03
trt-0.4/30	4,69±0.00	3,64±0.06	3,72±0.12	1,35±0.05	3,65±0.05
trt-0.4/60	4,69±0.00	3,10±0.09	3,39±0.09	1,00±0.00	1,81±0.04
trt-0.4/120	4,18±0.11	3,45±0.07	3,40±0.02	1,15±0.05	1,00±0.00
trt-0.6/10	4,63±0.06	3,25±0.05	2,15±0.10	1,89±0.01	3,65±0.04
trt-0.6/30	4,56±0.10	2,96±0.13	3,06±0.20	2,03±0.03	3,71±0.14
trt-0.72/10	4,46±0.03	2,90±0.09	2,96±0.10	2,02±0.04	3,04±0.09
trt-0.72/30	4,09±0.10	2,35±0.02	2,76±0.04	2,44±0.03	2,61±0.00

	(B) Lactic acid counts (log cfu/g)				
FM	3,80±0.14	5,77±0.10	5,38±0.00	5,20±0.24	5,30±0.25
trt-0	4,00±0.12	2,41±0.38	<1	<1	<1
trt-0.4/30	4,54±0.21	2,78±0.05	1,49±0.09	<1	<1
trt-0.4/60	4,54±0.21	2,71±0.02	1,25±0.07	<1	<1
trt-0.4/120	4,49±0.09	2,86±0.03	2,04±0.06	<1	<1
trt-0.6/10	4,31±0.12	2,51±0.05	<1	<1	<1
trt-0.6/30	4,34±0.12	<1	<1	<1	<1
trt-0.72/10	3,82±0.08	2,62±0.09	1,16±0.09	<1	<1
trt-0.72/30	4,26±0.12	<1	<1	<1	<1
	(C) E. coli counts (log cfu/g)				
FM	1,75±0.07	2,44±0.06	2,11±0.00	2,09±0.07	1,00±0.00
trt-0	<1	<1	<1	<1	<1
trt-0.4/30	<1	<1	<1	<1	<1
trt-0.4/60	<1	<1	<1	<1	<1
trt-0.4/120	<1	<1	<1	<1	<1
trt-0.6/10	<1	<1	<1	<1	<1
trt-0.6/30	<1	<1	<1	<1	<1
trt-0.72/10	<1	<1	<1	<1	<1
trt-0.72/30	<1	<1	<1	<1	<1

Data are expressed as means± standard deviation (n=2)

FM: frozen meat; Trt-0 (freeze-dried meat, no treated with ozone); Combined treatment: freeze-drying and ozonization (trt-[O3] in ppm/time of exposure in minutes)

However, the LAB counts in freeze-dried and combined treated samples were significantly ($P<0.05$) higher at the beginning of storage (month 0) than those for the untreated control samples (FM). From the 2nd month of storage, the LAB counts were significantly reduced and reached values less than 1 log cfu/g for combined treated samples (trt-0.6/30 and trt-0.72/30). These results show a strong antimicrobial effect of ozone, as also Kim, Yousef, & Khadre (2003) recently noted. The same authors suggested that gaseous and aqueous ozone, at a low dose and with short contact time is effective against numerous bacteria. Furthermore, ozone concentration seemed to be more effective for the inhibition of LAB in meat samples than contact time. Samples treated with 0.6 and 0.72 ppm of ozone for 30 min had lower LAB counts than those treated with 0.4 ppm ozone for 30 min. More than 4.77-log reduction of LAB counts was observed from the second month of storage in combined treated samples (trt-0.6/30 and trt-0.72/30) when compared with untreated meat samples (FM) and 1.41-log reduction respect to non-ozonated meat (trt-0). In agreement with the present findings, a previous research work performed in our laboratory also showed a positive effect of ozone when applied with partial dehydration, as the growth of LAB was retarded in *Broiler* chicken meat (Clavijo, 2005). Nevertheless, no significant differences ($P>0.05$)

were found between samples treated with 0.6 and 0.72 ppm O_3 along the months of storage. With respect to *E.coli* counts, more than 0.75 log units of *E.coli* were killed at time zero (month 0) in treated samples. Nevertheless, the microbial counts for both untreated and treated samples with ozone did not exceed the Spanish legal limits (EC Regulation No. 2073/2005 amended by EC regulation 1086/2011). Finally, *Salmonella* was not detected in any of the chicken samples.

Sensory analyses

The results of the visual attributes for appearance, percentage surface discoloration, chicken odour and overall impression corresponding to the different treated samples during storage are shown in Figure 4.1.a-d. Frozen chicken meat was significantly scored (P<0.05) highly for appearance (Figure 4.1a) and percentage surface discoloration (Figure 4.1b) compared to other treated samples. The limit of acceptability for appearance was reached after 4 months for the freeze-dried meat (the score obtained was lower than 4), which means the end of its shelf-life. In contrast, all samples treated with ozone remained acceptable for the panellists until the end of the storage (month 8). Concerning the percentage of discoloration, panellists gave similar scores for all treated samples. Statistical analyses did not show significant differences (P>0.05) between frozen meat and all treated samples from the 6^{th} month of storage and reached average percentages of 40-59%. In previous studies, Stivarius et al. (2002) used the same scale and reported lower percentages of discoloration between 20 and 39% in beef trimmings treated with ozone, compared to the results of our study.

Related to chicken odour (Figure 4.1c), frozen meat had the highest score (P<0.05) of chicken odour in most of the months. At the end of storage, (months 6-8), samples treated with ozone concentration higher than 0.4 ppm had lower odour alteration and kept an acceptable chicken odour after 8 months of storage. On the contrary, the limit of acceptability of odour was reached from the 4^{th} month for the non-ozonated freeze-dried and 0.4 ppm-ozonated samples. Regarding the odour characteristics (results not shown), samples treated with lyophilisation (trt-0) and also with ozone maintained a score of 7 during the whole storage period. However, in the case of frozen samples, a slight perceptible odour was detected from the sixth month of storage, maybe caused by their higher microbial load (i.e. total aerobic mesophilic bacteria). These results are in agreement with those of Manousaridis et al. (2005) who reported better scores for odour attributes of ozone-treated shucked mussels (O_3/90 min) when compared with the control ones. Similarly, Stivarius et al. (2002) found that the use of ozone in ground beef production process can be effective for reducing microbial pathogens with minimal effects on odour characteristics.

Likewise, based on the overall acceptability (Fig 4.1d), a significant decrease (*P*<0.05) in average scores was observed during storage for all treated samples. The combined use of ozone and lyophilisation resulted in better acceptability of samples during all months. However, the frozen (FM), the freeze-dried (trt-0) and the combined treated samples at 0.4 ppm (trt-0.4/30 and trt-0.4/60) were not considered acceptable for panellists at the end of the storage period.

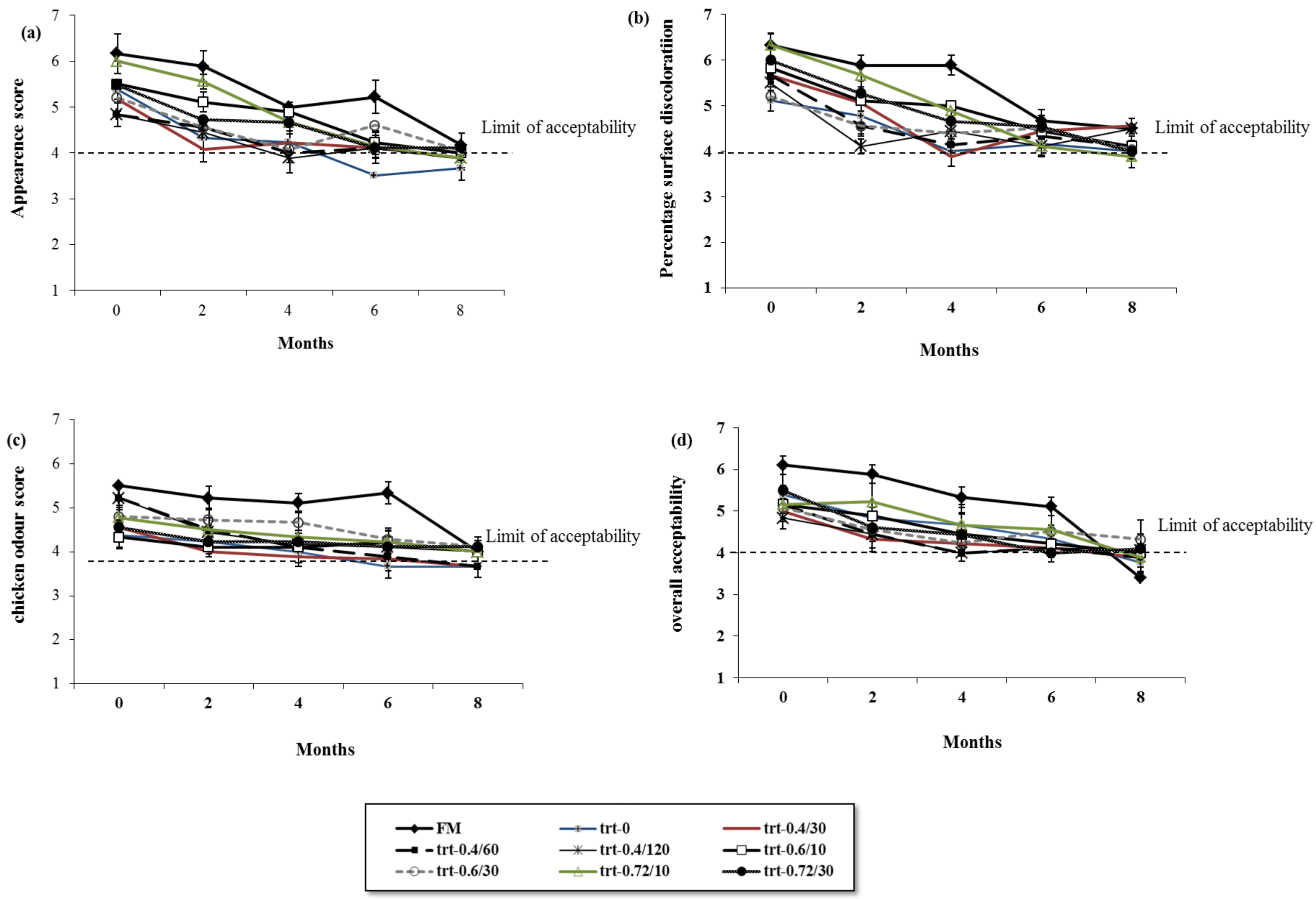

Figure 4. 1. Changes in the visual attributes for appearance (a), percentage surface discoloration (b), chicken odour (c) and overall acceptability (d) of chicken freeze-dried meat treated with different time and concentration of ozone vs control (freeze-dried meat with no ozone) (trt-0) and fresh meat (FM) during 8 months. Error bars represent standard deviation (n=18). (4=limit of acceptability)

The results of the texture profile attributes (hardness, juiciness and chewiness) evaluation of the different treated samples are presented in Figures. 4.2e-g. During the storage period, the hardness and chewiness scores of all samples decreased gradually. The samples treated with 60 and 120 min of ozone (trt-0.4/60 and trt-0.4/120) were considered unacceptable from the 2nd month of storage (scored below 4), while samples of treatment trt-0.6/10 were above the limit of acceptability throughout the whole storage period. Our results suggest that the combination of ozone and lyophilisation was fairly successful in maintaining acceptable scores of hardness and chewiness up to 8 months of storage. The freeze-dried samples (tr-0) scored under the limit before month 6 of storage.

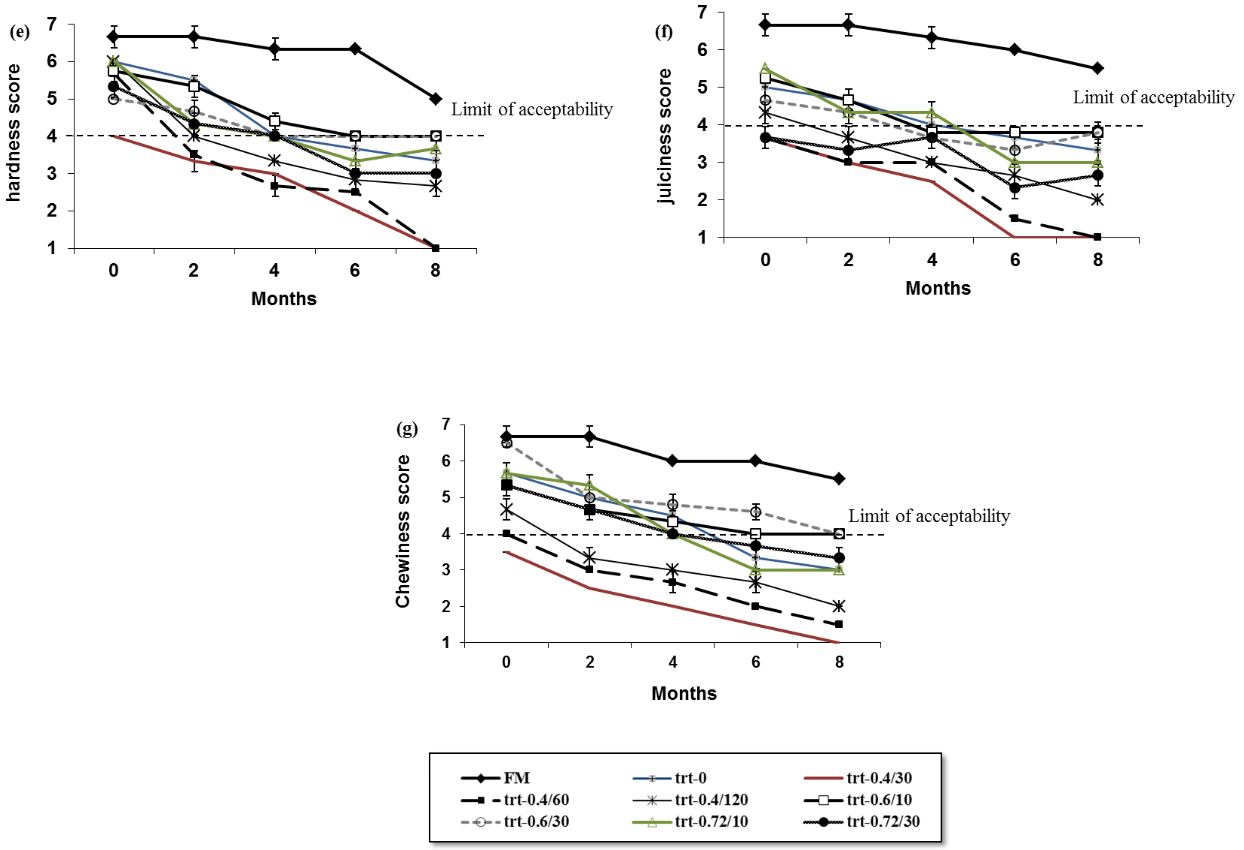

Figure 4. 2. Changes in the texture profile attributes for hardness (e), juiciness (f) and chewiness (g) of chicken freeze-dried meat treated with different time and concentration of ozone vs control (freeze-dried meat with no ozone) (trt-0) and fresh meat (FM) during 8 months. Error bars represent standard deviation (n=18), (4=limit of acceptability)

Relating to the juiciness attribute, frozen meat samples (FM) registered higher scores than the other treated samples throughout the months of storage, which indicated a moderately juicy meat (scores between 6.5 and 5.5) in the case of frozen samples. Also, samples of treatment trt-0.6/10 were acceptable throughout the whole storage period. On the contrary, the worst-scored samples were those treated with 0.4 ppm O_3. Moreover, the scores for juiciness decreased significantly (P<0.05) over the storage period in all treated samples. Lawrie (1998) suggests that the lyophilisation process determines some loss of juiciness in freeze-dried meat products. Furthermore, Casp and Abril (1999) reported that freeze-dried products stored in unfavourable conditions, are susceptible to all physical and chemical changes, as well as product oxidation which causes undesirable organoleptic characteristics. Therefore, a suitable packaging would be necessary for retaining the majority of their physical, chemical and sensorial proprieties of dried meats.

4.4. Conclusions

The combination of ozone (0.6 ppm for 10 min) and lyophilisation would be useful in enhancing the microbial properties of meat, in achieving a sensory acceptable product, as well as, in extending the shelf-life of raw chicken breast meat up to 8 months. On the contrary, the samples treated with lyophilisation alone had a shelf-life of only 4 months. Likewise, the 0.4 ppm exposure to ozone had a negative effect on increasing both the hardness and chewiness of chicken meat. Further research work would be needed to determine the optimum conditions of modified atmosphere packaging (MAP) for maximizing the shelf-life extension of ozonated freeze-dried chicken meat.

4.5. References

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CHAPTER V. Study of modified atmosphere packaging on the quality of ozonated freeze-dried chicken meat

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Chapter V. Study of modified atmosphere packaging on the quality of ozonated freeze-dried chicken meat

5.1. Introduction

Poultry meats are widely consumed freshly in Europe, but in fact they are highly perishable to bacterial contaminants due to their composition, a high water activity (a_w), and a high final pH, limiting the shelf-life of the products. Spoilage of fresh poultry products is an economic burden to the producer (Petrou, Tsiraki, Giatrakou, & Savvaidis, 2012); so, developing effective hurdle technologies to extend the shelf-life and to keep the product quality during long periods represents a major task for the poultry processing industry. According to Cantalejo et al.(2016), the combined effect of gaseous ozone and lyophilisation in chicken breast meat showed great antimicrobial effectiveness, due to the action of ozone, as well as the low percentage of humidity (<10%) and water activity below 0.5 of the product. These techniques also allowed extending the shelf-life of those products during 8 months of storage at room temperature without refrigeration.

However, the combination of those hurdles were not sufficient to maintain the physicochemical (texture) and sensory qualities of the ozonated dried meat for long time (Zouaghi, 2011). In fact, the loss of textural qualities (i.e., tenderness and juiciness) was the main problem in freeze-dried meats, maybe due to denaturizing of proteins, followed by their aggregation (Babić, Cantalejo, & Arroqui, 2009). Hence, as a result of an increasing demand for healthy and high-quality products, a need emerged for further research work involving the possibility of maintaining better sensory quality of ozonated freeze-dried chicken meat to reach more potential markets and satisfy consumer demands, hardness and juiciness being some of the main criteria influencing consumer´s acceptability (Ganhão, Morcuende, & Estévez, 2010). According to Babić et al. (2009), the freeze-dried meat products which have been adequately packaged can be stored for unlimited periods retaining the majority of their physical, chemical, biological and sensory properties as in the fresh state.

In this context, modified atmosphere packaging (MAP) has been considered in this study as a useful technique to maintain the sensory quality and to extend the shelf-life of several foods commodities, including chicken meat (Chouliara, Karatapanis, Savvaidis,

& Kontominas, 2007). García-Esteban et al., (2004) stated that modified atmosphere packaging preserved meat (i.e. dry-cured ham) from hardening and deterioration of textural properties more efficiently than vacuum packaging. The principle of MAP is the replacement of the atmosphere surrounding a product before sealing, carbon dioxide, oxygen and nitrogen, being the most commonly used gases. Carbon dioxide possesses bacteriostatic activity (Nair, Kiess, Nannapaneni, Schilling, & Sharma, 2015). Oxygen is important to retain meat color and nitrogen results essential to avoid oxidation of fats and pack collapse. These gases can be applied individually or in combination, in order to achieve an optimum effect, depending on the specific needs of the particular food products being preserved (Narasimha Rao & Sachindra, 2002). Therefore, the aim of this study was to evaluate the effects of MAP conditions on the physicochemical and sensory properties of ozonated freeze-dried chicken meat stored at room temperature, in order to develop new high-quality raw meat products from fresh chicken meat, safe, with a high nutritional value, with no additives added and stable over time at room temperature. Also, the new raw products from fresh poultry meat represent an alternative, as they would allow a length in the retail period in the case of natural catastrophes, military campaigns, export to third countries, scarcity in electricity supply, etc. This is the first time that these three combined techniques (ozonation, freeze-drying and MAP) have been applied on poultry meats.

5.2. Materials and methods

5.2.1. Samples preparation

Broiler chicken breast meat was provided by U.V.E., S.A. Company (Tudela, Navarre, Spain). Chickens were 42 days old before slaughtering with approximately 2 kg of weight. All breasts were stored in a refrigerated room (2-4 °C) for the time of reception until used. The initial load of total aerobic mesophilic bacteria (TAMB) (b5 log cfu/g), lactic acid bacteria (LAB) (<4 log cfu/g), *Escherichia coli* (<2 log cfu/g) and *Salmonella* spp. (not detected in any of the chicken samples) was determined before samples were processed. The samples were trimmed of visible fat and nerves; they were cut into pieces (approximately 3 x 3 cm^2 of section and of 0.7 cm in thickness). Then, they were divided into two trials: the first trial was vacuum packaging, deep frozen and stored at -40 ± 1 °C (Climas, Barcelona, Spain) and used as an external control of raw meat (untreated samples) for physical-chemical measurements (pH, color, and texture) and sensory analyses and to characterize the raw material. The second trial was subjected to a combined treatment of gaseous ozone, freeze-drying and modified atmosphere packaging (MAP) as described below.

5.2.2. Ozone treatments

After having prepared the breast samples, they were treated first with ozone. Ozonation assays were carried out in a 3 m^3 volume refrigerated chamber (Eurozon, Ecologyc 2000, Sestao, Vizcaya, Spain) to a continuous flow of ozone gas at 4 ± 0.5 °C and 90 ± 1 % relative humidity. These conditions are important for the efficiency of the bactericidal effect of ozone (Kim, Yousef, & Chism, 1999). Ozone in form of gas was generated *in situ* utilizing a UV radiation using an ozone generator (Rilize, model 3060, Eurozon, Sestao, Spain). Ozone concentrations inside the chamber were monitored continuously by circulating air from the chamber through an ultraviolet absorption ozone gas analyzer (Ozomat MP, Anseros, Germany). The conditions of ozonation were described by Zouaghi (2011) for *Broiler* chicken meat and were the same in all treatments, where the samples were exposed to gaseous ozone for 10 minutes with a dose of 0.6 ppm to reduce the initial levels of contamination (a reduction about 1.1 log cfu/g was observed in TAMB, LAB and *E. coli*, *Salmonella* spp. was not detected in any of the chicken samples).

5.2.3. Freeze-drying process

After ozone treatments, samples were dehydrated in a pilot scale freeze-dryer (Model Lyobeta 25, Telstar Industrial, S.L., Barcelona, Spain). The different parameters of the freeze-drying process assayed in this study were the same in all treatments and were the best conditions described in the research work of Babić et al. (2009). The initial a_w and moisture content of fresh chicken meat were about 0.984 ± 0.002 and 73.88 ± 0.06%, respectively. After lyophilisation, a significant decrease ($P < 0.05$) in those values was observed for the samples treated with lyophilisation (0.131 ± 0.002 for a_w and 2.93 ± 0.06% for humidity).

5.2.4. Packaging

After ozone and freeze-drying treatment, all samples were individually packaged in low-O2-permeable polystyrene/ ethylvinylalcohol (EVOH)/ polyethylene (PE) trays and heat-sealed using a low O2-permeable cling film consisting of polyethylene terephthalate (PET)/ EVOH/ Polypropylene (PP) on the inside of the outer layer as a gas barrier, supplied by Ilpra Systems, S.L. (Barcelona, Spain). The trays had an oxygen transfer rate of less than 50 cm^3 m^{-2} d^{-1} bar^{-1}, permeability to CO_2 less than 150 cm^3 m^{-2} d^{-1} bar^{-1} and a water vapour permeability of less than 2.8 g m^{-2} d^{-1}. Samples were packaged using a packaging machine (Ilpra Termosaldatrici, España) with a sample/gas ratio of 1:3 (w/v). The untreated samples (frozen meat) were vacuum packed in impermeable plastic trays (type PA/ PE 20/70 200 x 300) using a vacuum packaging machine (Model SAMMIC V-640, Gipuzkoa, Spain).

5.2.5. Modified atmosphere packaging experiments

In the present study, three different trials were carried out to evaluate the effect of modified packaging on the physicochemical and the sensory changes of ozonated dried chicken meat stored at different packaging atmosphere conditions, in order to choose the most suitable packaging conditions. The modified atmosphere gas conditions assayed are listed in Table 5.1.

Table 5. 1. Experiments and different conditions used for ozonated freeze-dried chicken meat in modified atmosphere packaging.

Trials	Experiments	Packaging conditions		
		O_2 (%)	CO_2 (%)	N_2 (%)
Trial i	1	20	-	80
	2	30	-	70
	3	0	-	100
Trial ii	1	-	20	80
	2	-	30	70
	3	-	40	60
	4	-	50	50
Trial iii	1	10	30	60
	2	20	30	50
	3	30	30	40
	4	20	20	60
	5	30	20	50
	6	40	20	40
	7	20	10	70

The first set of trials (i) consisted of three experiments in which meat was packaged with three different oxygen concentrations (0, 20 and 30 %O_2). This trial was planned in order to examine the influence of the effect of O_2 levels on the quality of MAP ozonated dried chicken. In the second trial (ii), the samples were packaged with four levels of CO_2 (20, 30, 40 and 50 %CO_2), in order to evaluate the effect of carbon dioxide concentration on the quality of MAP ozonated dried chicken. Based on the results of trail (i) and trial (ii), the third trial (iii) was designed by using seven different compositions of $O_2/CO_2/N_2$ mixture. The concentrations of O_2, CO_2 and N_2 varied from one treatment to another, in order to determine the best $O_2:CO_2$ ratio needed to maintain the quality of ozonated freeze-dried chicken meat during 28 days of storage.

5.2.6. Storage conditions

After packaging, samples were coded and stored in a dark place at room temperature (21 ± 1 °C) for 28 days. The untreated samples (frozen meat) were kept at -40 °C until analyses. All samples were analysed on days 1, 7, 15, 21 and 28 for physicochemical and sensory analyses. The 28 day period was the time allotted in order to verify the effectiveness of each MAP treatment where the degree of possible changes in hardness and juiciness was measured, because in preliminary studies they were the most affected by freeze-drying. A comparative study of those parameters of both the original fresh chicken meat and the treated meat was undergone.

5.2.7. Physicochemical analyses

Headspace gas composition

The concentrations of O_2, CO_2 and N_2 inside the trays (three from each experiment) were measured using a gas analyser (Gas-space Systech Instruments, S.A, Madrid, Spain) every day before meat analyses Gas analyses were performed by piercing a syringe needle through a rubber septum glued on the surface of the plastic film. Three measurements were carried out for each tray.

pH, color and texture profile analyses (TPA)

The pH was measured using a pH meter (Crison PH 25, S.A, Barcelona, Spain) with a combined electrode which penetrated the meat samples. The pH meter was calibrated with pH 4 and pH 7 standard solutions.Color measurements were performed using a Minolta Chrome Meter CM-2500d (Minolta Co. Ltd. Osaka, Japan), with specular reflectance excluded, 8 mm diameter measuring aperture and D65 illuminator at 10º standard observer angle. Color coordinates obtained in the CIELAB space with specular component included L* (lightness), a* (redness) and b* (yellowness). The total difference for two color measurements is given by the following formula (Chouhan, Pal, & Rao, 2015): $\Delta E^*_{ab} = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2}$ where ΔL*, Δa* an Δb* are the difference in the L*, a*, and b* measured at day 28 of storage and their values at day 0.

Texture profile analyses (TPA) were performed with a TA.XT Plus Texture Analyser (Stable Micro Systems Ltd, Aname S.L, England). Ten samples from each experiment were taken parallel to the longitudinal orientation of the muscular fibres. Prior to the analyses, samples were packaged in impermeable plastic bags and cooked in a water bath at 80 ^{o}C for 2 min. The samples were compressed perpendicularly to muscle fibre orientation to 70% original height through a two consecutive cycles, with 3 s between cycles, using a cylindrical probe of 12.8 mm diameter. The crosshead moved at a constant speed of 2 mm/s. The following texture profile parameters were determined as described by Bourne (1978) and Szczesniak (1995): hardness (N) maximum force required to compress the sample, cohesiveness (dimensionless), extent to which the sample could be deformed before it ruptures, and chewiness (N/mm), calculated as the product of hardness, springiness and cohesiveness (Meral & Mahmut, 2016; Savadkoohi, Hoogenkamp, Shamsi, & Farahnaky, 2014).

Treated samples had to be rehydrated and cooked in order to be analyzed. The duration of rehydration process was fixed in 3 h, as after that time period there was no more absorption of water by the samples (Babić et al., 2009)

5.2.8. Sensory descriptive analyses

The descriptive sensory evaluation was performed by 6 trained panelists in two sessions: the first one, to visually evaluate the attributes of the rehydrated chicken meat, and the second one to evaluate all the texture profile attributes of the rehydrated-cooked treated chicken meat. Each sample was served in white plates and shown with three random numbers. The method of Hunt et al. (1991) was adapted to describe the sensory characteristics of the rehydrated treated chicken meat in five attributes: appearance, percentage of surface discoloration, chicken odour, odour characteristics and overall impression. For the evaluation of the texture profile attributes, the panel evaluated the rehydrated-cooked treated chicken meat (3 cube-shaped samples per panelist) for the three following sensory attributes: hardness, juiciness and chewiness (Lyon & Lyon, 1990). Each attribute was rated on a seven-point scale, with score 1 equivalent to the lowest score and 7 indicates the highest score. In both evaluations, visual and textural, the limit of acceptability was 4.

5.2.9. Statistical analyses

An analysis of variance (ANOVA) using the General Linear Model (GLM) procedure of the SPSS package (SPSS 21, Chicago, IL, USA) was performed, in which the measured variables were set as dependent variables, treatments and storage time were assigned as fixed effects and replicates were assigned as random effects. The pairwise differences between least-square means were evaluated by Tukey's HSD test. Differences were considered significant when $P< 0.05$. The values were given in terms of mean values and standard errors in tables and figures. Correlations among variables were determined by correlation analyses using Pearson´s linear correlation coefficient with the above-mentioned software package.

5.3. Results and discussion

5.3.1. Trial i. Packaging under different oxygen concentrations (0, 20 and 30 %)

Headspace composition

A small but statistically significant ($P< 0.05$) change in gas composition of each package was recorded from day 15 onwards (data not shown). In fact, the O_2 concentration inside the trays showed a small decrease in both atmospheres (0 %O_2/ 100 %N_2 and 30 %O_2/ 70 %N_2) from initial values of 0.57 ± 0.01 % and 29.41 ± 0.03 % to final values of 0.50 ± 0.00 % and 28.25 ± 0.03 % respectively. This was caused by meat enzyme respiration (Rossaint, Klausmann, & Kreyenschmidt, 2014).

pH and color change

The evolution of pH and color parameters values (L*, a* and b*) during storage in different packs is summarized in Table 5.2. The pH values for frozen meat varied between 5.66 ± 0.04 and 5.92 ± 0.02, whereas those of treated samples ranged between 5.67 ± 0.03 and 6.02 ± 0.04. These values of pH were lower than those reported by Cantalejo et al. (2016) in chicken meat treated with ozone and lyophilisation. However, the pH values generally increased with time in all samples with significant differences ($P< 0.05$) on day 28 in samples under 20 %O_2/ 80 %N_2 packaging conditions (and on day 15 under MAP with 30 %O_2/ 70 %N_2). Samples packaged under high O_2 MAP conditions (30 %O_2/ 70 %N_2) showed the highest ($P< 0.05$) pH values from day 15 onwards with respect to that of the others treatments. Fernández-López et al. (2008) also observed differences in pH values due to storage time and conditions, in ostrich steaks of initial pH 6.04.

Regarding color parameters, meat stored under MAP with 30 $\%O_2$/ 70 $\%N_2$ showed significant change in L* value and developed a darker appearance till 28 days. Both gas composition and storage period had a significant effect on the a* values (redness) of chicken meat samples ($P< 0.05$). In the first day of storage, significant differences were observed among samples, the lowest a* values being obtained in samples packed in 0 $\%O_2$/ 100 $\%N_2$. From day 15, the redness value increased slightly ($P< 0.05$) in 0 $\%O_2$/ 100 $\%N_2$ packs and decreased significantly ($P< 0.05$) for O_2 packaged meat samples up to 28 days. Several authors reported decreases of redness for high O_2 packaged chicken (Keokamnerd, Acton, Han, & Dawson, 2008) and ostrich meat (Fernández-López et al., 2008; Seydim, Acton, Hall, & Dawson, 2006) during refrigerated storage. The same authors indicated this loss of redness due to oxidation of myoglobin to metmyoglobin in packaged meat. The parameter b* values related to yellowness changed significantly ($P< 0.05$) over time and among the packaging conditions. In N_2 packs, b* value increased progressively during storage, while, in O_2 MAP conditions, b* values decreased ($P< 0.05$) from day 15 onwards. Esmer et al. (2011) stated the loss of redness in meat and the alteration of its color to brownish red by formation of metmyoglobin that leads to the decrease in the b* value. Further statistical analyses of ΔE^* values showed that the samples packed in O_2 MAP conditions reflected the large color change (ΔE values of 3.7 and 4.4 for 20 $\%O_2$ and 30 $\%O_2$, respectively) during storage comparted to 0 $\%O_2$/ 100 $\%N_2$ packs ($\Delta E= 1.1$).

Table 5. 2. Changes in pH, L*, a* and b* values of chicken breast meat stored under different MAP (0 %O_2/ 100 %N_2, 20 %O_2/ 80 %N_2 and 30 %O_2/ 70 %N_2) during 28 days of storage at room temperature.

Parameters	Days of storage	Treatments			
pH		Frozen meat (FM)	0 %O_2/ 100 %N_2	20 %O_2/ 80 %N_2	30 %O_2/ 70 %N_2
	1	5.80 ± 0.02	5.71 ± 0.04^{Aa}	5.62 ± 0.04^{Aa}	5.73 ± 0.02^{Aa}
	7	5.92 ± 0.02	5.74 ± 0.02^{Aa}	5.67 ± 0.03^{Aa}	5.77 ± 0.03^{Aa}
	15	5.89 ± 0.02	5.70 ± 0.02^{Aa}	5.69 ± 0.03^{Aab}	5.95 ± 0.03^{Bb}
	21	5.75 ± 0.01	5.68 ± 0.03^{Aa}	5.70 ± 0.02^{Aab}	5.97 ± 0.03^{Bb}
	28	5.66 ± 0.04	5.73 ± 0.04^{Aa}	5.80 ± 0.03^{Ab}	6.02 ± 0.04^{Bb}
L*					
	1	43.84 ± 0.34	66,37 ± 1.13^{Aa}	64.23 ± 0.53^{Aa}	64.02 ± 0.77^{Aa}
	7	49.83 ±0.59	65,53 ± 0.87^{Aa}	65.27 ± 0.85^{Aa}	64.46 ± 0.83^{Aab}
	15	44.76 ± 0.27	65,67 ± 1.12^{Aa}	65.88 ± 0.32^{Aa}	67.36 ± 0.59^{Ab}
	21	43.97 ± 0.37	63,69 ± 0.81^{Aa}	65.24 ± 0.81^{Aa}	63.75 ± 0.55^{Aa}
	28	43.68 ± 0.50	67.23 ± 1.13^{Ba}	66.73 ±0.50^{Ba}	61.48 ± 0.67^{Aa}
a*					
	1	1.11 ± 0.00	1.37 ± 0.01^{Aa}	2.55 ± 0.02^{Cd}	2.38 ± 0.02^{Bf}
	7	1.14 ± 0.00	1.41 ± 0.02^{Aa}	1.62 ± 0.01^{Bc}	1.84 ± 0.02^{Cd}
	15	1.05 ± 0.00	1.60 ± 0.03^{Ab}	1.62 ± 0.03^{Ac}	1.62 ± 0.03^{Ac}
	21	1.04 ± 0.00	1.72 ± 0.03^{Cc}	1.28 ± 0.02^{Ab}	1.50 ± 0.02^{Bb}
	28	1.17 ± 0.01	1.84 ± 0.02^{Cd}	1.19 ± 0.02^{Aa}	1.27 ± 0.02^{Ba}
b*					
	1	8.09 ± 0.09	13.72 ± 0.10^{Aa}	17.42 ± 0.21^{Bc}	16.70 ± 0.21^{Bc}
	7	7.89 ± 0.05	15.13 ± 0.18^{Abc}	15.56 ± 0.20^{Ab}	17.58 ± 0.23^{Bd}
	15	11.67 ± 0.05	15.43 ± 0.12^{Cc}	14.60 ± 0.15^{Ba}	13.42 ± 0.09^{Aa}
	21	10.96 ± 0.10	17.08 ± 0.27^{Bd}	17.35 ± 0.20^{Bc}	15.08 ± 0.19^{Ab}
	28	9.51 ± 0.10	14.26 ± 0.26^{Bab}	15.06 ± 0.21^{Cab}	13.24 ± 0.16^{Aa}

Data are expressed as means±standard error (n=10). MAP samples were previously ozonated and freeze-dried.

^{A,B,C} Different capital letters in the same raw indicate that means are significantly different (P<0.05) between the different MAP packaging.

^{a,b,c} Different lowercase letters in the same column indicate that means are significantly different (P<0.05) between days of storage

Texture analyses

Table 5.3 shows the effect of packaging conditions and storage time on texture parameters measured instrumentally. Treated samples showed significantly (P< 0.05) higher textural parameters values compared to untreated meat samples (frozen meat). Similar results were reported by Cantalejo et al. (2016) who found that chicken meat treated with ozone and freeze-drying was tougher when compared with the control meat. During storage, increased hardness and chewiness values and reduced cohesiveness values were obtained for all samples.

Hardness and chewiness showed higher values in samples packed under high oxygen concentrations (30 %) throughout the storage time compared to samples packaged under low concentrations of O_2 (0 and 20 %). The increase of hardness and other related texture parameters are highly undesirable, as this could have a great impact on consumer acceptability (Ganhão et al., 2010).

Table 5. 3. TPA parameters of the chicken meat packed with different MAP (0 %O_2/ 100 %N_2, 20 %O_2/ 80 %N_2 and 30 %O_2/ 70 %N_2) during 28 days of storage

Parameters	Days of storage	Frozen meat (FM)	MAP condition		
			0 %O_2/ 100 %N_2	20 %O_2/ 80 %N_2	30 %O_2/ 70 %N_2
Hardness (N)	1	34.32 ± 0.44	45.66 ± 0.59^{Ba}	45.10 ± 0.33^{Ba}	39.16 ± 0.35^{Aa}
	7	33.44 ± 0.35	47.67 ± 0.52^{Bab}	51.69 ± 0.49^{Cb}	45.49 ± 0.60^{Ab}
	15	31.60 ± 0.25	49.68 ± 0.60^{Bb}	52.08 ± 0.72^{Cb}	47.84 ± 0.31^{Ac}
	21	30.78 ± 0.34	52.74 ± 0.20^{Bc}	56.50 ± 0.42^{Cc}	49.93 ± 0.57^{Ad}
	28	35.80 ± 0.09	58.19 ± 0.96^{Bd}	58.59 ± 1.13^{Bc}	50.55 ± 0.37^{Ad}
Cohesiveness					
	1	0.43 ± 0.01	0.54± 0.00^{Ae}	0.59 ± 0.00^{Ce}	0.58 ± 0.00^{Be}
	7	0.41± 0.01	0.52 ± 0.00^{Ac}	0.57 ± 0.00^{Cd}	0.55± 0.00^{Bd}
	15	0.42 ± 0.01	0.59 ± 0.00^{Cd}	0.56 ± 0.00^{Bc}	0.52 ± 0.00^{Ab}
	21	0.34 ± 0.00	0.51 ± 0.00^{Ab}	0.54 ± 0.00^{Cb}	0.53 ± 0.00^{Bc}
	28	0.44 ± 0.01	0.48± 0.00^{Aa}	0.52 ± 0.00^{Ca}	0.51 ± 0.00^{Ba}
Chewiness (N/mm)	1	14.22 ± 0.15	22.83 ± 0.26^{Cb}	21.94 ± 0.22^{Ba}	18.39 ± 0.15^{Aa}
	7	11.97 ± 0.14	21.37 ± 0.16^{Aa}	26.91 ± 0.17^{Cb}	24.80 ± 0.26^{Bc}
	15	12.28 ± 0.16	26.94 ± 0.51^{Bd}	27.69 ± 0.60^{Bb}	23.46 ± 0.18^{Ab}
	21	9.05 ± 0.01	24.61 ± 0.17^{Bc}	27.17 ± 0.27^{Cb}	23.46 ± 0.31^{Ab}
	28	14.46 ± 0.11	32.14 ± 0.34^{Bf}	31.60 ± 0.13^{Bc}	26.60 ± 0.28^{Ad}

TPA: Texture profile texture (n=15). MAP samples were previously ozonated and freeze-dried.

^{A,B,C} Different capital letters in the same row indicate that means are significantly different (*P*<0.05) between the different MAP packaging

$^{.a,b,c}$ Different lowercase letters in the same column indicate that means are significantly different (*P*<0.05) between days of storage

Sensory descriptive analyses

Results of sensory analyses for appearance, hardness and juiciness are represented in Figure 5.1. The appearance attribute, on high oxygen packaged samples (30 %O2/ 70 %N_2) was less scored (*P*< 0.05) by panelists than the other groups during the first days. Significant differences were no longer observed after day 15 for these three atmospheres-treated samples.

For hardness and juiciness attributes, significant differences were not observed between samples by day 15. TPA hardness showed a small but significant correlation with respect to sensory hardness (r=-0.273; *P*<0.05) and juiciness (r=-0.266; *P*<0.05) attributes.

High oxygen packaged samples (30 $\%O_2$/ 70 $\%N_2$) received better scores (between 5.5 and 6) by panelists than others treated samples (scores below 5) at day 28. These results did not agree with those of Jongberg, Wen, Tørngren, & Lund (2014), who found that chicken muscle stored in high-oxygen atmosphere packaging scored lower in tenderness compared to breasts stored in non-oxygen atmosphere.

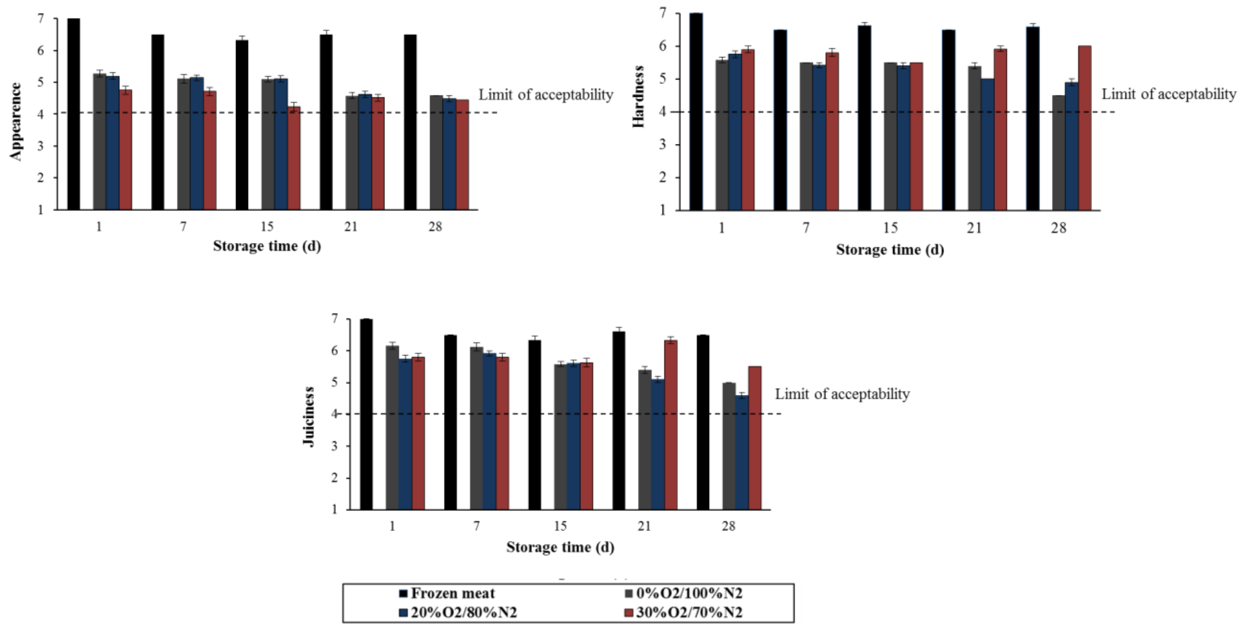

Figure 5. 1. Sensory evaluation of hardness and juiciness on ozonized dried chicken meat stored under different O_2 concentration and 0 $\%O_2$/ 100 $\%N_2$ during storage time in days. Means with standard errors (n = 18) are shown. Appearance score: 1= very different from that of fresh chicken meat, 7= very similar to that of fresh meat; Hardness score: 1=very hard, 7=very tender; Juiciness score: 1=very dry, 7= very juicy (4=limit of acceptability).

In general, the scores of all attributes were always above the limit of acceptability (score of 4) in the three types of packages during the storage period. During all storage period, no undesirable odour, flavor or discoloration due to lipid oxidation and non-enzymatic browning were observed by the panelists in the sensory evaluation among all samples. The scores found in our study were higher than those observed by Zouaghi, (2011) using the same scale on chicken samples treated with a combination of ozone, lyophilisation and vacuum-packaging. This suggests that ozonated & dried chicken meat samples packaged under modified atmospheres maintained desirable sensory characteristics better than those vacuum packaged samples.

5.3.2. Trial ii. Packaging with different CO_2 concentration (20, 30, 40 and 50 %)

Headspace gas composition

A decrease in CO_2 and an increase in N_2 levels were observed in all samples during the storage (data not shown). The CO_2 percentage on day 28 showed a significant decrease, the maximum CO_2 reduction being in 50 % high-CO_2 packages (around 5 %). Vergara, Berruga, & Linares (2005) found greater reduction of CO_2 levels than those observed in our study. They found around 10 %CO_2 reduction on rabbit meat packed with 40 %CO_2/ 60 %N_2 over 20 days of storage. The decrease of CO_2 inside the packages could be caused by the absorption of carbon dioxide in meat (Jakobsen & Bertelsen, 2002).

pH and Color change

The samples packaged with high CO_2 concentrations (50 %CO_2/ 50 %N_2) had significantly lower pH values than those with lower percentages of CO_2 throughout the storage period (Table 5.4). Vergara et al. (2005) also reported lower pH values in the range of 5.55-5.89 for rabbit meat packaged under high CO_2 MAP conditions during 20 days of storage. Likewise, the pH values of control and all treated samples slightly decreased during storage, but statistically significant differences ($P < 0.05$) were observed only after 15 days of storage in the case of samples packaged with 30 %CO_2 and 40 %CO_2. This reduction in the pH values could be due to the dissolution of CO_2 in the product (Lerasle et al., 2014).

Regardless color parameters, lightness (L*) values remained more or less stable with storage time for different modified atmosphere packs. The redness values decreased ($P < 0.05$) with time for all treatments, this decrease being more pronounced for high CO_2 MAP (40 and 50 %) packaged chicken samples than other MAP atmospheres. According to our results, Vergara et al. (2005) also reported color changes of rabbit meat, that are more relevant and happen more quickly with high concentrations of CO_2. This decrease in a* values may be associated with the oxidation of myoglobin and formation of metmyoglobin (Fernández-López et al., 2008). Finally, the b* values decreased significantly ($P < 0.05$) in all treatments. By day 28, the yellowness of meat packed with 50 %CO_2 was lower than that in other packaged samples. Significant differences ($P < 0.05$) were observed in ΔE values with respect to storage time for different modified atmosphere packs. According to Chouhan et al., (2015), the total color difference (ΔE*) values between 3.0 and 6.0 can be considerate as very distinct color difference 6.0 to 12.0 indicates a great visual change and the values higher at 12 for very great difference. According to this scale, there was large color changes in samples packed with 20 and 40% CO_2 (ΔE^* values of 9.8 and 7.8, respectively), while a very distinct color differences were obtained for samples stored at 30 and 50% CO_2 (ΔE^* values of 5.7 and 5.5, respectively).

Table 5. 4. Changes in pH, L*, a* and b* values of chicken meat stored in different modified atmosphere packs of CO_2 at room temperature during 28 days of storage

Parameters	Days of storage	Frozen meat (FM)	MAP condition			
			20 %CO_2/ 80 %N_2	30 %CO_2/ 70 %N_2	40 %CO_2/ 60 %N_2	50 %CO_2/ 50 %N_2
pH	1	6.03 ± 0.04	6.03 ± 0.04^{BCa}	6.15 ± 0.03^{Cc}	5.98 ± 0.03^{Bb}	5.74 ± 0.02^{Aa}
	7	6.00 ± 0.01	6.00 ± 0.03^{BCa}	6.12 ± 0.04^{Cbc}	5.91 ± 0.04^{Bab}	5.73 ± 0.04^{Aa}
	15	6.05 ± 0.03	5.97 ± 0.03^{Ba}	6.01 ± 0.03^{Bab}	5.88 ± 0.02^{Ba}	5.71 ± 0.04^{Aa}
	21	5.99 ± 0.02	5.95 ± 0.06^{Ba}	5.99 ± 0.03^{Bab}	5.87 ± 0.04^{Ba}	5.70 ± 0.02^{Aa}
	28	5.98 ± 0.03	5.86 ± 0.03^{BCa}	5.93 ± 0.02^{Ca}	5.79 ± 0.02^{Ba}	5.68 ± 0.03^{Aa}
L*	1	45.73 ± 0.18	71.68 ± 0.45^{Bc}	66.33 ± 0.73^{Aab}	65.15 ± 0.24^{Aa}	64.93 ± 0.91^{Aab}
	7	42.39 ± 0.88	63.17 ± 0.70^{Aab}	67.14 ± 0.74^{Bab}	64.41 ± 0.57^{ABa}	65.05 ± 1.00^{ABab}
	15	48.84 ± 0.73	65.55 ± 0.63^{Ab}	67.43 ± 0.88^{Ab}	65.06 ± 0.71^{Aa}	65.06 ± 0.66^{Aab}
	21	47.30 ± 0.58	64.06 ± 0.87^{Aab}	63.97 ± 0.82^{Aa}	64.89 ± 0.68^{ABa}	67.39 ± 0.67^{Bb}
	28	42.11 ± 0.46	62.53 ± 0.55^{Aa}	64.66 ± 0.54^{Aab}	71.81 ± 0.64^{Ab}	63.03 ± 0.89^{Aa}
a*	1	1.03 ± 0.01	2.66 ± 0.03^{Ae}	2.66 ± 0.02^{Bd}	3.48 ± 0.02^{Bd}	3.46 ± 0.01^{Ae}
	7	1.01 ± 0.01	2.56 ± 0.01^{Cd}	2.25 ± 0.01^{Ac}	2.57 ± 0.02^{Cc}	2.35 ± 0.01^{Bd}
	15	1.15 ± 0.04	2.28 ± 0.02^{Bc}	2.29 ± 0.01^{Bc}	2.31 ± 0.02^{Bb}	2.06 ± 0.01^{Ac}
	21	1.27 ± 0.01	1.84 ± 0.02^{Bb}	2.10 ± 0.03^{Cb}	1.62 ± 0.02^{Ab}	1.90 ± 0.01^{Bb}
	28	0.97 ± 0.01	1.66 ± 0.01^{Ba}	1.55 ± 0.01^{Aa}	1.55 ± 0.02^{Aa}	1.60 ± 0.02^{ABa}
b*	1	10.36 ± 0.40	17.20 ± 0.17^{Ac}	19.94 ± 0.24^{Cc}	18.98 ± 0.25^{Bd}	18.76 ± 0.12^{Bc}
	7	10.12 ± 0.13	14.28 ± 0.18^{Aa}	14.00 ± 0.10^{Aa}	16.13 ± 0.23^{Bc}	17.95 ± 0.21^{Cb}
	15	7.26 ± 0.08	17.41 ± 0.20^{Ac}	19.57 ± 0.15^{Cc}	18.69 ± 0.16^{BCd}	18.52 ± 0.15^{Bbc}
	21	7.36 ± 0.06	15.91 ± 0.16^{Cb}	15.27 ± 0.13^{Bb}	12.95 ± 0.14^{Aa}	13.41 ± 0.11^{Aa}
	28	9.95 ± 0.05	13.98 ± 0.16^{Aa}	14.60 ± 0.19^{Bab}	15.39 ± 0.10^{Cb}	13.91 ± 0.16^{Aa}

Data are expressed as means±standard error (n=10). MAP samples were previously ozonated and freeze-dried.
^{A,B,C} Different capital letters in the same row indicate that means are significantly different (P<0.05) between the different MAP packaging;
^{a,b,c} Different lowercase letters in the same column indicate that means are significantly different (P<0.05) between days of storage

Texture analyses

Results for texture profile analyses of control (untreated) and treated chicken meat stored in different modified atmosphere packs of CO_2 are presented in Table 5.5. Both MAP conditions and storage time had significant effects on meat texture parameters. Hardness, cohesiveness and chewiness values of all treated samples meat decreased significantly from day 7 onwards, such decreases being greater in the samples packaged with 20 % CO_2 than with others atmospheres. According to these results, the MAP with 20 %CO_2/ 80 %N_2 could be sufficient for preserving the initial textural properties of the ozonated dried chicken meat similar to those of raw meat for a period of 28 days.

Table 5. 5. Effects of carbon dioxide level on texture parameters of chicken meat stored in different modified atmosphere packs

Parameters	Days of storage	Frozen meat (FM)	MAP condition			
			20 %CO_2/80%N_2	30 %CO_2/70%N_2	40%CO_2/60%N_2	50%CO_2/50%N_2
Hardness (N)						
	1	34.32±0.44	65.12 ± 0.65^{Cc}	68.27 ± 0.20^{Dc}	61.81 ± 0.60^{Bc}	59.17±0.50^{Ab}
	7	33.44±0.35	64.46 ± 0.49^{Bc}	52.90 ± 0.45^{Ab}	52.20 ± 0.56^{Aab}	50.85±0.44^{Aa}
	15	31.60±0.25	55.27 ± 0.22^{Bc}	53.66 ± 0.34^{Bb}	55.13 ± 0.45^{Bc}	49.43±0.30^{Aa}
	21	30.78±0.34	54.04 ± 0.55^{Bb}	52.94 ± 0.33^{Bb}	54.28 ± 0.50^{Bb}	49.02±0.47^{Aa}
	28	35.80±0.09	40.98 ± 0.49^{Aa}	50.52 ± 0.65^{Cc}	50.24 ± 0.40^{Cc}	47.99±0.34^{Bb}
Cohesiveness						
	1	0.42±0.00	0.58± 0.00^{Cd}	0.56 ± 0.00^{Ad}	0.57±0.00^{Be}	0.59±0.00^{De}
	7	0.41± 0.01	0.56± 0.00^{Cc}	0.56 ± 0.00^{Cd}	0.54±0.00^{Ac}	0.55±0.00^{Bb}
	15	0.43 ± 0.01	0.56 ± 0.00^{Cc}	0.55 ± 0.00^{Ac}	0.56±0.00^{Bd}	0.54±0.00^{Ab}
	21	0.34 ± 0.00	0.50 ± 0.00^{Ab}	0.52 ± 0.00^{Bb}	0.53±0.00^{Cb}	0.54±0.00^{Db}
	28	0.41 ± 0.01	0.44 ± 0.00^{Aa}	0.48± 0.00^{Ba}	0.52±0.00^{Ca}	0.53±0.00^{Ca}
Chewiness (N/mm)	1	14.22±0.15	36.12 ± 0.11^{Ce}	37.11 ± 0.39^{Dd}	29.76±0.17^{Ac}	30.80±0.09^{Bc}
	7	11.97±0.14	30.32 ± 0.24^{Cd}	23.78 ± 0.14^{Aa}	27.07±0.26^{Bc}	24.86±0.22^{Ab}
	15	12.28±0.16	31.70 ± 0.37^{Cd}	26.76 ± 0.29^{Bb}	29.04±0.30^{Cc}	24.94±0.22^{Aa}
	21	9.05±0.01	28.28 ± 0.30^{Bc}	26.67 ± 0.33^{Ab}	28.54±0.09^{Bc}	25.16±0.19^{Aa}
	28	14.46±0.11	18.25 ± 0.17^{Aa}	28.08 ± 0.30^{Cd}	26.65±0.22^{Cc}	23.40±0.21^{Bb}

Data are expressed as means±standard error (n=15)

^{A,B,C} Different capital letters in the same row indicate that means are significantly different (P<0.05) between the different MAP packaging. ^{a,b,c} Different lowercase letters in the same column indicate that means are significantly different (P<0.05) between days of storage

Sensory descriptive analysis

In terms of the overall acceptability descriptor **(**Figure 5.2), panelists preferred (P< 0.05) the control samples than the treated samples throughout the whole storage period. In general, chicken breast fillets packed with 50 %CO_2/ 50 %N_2 were also less acceptable in the last days of storage (days 21 and 28) by panelists than the other MAP packaged samples (20, 30 and 40 %CO_2) .

Regarding hardness and juiciness attributes, both time of storage and the type of packaging significantly affected them. In agreement with texture data, the same samples that showed low values in hardness and chewiness corresponded to those that received better scores by the panel. TPA Hardness had significant correlations with respect to sensory perceived hardness and juiciness with correlation coefficients higher than 0.5 (hardness r= -0.638; chewiness r= -0.540, $P< 0.05$). Chewiness produced significant correlations with panel juiciness (r= -0.540; $P< 0.05$) and hardness (r= -0.610; $P< 0.05$). Significant correlations were also observed between cohesiveness and sensory hardness (r=-0.526; $P< 0.05$). González-Fernández, Santos, Rovira, and Jaime (2006) found interrelationship between sensorial and instrumental hardness and chewiness in other meat products.

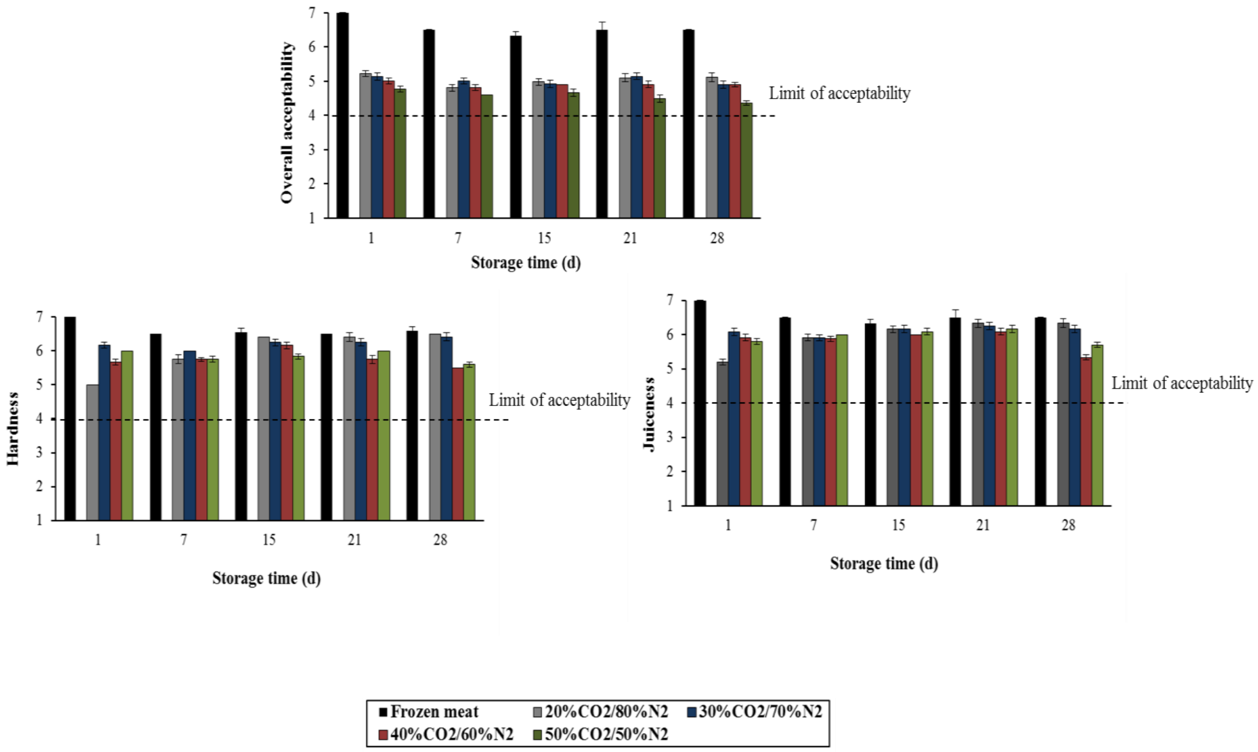

Figure 5. 2. Sensory descriptive analyses on ozonated dried chicken meat for MAP experiments with different CO_2 concentration. Means with standard errors (n = 18) are shown. Overall acceptability: 1=Reject (very different from fresh chicken meat), 7= acceptable (very similar to fresh chicken meat); Hardness score: 1=very hard, 7=very tender; Juiciness score: 1=very dry, 7= very juicy (4=limit of acceptability).

The samples packed with 20 and 30% CO_2 were perceived by panelists as significantly (*P*< 0.05) more tender and juicier than the other treated samples. Interestingly, under the 20% CO_2/ 80% N_2 packaging condition, samples received scores in hardness and juiciness similar to those of control samples (raw meat) from day 15 onwards, as the panelists did not find significant differences between the samples. Based on these results, the 20 %CO_2/ 80 %N_2 treatment was considered to be the most effective one in maintaining the sensory quality of chicken breast fillets treated with ozone and freeze-drying during 28 days of storage.

5.3.3. Trial iii. MAP gas mixtures with different concentrations of CO_2 and O_2

Headspace gas composition, pH and color change

The headspace atmosphere showed significant changes in composition throughout storage time (data not shown). O_2 and CO_2 concentrations decreased in all samples, while N_2 concentrations increased (*P*< 0.05) during the storage. The greatest changes within packs were observed in those containing low oxygen levels (10 %O_2/ 30 %CO_2/ 60 %N_2). Similar results were reported by Esmer et al. (2011), who stated relative variations in gaseous atmospheres composition within the modified atmosphere packs, in which the fluctuations were higher at a lower oxygen level.

After day 15, a slight increase in pH was observed in the samples packed under high O_2 MAP conditions (40%O_2/20%CO_2/40%N_2) from initial pH of 5.77 ± 0.01 to 6.30 ± 0.02, while it continued decreasing until day 28 in the case of other package treatments (data not shown). The lowest values of pH were obtained for both MAP (10 %O_2/ 30 %CO_2/ 60 %N_2 and 20 %O_2/ 30 %CO_2/ 50 %N_2) samples, whose pH values were below 5.6 at day 28 of storage for both samples.

In relation to color parameters, the type of packaging and storage time had no significant effects on lightness parameter, while minor variations were observed in a* and b* values of chicken meat stored in a gas mixture without CO_2 and O_2. Redness values for all MAP packaged samples decreased (*P*< 0.05) during storage. This reduction was higher (*P*< 0.05) for high O_2 MAP (40 %O_2/ 20 %CO_2/ 40 %N_2) packaged chicken samples by day 28. The decrease in a* values of meat packaged in high-O_2 and low-CO_2 atmospheres in this study may be caused by myoglobin oxidation due to high meat´s pH observed (6.30 ± 0.02 on day 28) in the same samples. Seydim et al. (2006) stated that at higher pH values (more than 6 units), the oxidation of oxymyoglobin is important, because mitochondrial enzyme systems do not shut down and have the ability to utilize available oxygen.

Texture analyses

Results of the instrumental texture profile analyses of untreated meat (frozen meat) and samples packed in different gas mixture without CO_2 and O_2 are shown in Figure 5.3. As can be seen, textural parameters were affected ($P < 0.05$) by the packaging conditions and storage time. Both hardness and chewiness increased significantly throughout storage time in most of packed samples. The increase of these parameters was particularly evident in samples packaged with 40 %O_2/ 20 %CO_2/ 40 %N_2, becoming harder and less chewy meat on day 28. Samples packed with 30 %O_2/30 %CO_2/ 40 %N_2 showed the lowest values of all textural parameters compared with the rest of packaged samples ($P < 0.05$) on day 28.

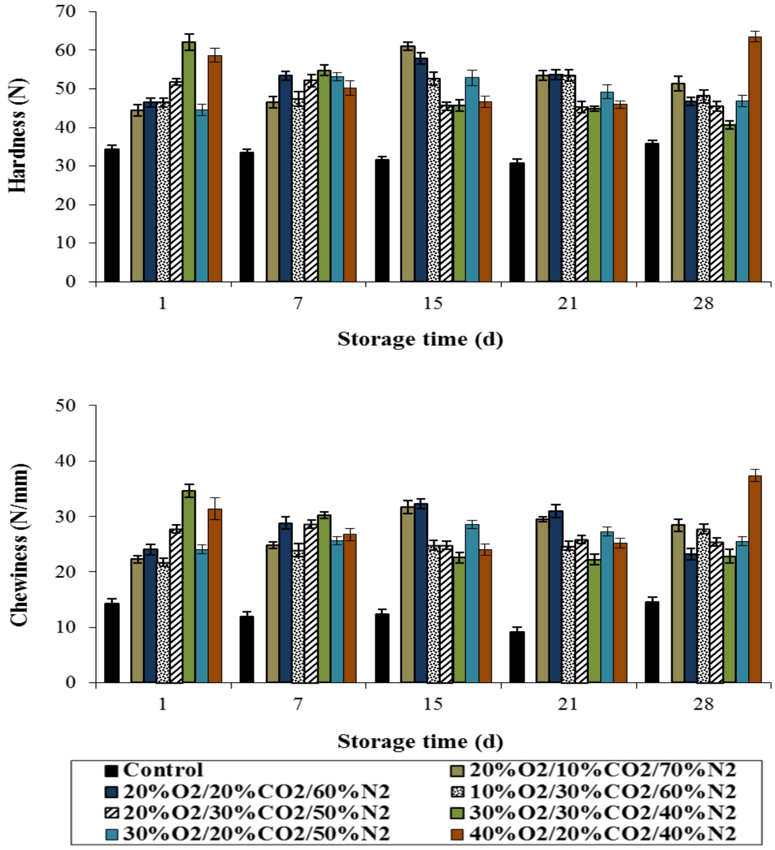

Figure 5. 3. Changes in texture parameters (hardness and chewiness) for control (frozen meat) and samples meat packaged under different various gas mixtures of O_2:CO_2:N_2 during storage time (in days). Means with standard errors (n = 15) are shown

Sensory descriptive analyses

The sensory results were in agreement with instrumental measurements of texture. The instrumental and sensory parameters of hardness showed a significant correlation of (r=-0.602, $P< 0.05$). TPA chewiness also was significantly correlated ($P< 0.01$) to hardness and chewiness sensory parameters with r= -0.724 and -0.761, respectively. Results from sensory analyses showed that meat samples packaged with high O_2 concentrations (40 %O_2/ 20 %CO_2/ 40 %N_2) scored lower for hardness and juiciness attributes than meat from the others treatments (Figure 5.4). These samples were perceived as harder, less juicy and chewy by panelists, the same samples having showed the highest hardness, cohesiveness and chewiness values in the TPA analyses. These results are in agreement with those of Zakrys-Waliwander, O'Sullivan, Walsh, Allen, and Kerry (2011), who reported that beef steaks stored under high oxygen atmospheres were significantly perceived as less juicy by consumers. The sensory attributes hardness and chewiness did not show significant differences during the storage period, whereas juiciness decreased ($P < 0.05$) in all samples.

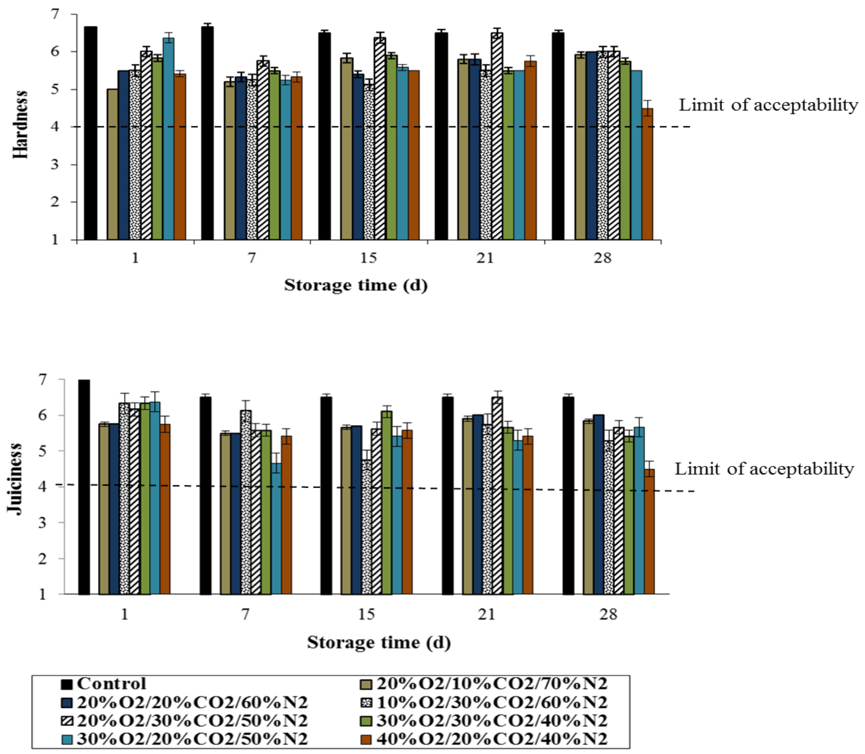

Figure 5. 4. Sensory descriptive analyses on ozonated dried chicken meat for MAP experiments with different CO_2 concentration. Means with standard errors (n = 18) are shown. Hardness score: 1=very hard, 7=very tender; Juiciness score: 1=very dry, 7= very juicy (4=limit of acceptability).

At 28 days of storage, all samples received high scores between 4.5 and 6 for juiciness, indicating "juicy" and "very juicy" meat according to the scale used. Zouaghi (2011) found an important decrease in juiciness for ozonated dried chicken meat stored in vacuum packed and reported lower juiciness compared to our study. To sum up, modified atmosphere packaging preserved juiciness of samples more efficiently than vacuum packaging.

5.4. Conclusions

Increasing the concentrations of oxygen (more than 30 % with or without CO_2) in modified atmosphere gas compositions resulted in loss of redness and an increase in the pH values. Also, when the concentration of CO_2 in modified atmosphere was more than 40 %, a decrease of the a* and b* values of treated samples happened. However, the texture and sensory properties of ozonated dried chicken meat packaged in modified atmospheres were best preserved in atmospheres containing low CO_2 concentrations (20-30 %) rather than high (40–50 %) concentrations.

As a result, the best preservation conditions for ozonated dried chicken breast fillets stored at room temperature was in MAP (20 %CO_2/ 80 %N_2) gas combination, maintaining acceptable color together with texture and sensory quality (hardness and juiciness attributes were scored above the limit of acceptability, and also being more similar to the characteristics of raw meat during 28 days of storage). A long-term study is being carried out to determine the self-life of the new product under those optimal conditions.

5.5. References

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CHAPTER VI. Conclusions

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This last chapter discusses the general conclusions and contributions of the study as a whole.

The overarching questions in this research are: what are the relevant drivers (e.g. innovation inputs) of firm innovative performance? How can firms use open innovation strategies (e.g. cooperation with external partners) to facilitate the acquisition of external knowledge? And how can the development of the new products occur at industry level through the introduction of combined food processing technologies that could lead to improve food quality and provide, at the same time, benefits in terms of a longer shelf life?

In the **second chapter** of this thesis, we studied different forms of innovation that could be of interest in the food sector when compared to other sectors. Thereafter, we endeavored to explore whether input factors could affect firm innovation performance. This study provides a series of important results for the theory and practice of firms' innovation management. First, our study confirms that the recent economic crisis led to substantial drops in innovation activities as a result of uncertainty and declines in the demand for innovative products. The agri-food sector is the one which has best withstood the crisis when compared to the rest of Spanish industry sectors. Innovation persistence plays a substantial role as a key mechanism for organizational growth and even survival in tough economic times in the agri-food sectors. Second, by exploring the role exercised by different sources of innovation on the probability of innovation success and a firm's total turnover, our study underscores the importance of strong policy support for stimulating in-house innovation activity by companies to generate more innovations which helps them to survive in periods of crisis and maintain their market share. Our empirical evidence also confirms the importance of other sources based on external contracting and collaboration in times of crisis for improving firm innovative performance through access to valuable scientific and technological knowledge. External information and technologies acquisition can help agri-food firms to overcome the limitations of a firm's own resources to support their innovation process. Thus, this finding opens potential discussions about the role of absorptive capacity in assimilating and exploiting the knowledge generated outside firms' boundaries effectively. Thirdly, the ability of a firm to maintain its innovation intensity in periods of crisis will also depend on firm-level characteristics which play an important role in shaping innovation activity within industries. This study showed the crucial role played by human capital in reducing the effects of the crisis on innovation investments of firms and the success of innovative sales for firms. Managers should keep their stock of skilled employees by monitoring the loss of innovation during times of crisis, which promotes higher levels of employment and job creation.

The **third chapter** explores the effect of open innovation on the innovation process and explores the role of human capital in facilitating the creation of cooperation networks and incorporating external information. Furthermore, we take accounts of the heterogeneity of technological intensity in manufacturing sectors by differentiating between high- and low-technology (i.e. food sector) industries, which exhibit marked differences in their internal capabilities to manage external knowledge. In doing so, this study provides a series of academic contributions to the existing research on open innovation and alliance diversity, and confirms the relevance of a firm's absorptive capacity in leveraging the potential benefits of open collaborative modes of innovation. Firstly, this study contributes to a better understanding of how manufacturing firms should configure their alliance portfolio depending on the type of innovation they seek to develop in order to reduce the usual problems associated with diversity. Secondly, our study highlights the importance of the absorptive capacity created and accumulated in R&D efforts and in qualified human resources to mitigate the difficulties in transferring and leveraging very diverse knowledge from partners. Firms need to invest in their own research processes and in qualified human resources in order to be able to establish and maintain linkages with external partners.

The **fourth chapter** examines empirical experiments through the combinations of multi-hurdles-technology on the shelf-life extension of broiler chicken meat fillets, in order to develop new high-quality raw meat products from fresh chicken meat, safe, with a high nutritional value, with no additives added and long-lasting at room temperature. In this sense, ozonation and freeze-drying were used as hurdles to preserve chicken meat for up to eight months at room temperature. Firstly, ozone and lyophilisation are shown to be valid in retarding the growth of most microbial groups from the first month of storage. Secondly, the combination of ozone with a 0.6 ppm concentration for 10 minutes combined with lyophilisation was found to be the best method to maintain meat safety at room temperature for a long period of time. Thirdly, this study highlights the potential role of food product innovation and food safety in improving the utilization of foods and to extend the storage period.

The **fifth chapter** addresses the use of modified atmosphere packaging for the preservation of ozonated freeze-dried chicken meat. Deterioration in sensory qualities can result in economic losses due to consumer rejection of the product. This study reveals that the texture and sensory properties of ozonized dried chicken meat packaged in modified atmospheres were best preserved in atmospheres containing low CO_2 concentrations (20-30 %) rather than high (40–50 %) concentrations. The gas composition of 20 %CO_2/ 80%N_2 was the most effective treatment for maintaining the physicochemical and sensory quality of ozonated freeze-dried samples chicken similar to that of raw meat. This study adds to previous research the benefits of the innovations in using modified atmosphere packaging technologies for providing better sensory quality of ozonated freeze-dried chicken meat to reach more potential markets and satisfy consumer demands.

Conclusiones

En este último capítulo se analizan las conclusiones generales y aportaciones extraídas de los diferentes capítulos.

Las preguntas de esta investigación son: ¿Cuáles son los factores claves (inputs de innovación) que influyen en el proceso innovador de las empresas? ¿Cómo utilizan las empresas las estrategias de innovación abierta (la cooperación con los socios externos) para facilitar la adquisición del conocimiento externo? y ¿cómo se produce el proceso de desarrollo de un nuevo producto a nivel de planta piloto a través de la introducción de tratamientos combinados que podrían conducir a mejorar la calidad de los alimentos y proporcionar una larga vida útil de los alimentos?.

En el **segundo capítulo** de esta tesis se analizaron los tipos de innovaciones que pueden ser de interés para las empresas agroalimentarias, en comparación con otros sectores de la economía española. También, se examinó el efecto ejercido por los indicadores de inputs de innovación sobre el rendimiento innovador de las empresas. Este estudio proporciona varias implicaciones para la gestión de la innovación en las empresas. En primer lugar, nuestro estudio confirma el efecto negativo de la recesión económica sobre la inversión en innovación frente a la incertidumbre de las empresas respecto a la demanda del mercado así como en relación con la recuperación de sus costes de producción. Más concretamente, en España, el sector agroalimentario es uno de los que mejor está afrontando la situación de crisis económica. Los resultados revelan que tanto los beneficios como el crecimiento de las empresas agroalimentarias dependen de su capacidad de mantener la actividad innovadora, especialmente en tiempos de crisis. En segundo lugar, explorando el papel ejercido por las diferentes fuentes de innovación en la determinación del desempeño innovador de las empresas, nuestro estudio demuestra que la capacidad de las empresas para lograr más innovaciones y salir de la crisis depende de su esfuerzo interno. Nuestra evidencia empírica confirma también la importancia de otras fuentes de innovación como la contratación externa y la colaboración, para mejorar el rendimiento innovador de las empresas en tiempo de crisis mediante el acceso de valiosos conocimientos científicos y tecnológicos. Las redes de cooperación ayudan a las empresas agroalimentarias para apoyar el proceso de innovación debido a sus recursos limitados y a su menor capacidad para absorber los riesgos. Por lo tanto, un equilibrio entre la adquisición externa y la inversión en I+D interna denominado “capacidad de absorción” resulta necesario para identificar, asimilar y explotar el conocimiento disponible fuera de las fronteras de las empresas. En tercer lugar, la capacidad de la empresa para mantener su esfuerzo innovador en el período de crisis dependerá también de las características internas de las empresas mismas. Este estudio muestra el papel crucial que desempeña el capital humano en la reducción de los efectos de la crisis sobre las inversiones en innovación de las empresas y para el éxito de las ventas innovadoras. Los gerentes deben mantener a sus empleados, sobre todo a los trabajadores cualificados que pueden tener un impacto significativo en los procesos de innovación y de creación del empleo durante las épocas de crisis.

En el **tercer capítulo** se analizó el efecto de la innovación abierta en el proceso de innovación y se exploró el papel del capital humano para facilitar la creación de las redes de cooperación para la asimilación de la información externa. Por otra parte, hemos tenido en cuenta la heterogeneidad entre los sectores de la industria manufacturera según su intensidad tecnológica mediante la diferenciación entre las industrias de alta y de baja tecnología (es decir, el sector alimentario), los cuales presentan diferencias en sus capacidades internas para gestionar el conocimiento externo. De este modo, este estudio proporciona varias contribuciones a la investigación existente en la innovación abierta y la diversidad de socios de cooperación, y confirma la importancia de la capacidad de absorción de la empresa para mayor acceso al conocimiento y mejor aprovechamiento de la cooperación abierta. En primer lugar, este estudio contribuye a una mejor comprensión de cómo las empresas fabricantes deben configurar su número de socios dependiendo del nivel de innovación que se persiga para reducir los problemas habituales asociados con el exceso de diversidad. En segundo lugar, nuestro estudio destaca la importancia de la "capacidad de absorción" acumulada en los esfuerzos de I + D y en los recursos humanos cualificados para mitigar las dificultades en la transferencia y la exploración de diversos conocimientos de los socios de cooperación. Las empresas tendrán que invertir en sus propios procesos de Investigación y Desarrollo, sus recursos humanos cualificados y mejorar las competencias del personal con el fin de mantener sus vínculos con los socios externos.

En el **cuarto capítulo** se aplicó el uso de métodos combinados para alargar la vida útil de pechugas de pollo *Broiler*, con el fin de conseguir un nuevo producto cárnico crudo de alta calidad, sin aditivos, estable a lo largo del tiempo a temperatura ambiente. En este sentido, la ozonización y la liofilización fueron utilizadas como barreras para conservar la carne de pollo hasta los 8 meses de almacenamiento a temperatura ambiente. En primer lugar, la combinación del ozono y la liofilización resultó eficaz para reducir los recuentos microbiológicos desde los primeros meses de almacenamiento. En segundo lugar, el mejor tratamiento combinado fue una concentración de ozono del orden de 0.6 ppm y un tiempo de 10 min junto con la liofilización. Con estas condiciones fue posible mantener una gran estabilidad microbiológica de la carne de pollo durante un largo tiempo de almacenamiento. En tercer lugar, este estudio pone de relieve el gran potencial de la innovación en productos alimentarios y la seguridad alimentaria para mejorar la utilización de los alimentos y extender sus períodos de almacenamiento.

El **quinto capítulo** se centró en el uso del envasado en atmósfera modificada para la conservación de la carne de pollo ozonizada y liofilizada. El deterioro de las propiedades sensoriales puede ocasionar pérdidas económicas importantes, debido al rechazo del producto por parte del consumidor final. Para ello, en este estudio se mostró que tanto la textura como las propiedades sensoriales de la carne de pollo ozonizada, liofilizada y envasada en atmósferas modificadas se conservaron mejor en atmósferas que contenían bajas concentraciones de CO_2 (20-30%) que en altas concentraciones de CO_2 (40-50%). Además, la composición gaseosa de 20% de CO_2 / 80% de N_2 fue el mejor tratamiento para mantener las propiedades físico-químicas y la calidad sensorial de la carne ozonizada y liofilizada bastante similar a la carne cruda sin tratar. Este estudio añade al estudio anterior los beneficios de la innovación en el uso del envasado en atmósfera modificada para proporcionar una mejor calidad sensorial de la carne de pollo ozonizada y liofilizada, para llegar a nuevos mercados y satisfacer las demandas del consumidor.