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TESIS DOCTORAL

TEXTURA DE SNACKS CRUJIENTES CULINARIOS

(TEXTURE OF CULINARY SNACKS)

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To my beloved father and brother

António da Luz Carvalho

Alexandre José Carvalho

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RESUMEN

Piel de cerdo es un subproducto resultante del corte y desosar de la carcasa (6.7% del peso de carcasa), con una textura mucho particular, y existen algunos estudios sobre snacks cárnicos, de modo a posibilitar una manera alternativa de valorar ciertos tejidos cárnicos. Freír es un método mucho utilizado en este tipo de snack, dado que permite desarrollar una textura y flavour únicos, mientras el consumidor está más exigente y es necesario desenrollar algunas técnicas de producción de snacks sin freír. Así, deshidratación por aire caliente seguida de microondas puede ser una opción para obtener este tipo de producto. Una nueva técnica de investigación de la naturaleza acústica de crujiencia se desarrolló recientemente, e es basada en el registe simultaneo de sonido y fractura/eventos mecánicos, de modo a percibir las propiedades sensoriales de los alimentos (crujiente, crocante), y establecer relaciones entre el sonido emitido y características estructurales de los productos crujientes. Así, el objective de este estudio es desarrollar métodos deconianza y fáciles de implementar de forma a evaluar el crujiencia sensorial y crocancia de los alimentos, y como son las condiciones experimentales instrumentales de sonido-textura y de análisis de microestructura por “scanning electron microscope” (SEM) se pueden relacionar con la percepción de textura sensorial. Otro objetivo es determinar el efecto de diferentes formulas de rebozados con ingredientes menos comunes (CO₂ y etanol) además de harina y agua. Los parámetros instrumentales de textura, más precisamente, crujiente y dureza, fueron significativamente diferentes entre los rebozados solo con agua y harina y los rebozados con etanol; así la dureza es superior en los rebozados con agua, y la crujiencia es superior en los rebozados con etanol, y durante el almacenamiento, la incorporación de CO₂ aumentó el crujiencia de los rebozados etílicos. La adherencia después del almacenamiento, presentó diferencias, principalmente en los rebozados con agua y sin CO₂, y la dureza sensorial está asociada a los parámetros texturales, y así los rebozados sin CO₂ fueron los menos crujientes y crocantes. Una fórmula para desarrollar espumas a partir de hidrocoloides por liofilización y desecado. Imágenes SEM evidencian que las espumas liofilizadas nada colapsaron y las desecadas presentaron un enorme colapso en la estructura.

Palabras-Clave: Liofilización, Desecado, Piel de cerdo, Sonido-Textura, Crujiente

Abstract

ABSTRACT

Pork skin is a by-product that results from carcass cut and de-boning (6.7% of carcass weight), with a very particular texture and there are several studies about meat snacks in order to increase the alternative value of certain meat tissues. Frying is a largely used method to produce these type of snacks since it permits to develop an unique texture and flavour, however due to a large demand from consumer it becomes necessary the use of some techniques to produce “snacks” without frying. So, dehydration by hot air followed by microwaving may be an option to obtain a similar kind of product. A new approach to investigate the acoustic nature of crispness has recently emerged and it is based on the simultaneous recording of sound and fracture/mechanical events, in order to understand sensory properties of foods (i.e., crispness, crunchiness) and establish relationships between emitted sound and structural characteristics of brittle food products. Hence, the objective of this study was to develop a reliable and easy way to implement methodologies for evaluating the sensory crispness/crunchiness of crispy food and how experimental conditions of instrumental sound-texture assessment and microstructure analysis by scanning electron microscope (SEM) could be related to the sensory perception of texture. Another aim of this study was to study the effect of different batter formulations with uncommon ingredients (CO₂ and ethanol) besides water and flour. Instrumental texture parameters, namely, crispness and hardness, were significant different on water batters and on ethanol batters; and thus hardness was higher on water batters, and crispness was higher on ethanol batters, and during storage, CO₂ incorporation increased ethanol batter crispness. Adherence of batter had significant differences at day 2, mostly on water formulation without CO₂, and sensory hardness was associated to textural parameters, and water formula without CO₂ was the least crispy and crunchy in the face of the most spongy and elastic. A formula to prepare whipped foam to freeze-drying or drying was developed. SEM images evidences that on the freeze-died foams weren't any sign of collapse and on dried foams an enormous collapse in all structure. Sensory results indicated that a certain degree of hardness is necessary for crispness perception, as those values had a similar behaviour, and were higher on dried foams than on freeze-dried foams, as well as it was overall acceptability.

Keywords: Freeze-drying, Drying, Hydrocolloids, Scanning Electron Microscopy, Pork Rind, Microwave, Texture, Sound, Sensory crispness

I – INTRODUCTION

(I - INTRODUCCIÓN)

Food Science became a legitimate awareness just following World War II. Prior to that, the vast majority of foods in the world were prepared locally, and so consumers had no option but to visit butchers, bakeries, dairies, greengrocers, and other purveyors close to their homes and purchase mainly unbranded goods of questionable quality.

Technologies developed in the early and middle of the past century had allowed the manufacture and distribution of canned, chilled, and frozen foods and hence consumers happened to be more demand. At this point, it became imperative for food processors to provide consistently high-quality branded products, and thus they needed to understand the materials and ingredients used in the manufacture of these products. In 1990, a new trendy appeared on culinary history noticing new sensations and emotions supported on recent creative culinary technologies.

These new techniques and tools exposed outstanding results and lead to the concept of “Molecular “or “Techno-emotional” Culinary, reflecting each “Chef” culture and lifestyle, for instance Ferran Adrià, Heston Blumenthal, Grant Achatz, René Redzepi, Toño Pérez, Pierre Gagnaire (Moura, 2011) and Juan Mari Arzak (Arzak, 2008). Another goal was to emphasize some less used ingredients (sardine, tongue, liver, pork rind) and above all to achieve two main concerns: environmental sustainability and establishing constant relation with scientific world (universities); and so the final product have been creativity and scientific knowledge to improve and develop new tastes and textures, given that eating is a multisensory experience.

The cultural patrimony of gastronomic richness is a playground of infinite dimension in which to indulge both sensory and emotional experiences (Adrià *et al.*, 2007), cooking provides an ideal framework to study a variety of complex phenomena - from basic chemistry to materials science to applied physics (Aguilera, 2012). And cuisine interacts with a guest ever more disposed to the creation of integrated experiences, combining the sensory messages with emotional ones (Blumenthal, 2008). This fact allowed considering an idea of the power of gastronomy to serve as a representation of culture, science, art, or identity (Aduriz *et al.*, 2012).

Introduction

In the past, the aim of a diet was to provide nutritional requirements in order to avoid nutrient deficiencies. This aim has evolved into the desire to keep or improve health status through the diet. Likewise, the evolution of gastronomy into haute cuisine has led to the utilization of new ingredients and technologies that could interact with nutrients and alter the contribution of the dishes to the overall diet. The main goal of haute cuisine cooks is to innovate and design delicious dishes with new textures and flavors, which promote new sensations in customers (Navarro *et al.*, 2012). And so, for the first time in the history of foods and gastronomy a good body of scientific knowledge on the structure-forming capabilities of old and novel ingredients and how some of the complex microstructures of foods come into being, was accumulated in a way that allows food technologists and chefs to design food structures for health and pleasure (Aguilera, 2012).

I.1 – CRISPY FOOD PRODUCTS

Food processing is facing new challenges, which include providing, in addition to microbiologically safe and high quality foods, products that fulfil the new demands of well-informed consumers (Gazmuri *et al.*, 2009). Since there is an increasing awareness amongst the consuming public regarding the quality of foods available in the market (Jonnaladadda *et al.*, 2001), then people ask that products contribute to their wellness and health, but they also require specific textures, flavours and colours. In the light of this approach, product formulation appears as a good alternative for developing new products with controlled attributes. Therefore, formulated products are gaining importance in the snack industry as a good alternative to the use of raw materials because of the advantages of reproducibility, uniformity and lack of defects (Gazmuri *et al.*, 2009).

Appearance, color, texture and flavor are important factors in consumer perceptions of food products and among them, texture is one of the most important one (Bourne, 2002) and it is particularly crispness the most critical properties determining consumer acceptance, in fact, crispy products are usually low moisture foods and a change in moisture content directly affects texture, and thus, products will become less crispy, and consequently that will define their acceptance (Mazumder *et al.*, 2007). Indeed,

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crispness is a highly valued textural characteristic, in particular, in breaded and battered foods like fish, seafood, poultry, cheese or vegetables which are favored by consumers and that have become very popular over the last decades both in high-convenience consumer societies and in developing countries (Varela *et al.*, 2008).

The snack food industry includes manufacturers of potato chips, corn chips, popped popcorn, pretzels, extruded cheese snacks, seed snacks, mixed nuts, peanuts and others. This market is relatively saturated; however, room remains for niche products that are high in nutrients such as fibre, protein, and omega-3 fatty acids or that has no gluten (Han *et al.*, 2009), and also with cost effective and highly acceptable fried snacks (Senthil *et al.*, 2002). The acceptance of snacks is critical because of the specific quality attributes that attract people (Bourne, 2002).

Crispy foods are enjoyable snack food products that are consumed throughout the world (Kerr *et al.*, 2001), play very important roles in the diet of the modern consumer (Nath *et al.*, 2008), and are design to be less perishable, more durable and appealing than natural food. Different types of snacks fall into several categories such as low fat, baked but not fried, coated snacks, etc (Mazumder *et al.*, 2007). Sometimes these crispy products are often an impulsive purchase, and one of convenience, and can be defined as a light meal between regular meals which include a broad range of products that can take many forms (Sajilata *et al.*, 2005).

As a result, people are at the mercy of the food choices most available. Eating healthy snacks can offer many benefits to consumers, such as increased energy and feeling fuller longer, so there is a big opportunity for the makers and marketers of snacks to leverage these connections to maximize health positioning (Ballard, 2012), becoming a significant part of the diet of many individuals, particularly children, and influence overall nutrition (Shukla, 1994). Due to the growing consumer demand for healthy, natural and convenient foods, attempts are being made to improve snack food nutritional values (Sun-Waterhouse, *et al.*, 2009; Meng *et al.*, 2009). Several techniques have been used to develop healthy crispy food products namely the traditional art of deep fat frying with or without batter coatings, the ancestral dehydration technique, and also the innovative microwave heating and the astonishing freeze-drying process, which most remarkable advantage is maintaining the natural attributes of foodstuffs.

I.1.1 – TECHNOLOGY

DEEP-FRYING

Deep fat frying is an important, ubiquitous and highly versatile food processing technology, which has been used since antiquity to cook a wide spectrum of products (Saguy *et al.*, 2003), and is especially suited to develop snacks with unique flavours and textures (Gazmuri *et al.*, 2009). Its unique contribution to sensory characteristics, together with the relatively low cost of large-scale frying, has made fried foods the staples of the ever growing late 20th century fast food industry (Saguy *et al.*, 2003). Furthermore, fried foods play a significant role in USA economic growth since has accelerated acceptance of ethnic foods, bolder flavours, and added crunch to traditional meals (Sloan, 2000).

Despite its considerable fat content and intensified consumer's awareness of the relationships between food, nutrition and health, frying remains a principal cooking method (Saguy *et al.*, 2003). In fact, frying of food is one of the oldest cooking methods and also one of the most common techniques throughout the world. That complex operation represents a process, which involves several chemical and physical changes including starch gelatinization, protein denaturation, water vaporization and crust formation (Rimac-Brneié, *et al.*, 2004).

Frying is basically a dehydration process and considered by many to be more an art than a science or technology (Math *et al.*, 2004). Immersion frying, or deep fat frying, may be defined as the process of cooking foods by immersing them in an edible oil or fat, which is at a temperature above the boiling point of water, typically 150-200°C (Yamsaengsung, *et al.*, 2002). The process involves simultaneous heat and mass transfer (Debnath *et al.*, 2003), which cause significant microstructural changes to both the surface and the body of the product (Gazmuri *et al.*, 2009). Heat is transferred from the oil to the food, which results in evaporation of water from the food and absorption of oil by the food (Gamble *et al.*, 1987).

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As the product fries, the inner moisture is converted to steam, creating a pressure gradient as the surface dries out and causing oil to adhere to product's surface at the damage areas. Most of the oil enters the product from the adhering oil being pulled into the product, when the product is removed from the fryer, adhering oil enters the product due to vacuum created by condensation of water vapour in the pores (Gamble *et al.*, 1987).

Some foods are fried in small quantities of oil whereas others are deep fried. The quality of fried foods depends upon the quality of the frying oil and thus it is of prime importance to maintain and protect the quality of the frying medium. The necessity of using a good frying oil and maintaining it in that state as long as possible becomes clear when one considers that all fried food absorbs a certain amount of fat. In this drying cooking process, fat serves as the heat transfer medium and also migrates into the food providing nutrients and flavour (Albert *et al.*, 2002). This process is widely used in an industrial as well as institutional preparation of foods, because the consumers prefer the taste which confers unique characteristics such as smooth mouth feel and distinct flavour (Adedeji *et al.*, 2009), appearance, color, texture and palatability of fried food products (Rimac-Brnei , *et al.*, 2004).

In some studies about fried foods (Yamsaensung *et al.*, 2002), during frying processing the product undergoes both shrinkage in radial direction and expansion in the thickness due to gas bubble expansion inside the product. So, expansion occurred at about time the crust was forming. The formation of the crust greatly reduces the rate of moisture transfer and causes an increase in pressure inside the frying product. Thus, this pressure buildup leads to an expansion of the pores, which results in a crispy final product.

BATTERED FOOD

Many fried products incorporate coatings that are used to add value to the products by improving their texture, flavour, weight and volume. Coatings can take the form of a batter and/or breading and often these coatings are applied in combination to

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produce a desired effect. Food batters are complex systems comprised of water, flour (Fizman *et al.*, 2003) or starch, and seasonings (Xue *et al.*, 2006) into which food products are dipped prior to cooking, normally by frying (Fizman *et al.*, 2003). No exact recipes exist for batter systems and formulations can be extremely flexible to allow for maximum adaptability to the product development process, depending on the food substance and the desired coating appearances (Xue *et al.*, 2006).

Batter application prior to frying, forms a continuous and uniform layer over the food surface (Akdeniz, 2006). Edible coatings might offer a potential solution to form a barrier to fat absorption during frying process (Albert *et al.*, 2002). In addition, it enhances the texture, flavour and appearance of foods (Akdeniz, 2006).

The viscosity of a batter plays a major role in the quality of the coating. It is also one of the most important factors in determining a batter's performance during frying (Shih *et al.*, 1999; Dogan *et al.*, 2005). The factors that affect rheological properties of batters include composition and proportion of the ingredients, the solid to water ratio, and the frying temperature (Xue *et al.*, 2006). Those rheological properties of batters depend on the mixing and heating process which, in turn, determines the batter behaviour and the textural properties of the final product. Physical and structural changes during aerated batter processing may alter their performance during thermal processing or the quality of the final product. The ingredients of the batter and the processing conditions have been related to the rheology and texture of the final product (Baixauli *et al.*, 2007).

Crust brittleness or crispness is a critical element in a consumer's evaluation of a particular fried battered food product. To achieve the desirable texture of crust in fried battered products, design of appropriate ingredients with wide-ranging functionalities is available (Chen *et al.*, 2008).

In fact, edible coatings and incorporation of active ingredients can improve food quality (Khan *et al.*, 2012). For instance, the proteins provide structure and increase the coating pick-up values and final yield in the fried products (Fizman *et al.*, 2003) and gluten is like a net, traps and holds air bubbles in batter (Chen *et al.*, 2008).

DEHYDRATION

Consumer demand has increased for processed products that keep more of their original characteristics. In industrial terms, this requires the development of operations that minimize the adverse effects of processing (Nijhuis *et al.*, 1998).

Food dehydration is still one of the most relevant and challenging unit operation in food processing, although the art of food preservation through the partial removal of water content dates back several centuries (Vega-Mercado *et al.*, 2001).

Thus this ancient process offers numerous advantages including prolonged preservation time, high productivity and quality of resulting products (Aguilera *et al.*, 2011; Madhlopa *et al.*, 2007; Zhang *et al.*, 2006; Schuck *et al.*, 2008) and dehydrated products that can have an extended life of a year but, unfortunately, the quality of a conventionally dried product is usually drastically reduced from that of the original foodstuff (Ratti, 2001).

In recent years, a variety of drying methods have been tried and much attention has focused on the quality of the products obtained by these methods (Jena *et al.*, 2005; Matuska *et al.*, 2006; Agnieszka *et al.*, 2010). In the particular case of food drying, this method indicates loss of volatiles and flavors, changes in color and texture, and a decrease in nutritional value. Furthermore, residual enzyme activity and microbial activity in dried foods are essential parameters that affect product quality and shelf-life (Nijhuis *et al.*, 1998).

Air-drying is an ancient process used to preserve foods (De Bonis *et al.*, 2008), in which the solid to be dried is exposed to a continuously flowing hot stream of air where moisture evaporates (Ratti, 2001; Krokida *et al.*, 2006). So, the main purpose of drying foods is to lower the moisture content in order to reduce water activity and prevent spoilage. Water activity is a critical factor that determines shelf life, and also plays a significant role in determining the activity of enzymes and vitamins in foods and can have a major impact on their color, taste and aroma. Additionally, moisture removal reduces the weight and the bulk of food products to facilitate transport and storage. While imparting these benefits, loss of moisture during drying may also inflict undesirable effects on the product's microstructure, such as non-uniform dried products,

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and slower drying rates (Aktas *et al.*, 2007). The phenomena underlying this process is a complex problem involving simultaneous mass and energy transport in a hygroscopic, shrinking system.

Besides those disadvantages, conventional air-drying is the most commonly used dehydration process in the food and chemical industry, throughout the use of convective dryers (Nijhuis *et al.*, 1998), where the product is exposed to a continuously flowing forced ventilated hot air stream (Rawson *et al.*, 2011; Konishi *et al.*, 2003) producing products with particular characteristics, such as low porosity, high apparent density, case hardening (the formation of a hard outer shell) and shrinkage (Nijhuis *et al.*, 1998).

Shrinkage of foods during drying has an impact on product quality, but if the extension of this phenomenon during drying process is controlled, the quality of the dehydrated product may be improved. For this purpose, a good knowledge of shrinkage mechanism and the influence of process variables, in particular, air velocity, air humidity and products moisture content (Mayor *et al.*, 2004), has to be taken account in order to obtain different textures depending on food product requirement on crispness (Ratti, 1994; Del Valle *et al.*, 1998; Mcminn *et al.*, 1997; Wang *et al.*, 1995; Khraisheh *et al.*, 1997; Lang *et al.*, 1993).

This crispness was mainly related to the crust formation and structural changes, and a large porous structure usually conferred a high crispy product, and small pores collapsed and thick crust formed corresponded to reduced crispness (Pan *et al.*, 2008), and thus to a more shrunk sample, due to the fact that when water is removed from the material, a pressure unbalance is produced between the inner of the material and the external pressure, generating contracting stresses that lead to material shrinkage or collapse, changes in shape and occasionally fracture of the product (Mayor *et al.*, 2004).

In fact, the high temperatures commonly used during industrial air-drying (typically 65–85 °C) cause damage to the microstructure and may also have a negative influence on the color, texture, taste, aroma and nutritional value of the product thereby influencing the quality of dried products (Rawson *et al.*, 2011; Guiné *et al.*, 2011).

Actually, food drying is achieved by means of different techniques which combine heat or pressure sources to remove water from the interior of the product and mechanical energy to remove water from its surface (convection, drip, etc) (Sebastian *et*

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al., 2005). Evaporation of water desiccates solid matrix of the material and migration of water to the surface by capillary and diffusion mechanisms is of critical importance to the moisture loss process (Lewicki, 2006). In dehydration processes, the heat and mass transfer flows can modify physicochemical properties of the material such as volume and shape changes (Mayor *et al.*, 2011).

Dehydration is thus a common method for preserving foodstuffs (Vasquez *et al.*, 1999). Although the influence of hot air drying on food quality is well recognized the understanding of processes caused by dewatering and adversely affecting material properties is limited. This is because evaporation of water at elevated temperature causes chemical, physical and biological changes in food, which can proceed simultaneously or in sequence, some can be advanced while others are just initiated (Arslan *et al.*, 2005). The quality of dehydrated foods is dependent in part on changes occurring during processing and storage. Some of these changes involve modification of the physical structure, affecting texture and appearance. Other changes are due to chemical reactions, but these are also affected by physical structure, primarily due to effects on diffusivities of reactants and of reaction products. The elucidation of the time dependence of mobility changes induced by glass transition seems to be a promising route to optimize and control dehydration processes (Slade *et al.*, 1993).

With literally hundreds of variants actually used in drying of particulate solids, pastes, continuous sheets, slurries or solutions, it provides the most diversity among food engineering units operations (Mayor *et al.*, 2004). The major disadvantages of hot air drying are low energy efficiency and lengthy drying times during the falling rate period. Because of the low thermal conductivity of food materials in the falling rate period, heat transfer to the inner sections of foods during conventional heating is limited (Feng *et al.*, 1998). Some heat-sensitive biomaterials such as fruits, vegetables, and the so-called wellness, or functional foods, requires special techniques to avoid product degradation due to thermal decomposition, oxidation, or enzyme browning (Marques *et al.*, 2005). Thus drying systems optimization is still required nowadays and therefore full understanding of these phenomena can help to improve process parameters and hence product quality, emphasizing on the external and internal process parameters that influence drying behaviour (De Bonis *et al.*, 2008).

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One of the most important physical changes that the food suffers during drying is the reduction of its external volume. Loss of water and heating cause stresses in the cellular structure of the food leading to change in shape and decrease in dimension. Changes in shape, loss of volume and increases hardness cause in most cases a negative impression in the consumer. There are, on the other hand, some dried products that have had traditionally a shrunken aspect, a requirement for the consumer of raisins, dried plums or peaches. Surface cracking is another phenomena that may occur during drying. This happens when shrinkage is not uniform during the drying process leading to the formation of unbalanced stresses and failure of the material (Mayor *et al.*, 2004).

In fact, conventional air drying method, that requires high temperatures and prolonged drying times, has been used to obtain stable dehydrated fruits with low water activity and crisp texture (crispness) (Pan *et al.*, 2008).

MICROWAVE

There is a large market for microwave foods; undeniably the application of microwave heating is of particular interest because of the operational efficiencies it affords (Ala'a *et al.*, 2010).

Microwave energy is not a form of heat. Heat is a secondary effect of an electromagnetic field interacting with matter, such as food. The microwave field changes direction millions of times per second in microwave ovens. The conversion of microwave energy into heat is explained by basically two phenomena: molecules, with a permanent dipolar moment, rotate in the rapidly changing electric field. When molecules rotate in a field that changes polarity at a frequency of many millions of times per second, heat is evolved because of friction forces between the molecules; and charge flow under the action of the field (ionic conduction). When the ions drift, due to the electric field, they collide with other molecules in a billiard ball fashion and heat is evolved because of friction. So, water molecules are polar, i.e. the centre of charge is displaced, which means that they can rotate under the influence of an alternating electrical field and thus easy to heat using microwave (Vega-Mercado *et al.*, 2001). As foodstuffs usually contain 50-97% water, thus food is very well suited for heating and drying with microwave energy (Nijhuis *et al.*, 1998).

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In general, drying causes the moisture to recede inwards from the surface, in particular, conventional systems apply heat at the surface in order to be transferred through a moisture-resistant dry layer for the evaporation of water at the receding waterfront. In a microwave drying system, the microwave can easily penetrate the inert dry layers to be absorbed directly by the moisture at the waterfront. The quick energy absorption causes rapid evaporation of water, creating an outward flux of rapid escaping vapour (Wang *et al.*, 2010).

This technique presents great benefits, such as, uniform heating throughout the material; better and more rapid process controlling (offers instantaneous heat generation and variation power); improved nutritional quality, and desirable chemical and physical effects promoted by the heat generated by the microwaves such as expansion, drying, protein denaturation and starch gelatinization (Ala'a *et al.*, 2010).

Indeed, using microwave energy in the drying of food products causes a more homogeneous dehydration and the developed internal pressure results in less reduction in the volume of the microwave dried product. Also the volume of air within the product is higher in the microwave-treated products than in air dried products with the same bulk volume (Nijhuis *et al.*, 1998). However, while microwave drying promotes quick drying, difficulties in controlling the rapid mass transport may induce to the food texture by “puffing” (Nijhuis *et al.*, 1998; Wang *et al.*, 2010). In reality, some studies with fruits refer after sensory analysis that panellists detected more strongly crispness on microwave-dried fruits than on freeze-dried fruits (Nijhuis *et al.*, 1998).

Microwave heating has advantages over baking or deep fat frying because it is faster due to its volumetric heating nature and microwave-expanded products are generally lower in fat content compared to deep fat fried products (Arimi *et al.*, 2008; Ernoult *et al.*, 2002).

In recent years, microwave drying has gained popularity as an alternative drying method for a wide variety of food products such as fruits, vegetables, snack foods and dairy products (Wang *et al.*, 2010). For instance for plain yogurt (Kim *et al.*, 1995), for cranberries (Yongsawatdigul *et al.*, 1996), for carrot slices (Lin *et al.*, 1998) for model fruit gels, for skimmed milk, whole milk, casein powders, butter and fresh pasta (Al-Duri *et al.*, 1992), for potato slices (Bouraout *et al.*, 1994), for grapes (Tulasidas *et al.*, 1996), for apple and mushroom (Funebo *et al.*, 1998) and ginseng roots (Ren *et al.*,

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1998). Most of the published information on microwave-expanded snack foods relates to pre-extruded cereal based pellets, and these expansion substrates are glassy and low in moisture content (Arimi *et al.*, 2008).

In actuality, microwave-expanded snack foods are becoming increasingly popular, although conventionally, snack foods are expanded either by extrusion, baking or deep fat frying. To date the main area of success for application of microwave ovens in snack food production is the expansion of popcorn at domestic level (Arimi *et al.*, 2008; Lin *et al.*, 1988; Pordesimo *et al.*, 1990; Mohamed *et al.*, 1993; Singh *et al.*, 1999).

Nevertheless, due to mass transport problematic in changing product properties during microwave drying process, more knowledge is needed about the influence of geometry, the size of the product being dried and phenomena like shrinkage, puffing and stress-cracking (Nijhuis *et al.*, 1998).

FREEZE-DRYING

Freeze-drying, also known as lyophilisation (Menlik *et al.*, 2010), is a dehydration process in which water is removed by sublimation of ice from frozen materials (George *et al.*, 2002; Ghio *et al.*, 2000; Rawson *et al.*, 2011), or where a solvent is removed from a frozen solution, suspension or cell structure through sublimation (Rovero *et al.*, 2001). As ice sublimates, the sublimation interface, which starts at the outside surface of the material shrinks back, and porous shell of dried material remains. Vaporized water is transported through the porous layer of the dried material (George *et al.*, 2002). The solid state of water during freeze-drying process protects the primary structure and minimizes changes in the shape of the product, with minimal shrinkage (Marques *et al.*, 2006; Ratti, 2001).

Nevertheless, freeze-drying is far more expensive than convective drying (Nijhuis *et al.*, 1998), due to the fact that the vapour pressure driving force in freeze-drying is very low compared to conventional drying methods, which causes the drying time to be longer and thus the final products have costs relatively higher than those from drying

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processes (George *et al.*, 2002; Lin *et al.*, 1998). However, it is well known that this process produces the highest-quality dried food product (Wang *et al.*, 2010; Anwar *et al.*, 2011, Chen *et al.*, 2000; Esteller *et al.*, 2005; Kopjar *et al.*, 2008; Marques *et al.*, 2009). This is largely because the structure of the food is not severely damaged as in other preservation procedures. In addition, it contributes to preserve constituents as minerals and vitamins, as well as to retain original flavour and aroma (George *et al.*, 2002). It is also generally accepted that the flavour of these freeze-dried foods is better than the air dehydrated products (Krokida *et al.*, 2006).

Hence, technological progress in freeze-drying will concern the reduction of running costs by increasing the process efficiency (reduction of the freeze-drying time, optimization of the operating parameters) while maintaining the quality of the final product (Wolff *et al.*, 1988) and the processing of new products that require a high final quality (Nijhuis *et al.*, 1998). Up till now, freeze-drying process is a well-known and established technology for some time and a lot has been done regarding the research and development (Menlik *et al.*, 2010).

There are two main steps required to transform the raw material to a freeze-dried product. The first is freezing of the raw material when the ice is removed by sublimation directly from the solid to the vapour phase followed by dehydration under vacuum to sublimate ice. The absolute operating pressure is usually lower than the triple point of water, in order to ensure ice sublimation (Genin *et al.*, 1996; Oikonomopoulou *et al.*, 2011). Indeed, the sublimation process is strongly affected by pressure, which determines the ice sublimation temperature. In many cases, at the end of the freezing stage about 65–90% of the initial (at the start of the freezing stage) water is in the frozen state and the remaining 10–35% of the initial water is in the sorbed (nonfrozen) state (Liapis *et al.*, 2002). The freeze-drying pressure and heat input can be manipulated to obtain an appropriate rate of water removal and a desired porosity and density. During this process, ice sublimation causes significant changes in the shape and volume of the food products, and depending on the freeze-drying conditions, the ice crystals which sublimate create pores or gaps with different characteristics Oikonomopoulou *et al.*, 2011; Krokida *et al.*, 1998).

Structural properties of freeze-dried foods are primarily formed in the prefreezing step and affect, e.g., porosity and strength of solids of freeze-dried foods as

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well as entrapment of functional food components. The smaller pores occupied by ice crystals in rapidly frozen materials give a higher resistance to vapour flow and the freeze-drying may need to be heat transfer controlled (Harnkarnsujarit *et al.*, 2012), i.e., ice sublimation rate needs to be reduced to avoid internal ice melting and collapse (Pikal *et al.*, 2002).

In various foods, the solids are in an amorphous metastable state that is very sensitive to changes in temperature and moisture content (Telis *et al.*, 2010). The change from the glassy to the rubbery state occurs at the glass transition temperature (T_g), (Telis *et al.*, 2002), which is specific for each material and affect chemical and physical changes during food processing and storage (Collares *et al.*, 2004) and it is thought that the higher the glass transition temperature the lower the molecular mobility is at a reference temperature (Ohkuma *et al.*, 2008, Levine *et al.*, 1986).

The water activity and the glass transition temperature stay among the parameters which are classically evaluated in the analysis of quality of dehydrated foods (Marques *et al.*, 2007). As the glass transition temperature is dependent on water activity, a change from a glassy to a rubbery state can also occur as a consequence of an increase in the product water content and water activity (a_w) (Moranga *et al.*, 2011). The solid state of water during freeze-drying protects the primary structure and minimizes changes in the shape of the product, with minimal reduction of volume (Ratti, 2001). In the rubbery state, crispy products are observed to undergo a loss of crunchiness becoming texturally unacceptable (Martínez-Navarrete *et al.*, 2004; Ross *et al.*, 1998; Moraga *et al.*, 2011).

In reality, the crispness of cereals has been related to their water content or water activity (Martinez-Navarraete *et al.*, 2004; Katz *et al.*, 1981), and the general conclusion of these studies were the fact that these cereals' crispness is affected when the samples exceeds a critical water activity values which had been reported to be above 0,5, and that at high moisture content, the same snack didn't appear to be brittle or crisp, since at a moisture content of about 10% the product lost all its characteristics crispness (Mazumder, *et al.*, 2007).

In fact, some studies with freeze-dried fruits refer that crispness was mainly related to the crust formation and structural changes, and as a modest crust and a large porous

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structure conferred a high crispy product, so pores in the central region collapsed and thick crust formed corresponded to reduced crispness (Pan *et al.*, 2008). When water is removed from the material, a pressure unbalance is produced between the inner of the material and the external pressure, generating contracting stresses that lead to shrinkage or collapse. This is the reason why drying under vacuum, as in freeze-drying, leads in general to much less shrinkage (Mayor *et al.*, 2004).

Freeze-drying appears, therefore, as a promising technique for dehydration of thermal-sensitive materials (Marques *et al.*, 2007; Sablani *et al.*, 2007), generates minor changes in color, flavour, chemical composition and texture (Nawirska *et al.*, 2009; Guiné *et al.*, 2011). And so, increasingly, it has been used for dehydrating foods otherwise difficult to dry and also foods with high value, such as coffee, vegetables (onions), soups, some aromatic herbs, ingredients for ready-to-eat foods (snacks), certain seafoods and crispy fruits (Liapis *et al.*, 2002; Pan *et al.*, 2008).

I.1.2 – INGREDIENTS

Chemically, foods are mixtures composed of organic compounds, inorganic compounds, and mainly, water. It is now becoming appreciated that, in order to completely deal with the components of foods, it is necessary to have a basic understanding of how they are assembled and disassembled, and therefore is crucial to be acquainted with the chemical and molecular structures of food biopolymers. Molecular functionalities interfere with distinct steps in the process of food microstructure formation. Whereas systems conditions are hard to vary in food textures, the choice of ingredients or balanced modification of the functional groups residing in these ingredients becomes more and more important (Jongh, 2007).

It was only a few decades ago that food scientists started viewing food as structures and materials. This came about at realizing that food components – mainly water, proteins, carbohydrates and fats – by themselves could not explain the richness of textures and tastes of foods with similar composition (Aguilera, 2012).

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Since structure is the combined result of processing and ingredients, the latter could also play an important role. However, it is unclear which components are the main contributors to the crispness of most food products (Roudaut *et al.*, 2002). In effect, ingredients affect the structural organization of products; they are likely to control their mechanical properties and most expectedly their crispness (Barret *et al.*, 1994; Desrumaux *et al.*, 1999; Faubion *et al.*, 1982; Mohamed *et al.*, 1998; Moore *et al.*, 1990; Onwulata *et al.*, 2001; Van Hecke *et al.*, 1998).

PROTEINS

A definition of food protein basically comprises proteins that are consumed by humans for nutritional or textural reasons. For the formation of protein aggregates and bulk networks, the most important functional aspect of proteins is that of generating reactive particles or reactive particles with a lower drive to aggregate by reducing exposed hydrophobicity. The increasing demands on minimal processing of food products opens new potential to better exploit this functionality. The ability of balancing the fraction of network *versus* non-network protein may be a useful strategy to develop high protein foods with well adjustable textural properties (Jongh, 2007). Proteins have natural functional roles, namely they are responsible for building materials for physical structures, chemical activity (e.g., enzymes, transport proteins); or storage nutrients (Aguilera *et al.*, 1999). In many foods they provide the structural elements, and the twenty-some amino acid building blocks of proteins can be arranged in many ways, leading to a broad spectrum of possible protein conformations and resulting structures that food scientist can modify both their structure and behaviour to fabricate protein-based foods (Jongh, 2007).

The ability of a protein to form structures depends mainly upon protein-protein and protein-water interactions. The structure achieved in turn allows certain processes to occur, such as gelatinization, texturization, dough formation, emulsification, and foaming, all of each lead to stable food structures. These processes are complicated in foods because of the intentional or unintentional modification of proteins resulting from processing steps such as heating which can lead to unfolding and association with other components, including carbohydrates and lipids (Aguilera *et al.*, 1999).

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It is a truism that protein conformation dictates function to form structures depends mainly upon protein-protein and protein-water interactions. The ability of a protein to form structures depends mainly upon protein-protein and protein-water interactions. The structure achieved in turn allows certain processes to occur, such as gelation, texturization, dough formation, emulsification, and foaming, all of which can lead to stable food structures. These processes are complicated in foods because of the intentional or unintentional modification of proteins resulting from processing steps such as heating, which can lead to unfolding and association with other components, including carbohydrates. Engineering proteins to create desirable physical properties and to facilitate materials applications is certainly within the realm of current knowledge (Aguilera, 1999).

In some studies about the effect of the addition of different ingredients on the characteristics of a batter coating, high protein content in batter flour has been associated with batter-fried foods with greater crispness (Olewinik *et al.*, 1993). In fact, in batters, gluten proteins enhance the retention of the gases formed by the leavening agent, resulting in a lower density and, consequently, a more porous and crunchy final batter texture. In particular, the addition of gluten is traditionally associated with greater adhesion and crispness in the final product (Breuil, 2001). The addition of egg albumin to rice flour-based batter improves crispness, while the addition of yolk increases the firmness of the fried batter (Mohamed *et al.*, 1998).

The interactions between proteins, polysaccharides and water have been suggested to play a role in the crispness of some products (Mohamed *et al.*, 1998; Van Hecke *et al.*, 1995).

POLYSACCHARIDES

One of the major achievements in food science in the past few decades has been an increased appreciation of the role of the polysaccharides in food structuring. The wide range of rheological behaviour demonstrated by polysaccharides in solution is due to the variety of possible conformations and chain flexibility (Morris, 2007). The gelation mechanism depends on the polysaccharide but invariably involves the physical entrapment of water in a three-dimensional network of ordered polysaccharide chain

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segments. Polysaccharides are used primarily to modify texture through thickening or gelling (Aguilera *et al.*, 1999), but also to stabilise emulsions, foams and dispersions or to generate texture (Morris, 2007).

There are many polysaccharides that are not yet exploited commercially, but are used extensively in traditional local recipes (Hussein *et al.*, 2011). These carbohydrate materials usually come from plants and their functional properties as food hydrocolloids remain largely unexplored.

Hydrocolloids are well known for their wide use in for many products provided by many branches of the food industry. More and more they play an important role in many applications of the avant-garde cuisine, where according to many chefs their real strength is determined by their isolated use, rather than in combinations with other food thickeners and gelling agents. Indeed their thoughtful use in dishes and food systems allows developing sensible physical and more systematic “models for taste” in (molecular) gastronomy. However, since their use in “molecular cooking” their special features became available to new applications and the products are used on a practically basis by a broader public (Adriá, 2005; Vilgis, 2012).

Several benefits in controlling texture and sensory properties of foodstuffs, rheology of aqueous solutions of hydrocolloids come from polysaccharide-polysaccharide interactions in their blends (Gibinski *et al.*, 2006). Polysaccharides such as cellulose, hemicelluloses, pectin substances (Albert *et al.*, 2002), starch, plant gums and bacterial gums (Morris, 2007) provide textural attributes such as crispness, hardness, and mouthfeel to many foods. Many can form gels that will provide microstructure and also enhance viscosity of solutions owing to their high molecular weight (Aguilera *et al.*, 1999).

These compounds are widely used as additives in the food industry (Guarda *et al.*, 2004), since they are multifunctional ingredients that are added to control and improve functional properties (Shalini *et al.*, 2007 Lazaridou *et al.*, 2007, Rosell *et al.*, 2007), and emulsion stability (Rimac-Brneié, *et al.*, 2004), and also add flexibility, functioning as texturizers (Vilgis, 2012) and adhesives (Shalini *et al.*, 2007) , and also reducing oil absorption during deep fat frying (Ang, 1992; Koelsch *et al.*, 1992; Nelson *et al.*, 1991; Williams *et al.*, 1999).

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In some studies, the use of dextrans in batter-fried foods has been associated with increased crispness in the final product, in particular in batter-fried squid rings produces a prolonged period of crispness retention after frying (Baixauli *et al.*, 2003). In fact, in a study about the effect of the addition of different ingredients on the characteristics of a batter coating for fried seafood without a pre-frying step, dextrin was the ingredient characterized by the highest crispness contribution (Salvador *et al.*, 2005). In fact, dextrin and cellulose fiber may act as crisping agents in batter. Crispness has been also found to be positively correlated with amylose content (Altunakar *et al.*, 2004). Starch resistance to gelatinization and granule disintegration are linked to a crisper fried batter (Matsunaga *et al.*, 2003). Indeed, incorporating a source of resistant starch increases the sensory score for crispness (Yue *et al.*, 1998) and some influence in the crispness profile of batter crust, mainly reflected by an increase in hardness and fragility of the final battered foods (Varela *et al.*, 2008).

I.2 – CRISPY TEXTURE EVALUATION

Texture is of paramount importance in most foods, contributing at least to half the enjoyment of food. There are several food products for which acceptance by consumers relies more on texture than flavour, as a good example are potato crisps, where “crispness” tells us about their quality and ageing (Rojo *et al.*, 2009).

To evaluate foodstuff crispy texture there are different direct and/or indirect methodologies available. The direct measurement methods include an objective instrumental texture and sound analysis with an equipment design specifically for that purpose – the texturometer, and also sensory analysis, by means of an subjective but reliable trained panel or untrained group of individuals. Indirect methods are related to structure and compositional intrinsic characteristics, namely moisture, protein, carbohydrates, fat or fiber content, and thus, those compositional parameter's analysis can be obtained by specific techniques and structure can be analyzed by different microscopy techniques or images scanning analyses or even by porosimetry methodologies, depending on foodstuff nature, and which of this mentioned methods will be describe in future sections.

In effect, the type of tests that are used for measuring food texture may be divided into objective tests that are performed by instruments and sensory tests that are

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performed by people. Objective tests can be divided into direct tests that measure real textural properties of materials, and indirect tests that measure physical properties that correlate well with one or more textural properties. Sensory tests can be classified into oral (those tests that are performed in the mouth) and nonoral (in which some part of the body other than the mouth is used to measure the textural properties) (Bourne, 2002).

Related to the previous subject, it seems important to remember that texture is primarily the response of the tactile senses to physical stimuli the results from contact between some part of the body and the food. The tactile sense (touch) is the primary method for sensing texture but kinaesthesia (sense of movement and position) and sometimes sight (degree of slump, rate of flow), and sound (associated with crisp, crunchy and crackly textures) are also used to evaluate texture. All five of the human senses (sight, hearing, taste, odour, touch) are important and have been, and continue to be the subject of considerable research effort. Although we do not have an entirely satisfactory definition of texture we can say with a high degree of confidence that texture of foods has the following characteristics (Bourne, 2002):

- (1). It is a group of physical properties that derive from the structure of the food;
- (2). It belongs under the mechanical or rheological subheading of physical properties. Optical properties, electrical and magnetic properties, and temperature and thermal properties are physical properties that are excluded from the texture definition;
- (3). It consists of a group of properties, not a single property;
- (4). Texture is sensed primarily by the feeling of touch, usually in the mouth, but other parts of the body may be involved (frequently the hands);
- (5). It is not related to the chemical senses of taste or odour;
- (6). Objective measurement is by means of functions of mass, distance, and time only.

Three classes of texture characteristics are often distinguished: mechanical, geometrical and others. The later ones are those related to the moisture and fat content of the food as perceived by the human senses. For solid products mechanical and geometrical characteristics are especially evaluated during the first bite and the chewing

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stage while “others” involve changes in evaluated characteristics during mastication, bolus formation, swallowing, and oral clearance (Van Vliet *et al.*, 2009).

Only humans can assess the textural quality of food. Instruments that measure physical properties are widely used and have led to great improvements in building and maintaining a high level of textural quality in most of our food supply (Bourne, 2002). Because the stimulus in texture perception is mainly mechanical, texture evaluation is done by mechanical measurements (Thussu *et al.*, 2012). Nevertheless, instrument readings are worth little unless calibrated against the human senses.

Sachs (1988) called touch ‘the intimate sense’ and described its importance in the following words: “Throughout the first few days of life, the baby continues to be most affected by the things that touch him: a soft blanket, warm breast, a firm bed. Even after he has begun to favour sight, he relies heavily on this most intimate of senses to gather information about her environment. If he spies, say, an alphabet block in her crib, she will pick it up, turn it over in her hands, then put it in his mouth – not, as one might think, to taste the block but to touch it with her lips and tongue, regions of the body that are particularly sensitive to tactile stimuli. He uses his sense of touch, which is not easily fooled, to confirm his sense of sight, which, even when fully mature, is subject to all manner of illusions. The fundamental nature of touch is even more apparent when the sense is deprived of stimulation. Being unable to hear or see does not prevent one from attaining a happy and fruitful existence....But an existence devoid of tactile sensation is another matter: sustained physical contact with other humans is a prerequisite for healthy relationships and successful engagement with the rest of one’s environment....And among humans, denial of physical contact during the first years of life can cause virtually irreversible states of withdrawal. Touch, in short, is the core of sentience, the foundation for communication with the world around us, and probably the single sense that is as old as life itself” (Bourne, 2002).

Crispness represents the key texture attributes for dry snack products and is perceived through a combination of tactile, kinesthetic, visual and auditory sensations (Mazumder *et al.*, 2007).

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This attribute is also associated with rapid drop of force during mastication process that, in turn, is based on fracture propagation in brittle materials (Vincent, 1998).

When force is applied to brittle snacks, rupture of the cellular structure occurs and, in line with some deformation, generates a typical sound contributing to crispness sensation. Thus, perceived crispness strongly affects consumer satisfaction (Salvador *et al.*, 2009). Therefore, efforts have been made to investigate and measure the crispness. Hence, mechanical tests serve as objective and efficient evaluation procedures (Taniwaki . *et al.*,2012).

I.2.2 – INSTRUMENTAL METHODS

TEXTURE

One of the most important challenges currently facing food producers is the measurement of the texture of their products (Kealy, 2006).

Szczesniak *et al.* (1975) reported that time of day exerted a strong influence on textural awareness and flavor. At breakfast, most people prefer a restricted range of familiar textures that lubricate the mouth, remove the dryness of sleep, and can be swallowed without difficulty. New or unfamiliar textures, and textures that are difficult to chew, are not wanted at breakfast. People are willing to accept a wider range of textures at the midday meal just so long as it is quick and easy to prepare and not messy to eat. After all, this is a practical meal with a limited time for preparation and consumption. Texture is most appreciated and enjoyed at the evening meal. This is the time for relaxation, which comes after the day's work and, for most people, is the largest meal of the day when several courses are served and a wide range of textures is expected and relished.

Scott-Blair (1958) categorized the instrumental techniques used to measure food texture into three groups: empirical tests, which measure something physical under well-defined conditions, imitative tests, which attempt to simulate the conditions to

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which the material is subjected in the mouth and fundamental tests, which measure well-defined physical properties such as viscosity or elastic modulus (Rosenthal, 1999).

The fundamental tests measure well-defined rheological properties. Before attempting to use this class of test on foods, it should be borne in mind that they were developed by scientists and engineers interested in the theory and practice of materials of construction, and they may not be very useful in measuring what is sensed in the mouth when food is masticated (Bourne, 2002). Fundamental tests are generally slow to perform, do not correlate as well with sensory evaluation as do empirical tests, and use expensive equipment. They are not used to any great extent in the food industry but they do have a place in some research laboratories (Szczesniak (1963).

The empirical tests measure parameters that are poorly defined, but from practical experience are found to be related to textural quality. This is the most widely used class of instruments in the food industry. The tests are usually easy to perform, rapid, and frequently use inexpensive equipment (Bourne, 2002).

The imitate tests uses the conditions to which the food material is subjected in practice. This class may be considered as a subtype of empirical test because the tests are not fundamental tests. Examples of this kind of test are the Farinograph and other dough-testing apparatus that imitate the handling and working of bread dough and the Bostwick Consistometer. The ideal texture measuring apparatus should combine the best features of the fundamental, empirical, and imitative methods and eliminate the undesirable features of each of these. At the present time there is no ideal texture measuring equipment or system. Empirical methods are used almost. Force measuring instruments are the most common of the texture measuring instruments. Because of their multiplicity, this heading is broken into the subclassifications (a) puncture, (b) compression–extrusion, (c) cutting–shear, (d) compression, (e) tensile, (f) torsion, (g) bending and snapping, and (h) deformation (Bourne, 2002).

In reality, food texture is determined by various tests such as compression, penetration and snap, and the resulting force-displacement plot (Iliassafov *et al.*, 2007) which is given by texturometer software. Bending and snapping tests are usually applied to food that is in the shape of a bar or sheet, and a three point bending probe is used (figure 1). Puncture testers embody one of the simplest types of texture measuring

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instruments and one of the most widely used. Puncture testing instruments are all maximum-force instruments. They may be classed into single-probe instruments. The puncture test measures the force required to push a punch or probe into a food. The test is characterized by (a) a force measuring instrument, (b) penetration of the probe into the food causing irreversible crushing or flowing of the food, and (c) the depth of penetration is usually held constant (Bourne, 2002).

The crisp final texture of fried products can be evaluated by means of instrumental and sensory techniques. Parameters such as crispness or crunchiness, fragility, tenderness, etc., are hard to quantify using empirical mechanical methods because what is perceived in the mouth is a complex sum of sensations (Mohamed *et al.*, 1998).

Definitely, crispness “measurements” are performed on instrumental on instrumental originally, developed for material science, providing physical parameters with fundamental significance in terms of rheological properties. These parameters cannot give straightforward crispness measurement, if any, but can be used as indicators, provided they are validated by sensory data (Roudaut *et al.*, 2002).



Figure 1 – Texturometer TAHDi (Stable Micro Systems (UK))

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Considering the perception of crispness upon eating, large deformation and fracture tests seem to be the most suitable instrumental tests. However, small deformation data, such as those acquired in dynamic rheology (Roudaut *et al.*, 1998; Le Mestre *et al.*, 1996; Nikolaidis *et al.*, 1996; Georget *et al.*, 1996) or prior to fracture at larger deformation (Fontanet *et al.*, 1997; Nicholls *et al.*, 1995), may provide information not directly related to crispness, but to the molecular basis of this attribute. Independently of the probe type or of the method used, they are all based on recording the force when a deformation is applied to the product. In crispy products, force-displacement plot shows a numerous and neighbouring fractures, and when crispness decreases (after rehydration for example), the number of fractures decreases which increases the width of the peaks on the force-deformation plot (Roudaut *et al.*, 2002).

The most commonly used tests can be categorized into three groups: flexure (Vickers *et al.*, 1980; Van Hecke *et al.*, 1995; Attenburrow *et al.*, 1992; Andersson *et al.*, 1973), shear (Faubion *et al.*, 1982; Bhattacharya *et al.*, 1987) and compression tests. The latter are probably the most commonly employed because of their similarities with the mastication process. In these tests, the specimen is compressed either between two parallel plates (Moskowitz *et al.*, 1974) or by a plunger compressing the sample held in a cylinder (Andersson *et al.*, 1973). Measurement of puncture with a plunger is the technique most used (Mohamed *et al.*, 1998). Samples can be tested individually or as bulk when contained in a cell (Nixon *et al.*, 1995). Puncture tests have been extensively used as well (Hayter *et al.*, 1988; Hutchinson *et al.*, 1987; Li *et al.*, 1998; Van Hecke *et al.*, 1998) for they stimulate the incisors impact at biting (Roudaut *et al.*, 2002). The compression–extrusion test consists of applying force to a food until it flows through an outlet that may be in the form of one or more slots or holes that are in the test cell. The food is compressed until the structure of the food is disrupted and it extrudes through these outlets. Usually the maximum force required to accomplish extrusion is measured and used as an index of textural quality. The Ottawa Texture Measuring System (OTMS) uses a forward extrusion test because the food moves in the same direction as the plunger. The standard cell of the Food Technology Texture Press (Kramer Shear is mixed; half the food is extruded forward through the slits in the bottom of the cell and the other half is extruded backwards up between the descending blades (Bourne, 2002).

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Shear compression tests have been used to measure the instrumental texture of fried crust of battered food in a Kramer cell applied up to breaking point; specific shear force and toughness (work or area under the loading portion of the curve/mass) were calculated. The curves force-deformation in crisp materials is usually irregular (Mazumder *et al.*, 2007). Compression is the most common method to determine the texture of foods, and it has been applied on snacks (Vincent, 1998). However, some authors claim that compressive force alone is insufficient to describe accurately the texture of crispy products (Guraya *et al.*, 1996).

Measurements of the acoustic and mechanical properties of a food during crushing (compression-test), provides information about its crispness (Iliassafov *et al.*, 2007). In fact, since crispness has an auditory component, it is not surprising that some methods, developed to study crispness, have focused on the sounds generated at fracture, the sound being recorded during instrumental crushing or during mastication (Roudaut *et al.*, 2002).

SOUND

Two approaches have been taken to study noisy textures using acoustic techniques. One is a measure of the perception of air-conducted sounds to establish the contribution of these sounds to the sensation of crispness and crunchiness (Christensen *et al.*, 1981; Vickers, 1981; Vickers, 1985). The second is to record the sounds produced during application of a force to a noisy product to obtain quantitative information regarding the crisp, crunchy or crackly sounds (Drake, 1963; 1965; Edmister *et al.*, 1985; Dacremont *et al.*, 1991; Lee *et al.*, 1988; Seymour *et al.*, 1988).

Auditory texture is to a large extent synonymous with crispness, crunchiness, and crackliness in foods. The early work in this area was done by Vickers *et al.* (1976). Lately there has been a resurgence of interest in the area with a review by Duizer (2001), and work by Luyten *et al.* (2004), Salvador *et al.* (2009), and Varela *et al.* (2009). Sounds are produced by mechanical disturbances which generate sound waves which are propagated through the air or other media, such as bone conduction from the jaw bone to the bones of the middle ear (Dacremont, 1995). Crisp and/or crunchy foods

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fall in two categories, namely wet foods and dry foods. Sound generation differs in these two types of foods (Vickers, 1979).

Wet crisp foods, like fresh fruits and vegetables, are composed of living cells that are turgid if enough water is available. When the structure is destroyed, by breaking or chewing, the cells pop and this produces a noise. The amount of noise produced is less when the surface tension of the liquid is high. On the other hand, exposing dry crisp foods, like cookies, crackers, chips, and toast to moisture (humid air) decreases the perceived crispness of the food. These products have air cells or cavities surrounded by brittle cell or cavity walls. When these walls are broken any remaining walls and fragments snap back to their original shape. When the walls snap back vibrations are caused that generate sound waves (similar to a tuning fork). When the moisture content of dry crisp foods increases, the walls are less likely to snap back and the amount of sound generated is less. Vickers (1981) and Christensen *et al.* (1981) showed that crispness and crunchiness of specified foods can be rated on the basis of sound alone, on the basis of oral–tactile clues alone, or on the basis of a combination of auditory and oral–tactile information (Lawless *et al.*, 2010). The sound emitted by crisp foods depends on several macroscopic and microscopic factors within the food. Structure has a large impact on the sounds produced when biting into products with crisp, crunch and crackly textures. So, within a “noisy” product, the arrangement of cells, chemical bonds, impurities and existing cracks, all affect sound production (Al Chakra *et al.*, 1996).

Therefore, these acoustic measures have also been combined with force-deformation measures to predict the sensations of crispness and crunchiness (Vickers, 1987; Szczesniak, 1988). It has been found that the combination of acoustic and mechanical techniques more adequately describes food sounds than either technique alone (Vickers, 1987).

Within humans, the sound wave produced upon the breakage of cell walls can be detected by air conduction to the ear and bone conduction (Van Vliet *et al.*, 2009) through the mandible as well as through the soft tissue of the cheeks and tongue (Duizer, 2001). The idea that auditory sensations are important for texture perception was first introduced by Drake (Drake, 1963; Drake, 1965) as a means of progressing texture research. There are a number of different textures that have an auditory component including crispness, crunchiness and crackliness. Of these, the most studied

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textural property is crispness. It is claimed to be one of the most important attribute affecting consumer acceptability (Szczesniak, 1990).

Crispness is a two components phenomenon: oral and aural. Therefore, it has been suggested that crispness may be evaluated by mechanical and acoustical properties (Edminster *et al.*, 1985; Mohamed *et al.*, 1982; Vickers, 1988). If a crisp product does not produce the expected sound upon biting, then it is considered to be stale and of poor quality or has been produced using inappropriate ingredients or processes (Duizer, 2001). If a crisp product does not produce the expected sound upon biting, then it is considered to be stale and of poor quality or has been produced using inappropriate ingredients or processes. Products which contain fluid within their cells, such as apples, have been termed wet crisp products, while cellular products containing only air within their cells, such as cheese balls or potato chips are termed dry crisp products (Duizer, 2001). Definitely, the mechanism of sound production differs between wet-crisp and dry-crisp food. Wet-crisp food is comprised of living plant cells, having a certain internal pressure, the turgor. When a turgid cell bursts, its contents expand rapidly, producing a sound. In a dry-crisp product, the cells are usually filled with air and the cell walls are brittle. The sound pressure wave is generated by the snap back of the remainder of the cell walls after they have been bent and broken (De Belie *et al.*, 2003).

Crisp and crunchy foods generate characteristic sounds when masticated. One person can stand behind a screen out of sight of a second person and chew on various foods and the second person can quickly decide when a crispy food is being chewed just by listening to the sounds being generated (Bourne, 2002). In fact, crispier samples produce more total noise. This could be the result of higher sound amplitude or a greater density of sound occurrence. In order words, changes in either one or both of the two parameters, (1) the loudness of the sounds produced and (2) the number of sounds produced in a given biting distance, seemed to cause a change in perceived crispness (De Belie *et al.*, 2002).

In several food structures, cell wall fracturing releases the internal pressure finally detected as the typical crispy sound. On this basis, recording sounds and stress patterns produced during the force application is an experimented way to obtain quantitative information regarding crispy sounds and to predict the sensorial appreciation of crispness (Ballabio *et al.*, 2012).

MICROSTRUCTURE

Foods have a structure which is imparted by nature or through processing. It is important to know the level or scale of structure: one could be macroscopic level (macrostructure) and another could be microscopic level (microstructure) (Aguilera, 2000). Structure has a large impact on the sounds produced when biting into products with crisp, crunch and crackly textures. The sound emitted by crisp foods depends on several macroscopic and microscopic factors within the food. Microscopically, within a “noisy” product, the arrangement of cells, chemical bonds, impurities and existing cracks, all affect sound production (Al Chakra *et al.*, 1996). Food microstructure is the spatial arrangement and interactions of identifiable elements in a food, whose sizes are <100 μm . For a physicist the range between the molecular size and the macroscopic scale is typical of soft condensed matter, or matter in a state between a liquid and a crystalline solid (Aguilera *et al.* 1999).

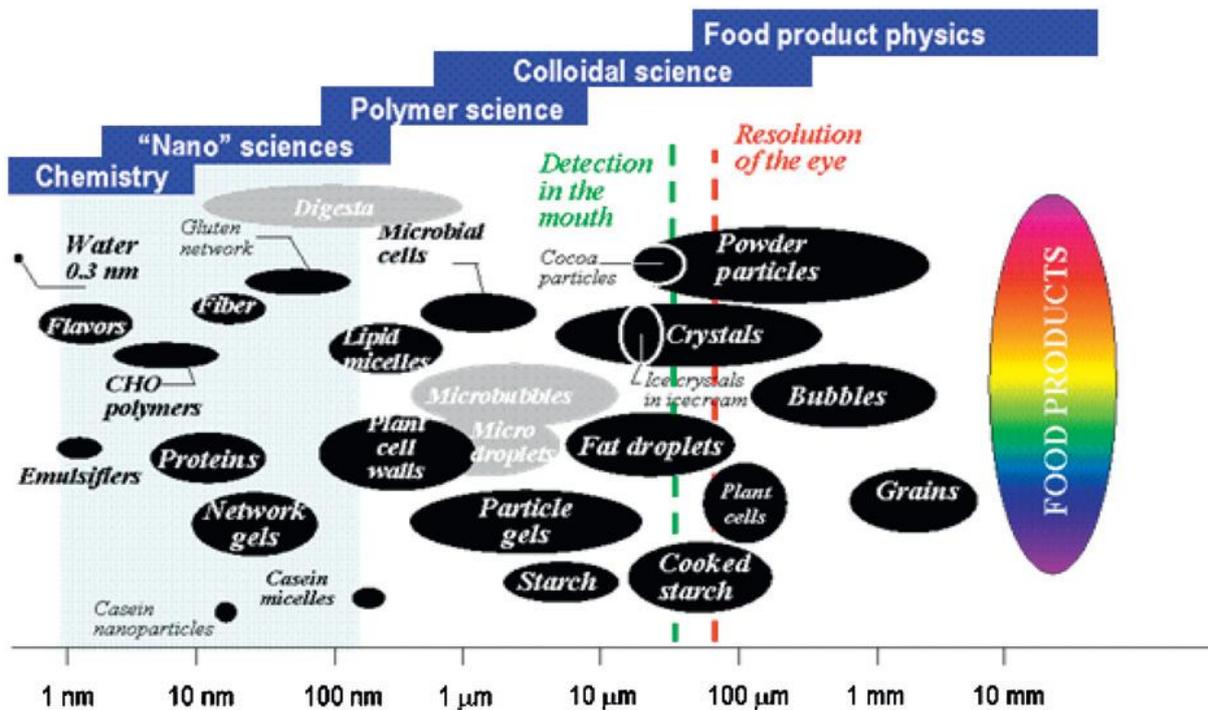


Fig.2. Important structural elements related to foods and their approximate sizes. Dotted lines show the upper limit for particles to go undetected in the mouth and the minimum size that can be resolved by the naked eye. Gray area is the size range of nanosciences (Aguilera, 2012).

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In figure 2 several important structural elements related to foods, their approximate sizes as well as the sciences behind the phenomena at each length scale are shown. The dimensions from molecules to products span almost eight decades. It is unfortunate that most of the structural engineering inside our foods occurs at sizes below 100 μm , being invisible to the naked eye (Aguilera, 2012). At these dimensions molecules may participate in the formation of emulsions, viscous polymer solutions, gels and glasses. Thus, foods can be classed as soft matter but their multi- component nature and complexity set them apart from other forms of soft matter present in our daily life (Mezzenga et al., 2005).

Textural responses are governed by structural organization, and so, alterations in structures are reflected by corresponding shifts in texture. In other words, it is the organization of the structural elements that is the primary determinant of texture. Consequently, this fact underscores the need to accompany textural studies with structural examination, whose goal is determining to which microstructure (s) the instrumental probe or human tooth responds (Aguilera *et al.*, 1999)

Although it is not possible to fully predict textural responses from structural information alone, the knowledge gained can go a long way in explaining processing effects and in selecting processing strategies. Therefore, microstructure has major roles as the determinant of appearance, functional properties and stability of foods, as emphasized by several authors describing relationships between food properties and structure at macro and molecular levels (Aguilera *et al.*, 2008).

Modern microscopy techniques assist in visualization of the structures and in combination with microanalytical methods and image processing may provide the needed data at the required scale (Kalab *et al.* 1995; Rahman *et al.* 2002). One of the most dangerous pitfalls of microscopy is our tendency to find what we are looking for. In order to make valid judgments, we need to augment our imaging apparatus with unbiased image analysis tools so that we can reliable quantitative information and numerical data from an image (Aguilera *et al.*, 1999).

IMAGE ANALYSIS

Image analysis relies heavily on computer technology to recognize, differentiate, and quantify images. Traditionally, the structure of foods was studied by means of images enhanced by glass lenses. The information thus obtained was used to augment that gained by direct macroscopic evaluation with the unaided eye and the sense of touch. Any time that an effort is made to gain knowledge from an image of a real object, there exists a serious risk of misunderstanding resulting from artifacts of magnification or sample preparation as well as psychological errors of interpretation. It is not within the scope of this work to detail the basic physical and optical principles that govern magnifying instruments, and the reader is referred to the numerous texts available on this subject (Aguilera *et al.*, 1999). Image analysis starts with a picture obtained using one of the following techniques: light microscopy (LM), transmission electron microscopy (TEM) and scanning electron microscopy (SEM) and others instrumentation such as magnetic resonance imaging (RMI), X-Ray or digitalized images without magnification (Aguilera *et al.*, 1999). Among the most widely used imaging techniques in microstructural food research are light microscopy (LM), transmission electron microscopy (TEM) and scanning electron microscopy (SEM) (McClements, 2007)).

These three mentioned above techniques are invasive in nature, since if the internal structure of a sample is to be observed, then it has to be cut in order to expose the inner zones (McClements, 2007). Modern image analysis systems begin by transforming an analog signal, such as a film-based "hard copy," into a digital "soft copy." The transformation may be achieved in a variety of ways, depending upon the resources available and the end use of the image, but in every case the result is a set of pixel values. The devices used for capturing images include cameras, scanners, and other equipment (Aguilera *et al.*, 1999). Although not directly related to food microstructure, it is relevant to point out that both scanners and digital cameras can also be used as stand alone imaging systems by food researchers when high levels of magnification are not required. There are various recent scientific literatures that prove the multiple uses of flat bed scanners for the evaluation of macroscopic food characteristics using image analysis (Riva *et al.*, 2005; Esteller *et al.*, 2006).

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The digital image in its original form, whether color or monochrome, is referred to as a gray scale image. With most modern equipment, 256 gray levels are available. Thus, in a typical image whose dimensions are 512 pixels X 512 pixels, each pixel has an integer value ranging from 0 (black) to 255 (white). The acquired gray scale image undergoes image processing for the discrimination of important features. The images can be additionally processed by thresholding to create a binary image that can be further processed by binary image editing. Segmentation divides the image into regions of structures intended for analysis. Object selection is followed by measurement, analysis and the collection of quantitative or qualitative data. The data are finally subjected to statistical analysis and, depending upon circumstances, used to support a stereologic interpretation and conclusions about the structures of interest (Aguilera *et al.*, 1999).

Segmentation may be the most important part in image processing, and refers to the process of extracting the desired object of interest from the background. When the image is segmented, every pixel in the image is included as an object or part of the background, and thus, pixels of an object form a connected region and have similar intensity values (McClements, 2007). This process of segmentation may be done by manual or automated methods and may be applied to an original image, to an image following filter transformation, or to a binary image (Aguilera *et al.*, 1999).

SCANNING ELECTRON MICROSCOPE (SEM)

Modern microscopy techniques assist in visualization of the structures. In combination with micro-analytical methods, image-processing techniques may provide additional insight on the structure (Kalab *et al.*, 1995). The sample preparation for microscopy, however, is tedious and complex, and in many cases it is difficult to maintain the sample at its original condition due to the initial cutting and preparation steps. Moreover, it always gives a two-dimensional picture although multiple sections at different locations could be translated to the three-dimensional picture (Karathanos *et al.*, 1996; Rahman *et al.*, 2002). The scanning electron microscope (SEM) was welcomed by food researchers in the mid- to late 1960s and brought the promise of an apparently three-dimensional picture and a great depth of field, 500 times that of a light microscope at the same magnification. Depth of field is the distance along the lens axis

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in the object plane at which an image can be focused without loss of clarity. The SEM also did away with the need to cut thin sections and allowed the examination of topographical details of external or internal surfaces. The SEM fills and overlaps the magnification gap between the LM and the TEM, from 20X to 100,000X. Shortly after the debut of the first commercial SEM, scientists began examining food specimens, and they have continued to do so at an increasing rate over the last several decades (Morris, 2007).

Innovations continue to be made in microscope technology. In the case of light microscopy, recent advances include the application of lasers, video enhanced imaging, confocal illumination, and tandem scanning microscopy. In the case of electron microscopy, advances include the medium voltage TEM, low kV SEM operation, and several ancillary methodologies, such as backscattered electron analysis and X-ray microanalysis. The food scientist can now apply several techniques for revealing structural organization that complement microscopic studies. In fact, the new approach to investigating food microstructure is to integrate structural data from many devices focused at different levels of organization. This approach has resulted in exciting new insights, especially insights into the relationship between microstructure and other important food characteristics, such as quality factors and food processing strategies. Thus, microscopy techniques have the advantage that the particle contour, shape, state of the dispersion, and even color can be observed. Images seen in a microscope are projected areas whose dimensions depend on the particle orientation on the slide, since particles in their stable orientation tend to present the maximum area, and hence sizes measured by other microscopy tend to be larger than those measured by other methods (Aguilera *et al.*, 1999).

Shapes are physical dimensional measurements that characterize the appearance of an object. Shape features can be measured by combining size measurements, and are relatively simple to determine using image analysis techniques. Some of the most widely used shape descriptors are elongation (McClements, 2007), roundness, compactness, aspect ratio, curl, convexity and extent (Aguilera *et al.*, 1999).

For example, roundness can be used to follow shrinkage of cells during drying or to follow particle deformation during caking (Aguilera *et al.*, 1999).

POROSOMETRY, DENSITY and PYCNOMETRY

Cellular solids contain different ordered and disordered structures (Nussinovitch *et al.*, 2004), and many foods are cellular solids (Nussinovitch *et al.*, 1992), such as breakfast cereals, wafers, cakes, bread, biscuits, chips, and dried and extruded product which consist of a porous structure that may be described as solid foam (Rahman *et al.*, 2003). These products behave like solid foams, or to be precise, like elastic or plastic materials consisting of a continuous solid phase in which a discontinuous air phase is dispersed. Their typical texture is due to the combination of three means of aeration at different stages of the process: use of chemical raising agents at the formulation stage, whipping or gas injection during the mixing stage and thermal expansion during baking (Edoura-Gaena *et al.*, 2007). Often these products are described as crunchy because of a complex failure mechanism that involves the repetitive deformation and fracturing of the cell structure. So it is critical in food product development to analyze the relationships between structural and textural characteristics (Lazou *et al.*, 2010). In fact, microstructure has major roles as the determinant of appearance, functional properties and stability of foods (Harnkarnsujarit *et al.*, 2012). Structure of food has been related to quality change experienced during food processing, especially at microscopic scale (Mellema, 2003; Donald, 2004; Aguilera, 2005). Structural properties, like density and porosity, can be used to characterize the texture and quality of dehydrated products (Oikonomopoulou *et al.*, 2011). The most common terminology used in characterizing the pores is the porosity, which is defined as volume fraction of pores relative to total volume (Rahman *et al.*, 2002). Mechanical and textural properties of food are correlated to the porosity and other characteristics of pores (Gogoi *et al.* 2000; Hutchinson *et al.* 1987; Scanlon *et al.* 1998; Vincent, 1989). The variation in porosity, average pore size, pore size distribution, and specific surface area has also significant effects on the mechanical, textural, and other quality characteristics of the dried foods (Huang *et al.* 1990; Karathanos *et al.* 1993; Farkas *et al.* 1991). Besides the porosity, pore size distribution plays a crucial role. And it can be estimated by image analysis of two dimensional images (Regier *et al.*, 2007), and in addition, mercury porosimetry and helium pycnometry have also been employed for characterization of pores (Du *et al.*, 2006).

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There has been renewed interest in studying microstructural properties of food such as porosity and pore size distribution and in better understanding of transport properties of foods and optimization of various processes that lead to production of porous foods (Pinthus *et al.*, 1995; Rahman *et al.*, 2002; Aguilera, 2005; Witrowa-Rajchert *et al.* 2006; Adedeji *et al.* 2009). Information on porous formation in foods during processing is need for process design influencing a wide variety of other properties, such as mechanical properties, thermal conductivity, thermal diffusivity and diffusion (Koc *et al.*, 2008; Rahman *et al.*, 2001). Indeed, pores occur in a variety of food products and have a significant effect on their qualities. The variation in porosity, average pore size distribution influences the mechanical and textural characteristics of dried foods significantly (Du *et al.*, 2006).

Food microstructure is the spatial arrangement and interactions of identifiable elements in a food, whose sizes are $<100\ \mu\text{m}$ (Aguilera *et al.* 1999). Porosimetry, unlike some other techniques, provides information on bulk and apparent densities, pore size distribution, pore area, pore shape (hysteresis phenomenon) and a wide range of porosity between 0.005 and $360\ \mu\text{m}$. This technique has been used extensively to study pore characteristics of dried foods (Karathanos *et al.*, 1996; Rahman *et al.*, 2002).

There are several techniques that have been used to study pore characteristics of foods. These include pycnometry (McDonald *et al.*, 2001; Rahman *et al.*, 2003; Kassama *et al.*, 2005; Taiwo *et al.* 2007), microscopy (Bouchon *et al.*, 2001; Liang *et al.*, 2006), x-ray computed tomography (Barcelon *et al.*, 1999; Van Dalen *et al.*, 2003, 2007; Lim *et al.* 2004; Kim *et al.*, 2007), magnetic resonance imaging (MRI) (Bows *et al.*, 2001; Wagner *et al.*, 2008), mercury porosimetry (Karathanos *et al.*, 1993; Karathanos *et al.*, 1996; McDonald *et al.*, 2001; Ngadi *et al.*, 2001; Rahman *et al.*, 2002; Rahman *et al.*, 2003; Kassama *et al.*, 2005) and gas adsorption (Karathanos *et al.*, 1996).

Mercury porosimetry particularly has been a useful quantitative tool for identification of the microstructure of dehydrated food products (Karathanos *et al.*, 1996; Rahman *et al.*, 2002). The principle of mercury intrusion porosimetry (MIP) is based on capillary law, non-reactive and non-wetting characteristics of certain liquid such as mercury. It is such that this liquid will not penetrate a pore until certain pressure is applied (Adedeji *et al.*, 2010). So, this method as well as helium pycnometry are destructives, laborious and inherently subjective (Du *et al.*, 2006).

I.2.1 – SENSORY METHODS

Sensory evaluation is a science of measurement. Like other analytical test procedures, sensory evaluation is concerned with precision, accuracy, sensitivity, and avoiding false positive results (Meiselman, 1993). The human senses have been used for centuries to evaluate the quality of foods. We all form judgments about foods whenever we eat or drink, which does not mean that all judgments are useful or that anyone is qualified to participate in a sensory test. In the past, production of good quality foods often depended upon the sensory acuity of a single expert who was in charge of production or made decisions about process changes in order to make sure the product would have desirable characteristics. This was the historical tradition of brewmasters, wine tasters, dairy judges, who acted as the arbiters of quality. Modern sensory evaluation replaced these single authorities with panels of people participating in specific test methods that took the form of planned experiments (Lawless *et al.*, 2010).

The sensory properties of food, as well as a large number of non-sensory factors, determine the decisions people make with respect to food (Jaeger, 2006). Sensory evaluation by industry typically relies on statistical analyses of trained human assessors intensity ratings on extensive sets of sensory descriptors, characterising a product in terms of several numbers representing different parameters, all generated during one session. It is common practice to seek correlations between these sensory profiling scores and the values given by bench instruments for physicochemical properties of the materials (Booth *et al.*, 2003).

There is still an open discussion whether or not sensory results should be correlated to instrumental data obtained under conditions that simulate oral breakdown (Hutchins *et al.*, 1988; Luyten *et al.*, 2004). However, the exact loading conditions in the mouth cannot be easily specified. The response of a solid food to loading is a function of its geometry and mechanical properties. Furthermore, the masticatory system is responsive to changes in food texture, even in the same chew, the force applied drops abruptly once the food particle starts to fracture (Heath, 2002; Lucas *et al.*, 2004). The breakdown of a food during mastication is highly variable, depending on the food characteristics and also on the consumer eating it (Bourne, 2004; Lillford, 2001).

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So, the perception of texture is a dynamic process in which the food breakdown is an essential input: sensory appreciation is affected by the oral process, which in turn affects the breakdown of the structure of the structure of the food in the mouth (Engelen *et al.*, 2008; Hutchins *et al.*, 1988; Lillford, 2001; Nishinari, 2004; Wilkinson *et al.*, 2000). The grinding of food into a fine state is the main function of mastication, but this process also imparts pleasurable sensations that fill a basic human need (Bourne, 2004). Consequently, it is necessary to understand the physical ways in which food breaks down, the particle distributions occurring during fracture and how the breakdown pattern affects consumer preferences (Varela *et al.*, 2008). Sensory research involves the study of texture perception using sensory panels and required input from e.g. psychology and neurophysiology. Oral physiology should study food breakdown during consumption and the relation with oral processes. Physics and chemistry research should involve the study of rheological and fracture characteristics, microstructure, ingredient interaction and other relevant food characteristics in relation to texture perception (Van Vliet *et al.*, 2009). Texture is an essential part of the whole spectrum of sensory properties of a food. Since it is a sensory attribute only a human being can perceive and rated it (Szczesniak, 2002). As mentioned above texture is a multi-parameter sensory attribute. As a consequence, advances in understanding of texture perception will depend on a multidisciplinary approach (Wilkinson *et al.*, 2000).

On Table 1 are presented the three types of tests that are commonly used, each with a different goal and each using participants selected using different criteria.

Table 1 – Classification of test methods on sensory evaluation (Lawless *et al.*, 2010)

Class	Question of interest	Type of test	Panellist Characteristics
Discrimination	Are products perceptibly different in any way	“Analytical”	Screened for sensory acuity, oriented to test method, sometimes trained
Descriptive	How do products in specific sensory characteristics	“Analytical”	Screened for sensory acuity, oriented to test method, sometimes trained
Affective	How well are products liked or which products are preferred	“Hedonic”	Screened for products, untrained

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The simplest sensory tests merely attempt to answer whether any perceptible difference exists between two types of products. These are the discrimination tests or simple difference testing procedures. Analysis is usually based on the statistics of frequencies and proportions (counting right and wrong answers). From the test results, we infer differences based on the proportions of persons who are able to choose a test product correctly from among a set of similar or control products (Lawless *et al.*, 2010).

A classic example of this test was the triangle procedure, in this test; two products were from the same batch while third product was different. Judges would be asked to pick the odd sample from among the three. Ability to discriminate differences would be inferred from consistent correct choices above the level expected by chance. In the duo-trio procedure, a reference sample was given and then two test samples. One of the test samples matched the reference while the other was from a different product, batch or process. The participant would try to match the correct sample to the reference, with a chance probability of one-half. As in the triangle test, a proportion of correct choices above that expected by chance are considered evidence for a perceivable difference between products. A third popular difference test was the paired comparison, in which participants would be asked to choose which of two products was stronger or more intense in a given attribute. Partly due to the fact that the panelist's attention is directed to a specific attribute, this test is very sensitive to differences. Typically a discrimination test will be conducted with 25–40 participants who have been screened for their sensory acuity to common product differences and who are familiar with the test procedures. This generally provides an adequate sample size for documenting clear sensory differences. Often a replicate test is performed while the respondents are present in the sensory test facility. In part, the popularity of these tests is due to the simplicity of data analysis. Statistical tables derived from the binomial distribution give the minimum number of correct responses needed to conclude statistical significance as a function of the number of participants. Thus a sensory technician merely needs to count answers and refer to a table to give a simple statistical conclusion, and results can be easily and quickly reported (Lawless *et al.*, 2010).

The second major class of sensory test methods is those that quantify the perceived intensities of the sensory characteristics of a product, known as descriptive analyses. The first method to do this with a panel of trained judges was the Flavor

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Profile method developed in the late 1940s (Caul, 1957). This group of panelists was faced with developing a comprehensive and flexible tool for analysis of flavor to solve problems involving unpleasant off flavors in nutritional capsules and questions about the sensory impact of monosodium glutamate in various processed foods. They formulated a method involving extensive training of panelists that enabled them to characterize all of the flavor notes in a food and the intensities of these notes using a simple category scale and noting their order of appearance. This advance was noteworthy on several grounds. It supplanted the reliance on single expert judges (brewmasters, coffee tasters, and such) with a panel of individuals, under the realization that the consensus of a panel was likely to be more reliable and accurate than the judgment of a single individual. Second, it provided a means to characterize the individual attributes of flavor and provide a comprehensive analytical description of differences among a group of products under development (Lawless *et al.*, 2010).

Other approaches were developed for descriptive analysis problems. At Stanford Research Institute in the early 1970s, a group proposed a method for descriptive analysis that would remedy some of the apparent shortcomings of the Flavor Profile method and be even more broadly applicable to all sensory properties of a food, and not just taste and texture (Stone *et al.*, 1974). This method was termed Quantitative Descriptive Analysis or QDA (Stone *et al.*, 2004). QDA procedures borrowed heavily from the traditions of behavioral research and used experimental designs and statistical analyses such as analysis of variance. This insured independent judgments of panelists and statistical testing, in contrast to the group discussion and consensus procedures of the Flavor Profile method. Other variations on descriptive procedures were tried and achieved some popularity, such as the Spectrum Method that included a high degree of calibration of panelists for intensity scale points (Meilgaard *et al.*, 2006).

Descriptive analysis has proven to be the most comprehensive and informative sensory evaluation tool. It is applicable to the characterization of a wide variety of product changes and research questions in food product development. The information can be related to consumer acceptance information and to instrumental measures by means of statistical techniques such as regression and correlation. The panel for such an analysis would consist of perhaps 10–12 well-trained individuals, who were oriented to the meanings of the terms and given practice with examples. Since they have been

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trained to use attribute scales in a similar manner, error variance is lowered and statistical power and test sensitivity are maintained in spite of fewer observations (fewer data points per product). Similar examples of texture, flavor, fragrance, and tactile analyses can be found in Meilgaard *et al.* (2006).

The third major class of sensory tests is those that attempt to quantify the degree of liking or disliking of a product, called hedonic or affective methods. The most straightforward approach to this problem is to offer people a choice among alternative products and see if there is a clear preference from the majority of respondents. The problem with choice tests is that they are not very informative about the magnitude of liking or disliking from the respondents. The larger panel size of an affective test arises due to the high variability of individual preferences and, thus a need to compensate with increased numbers of people to insure statistical power and test sensitivity. It may also provide an opportunity to probe for diagnostic information concerning the reasons for liking or disliking a product (Lawless *et al.*, 2010).

Traditionally, textural assessments have been carried out by taste panels, which may or may not be formally trained in the appraisal of textural characteristics. Defining textural properties and their relative magnitude with respect to other similar products will increasingly become a critical criterion for food manufacturers seeking to design new products, maintain the quality of current ones or understand strengths and weaknesses relative to their competitors (Kealy, 2006).

Texture is a sensorial parameter that only man can be aware of, express and quantify (Barata de Carvalho, 2006). Szczesniak (2002) states that a generally accepted definition of texture is the following “texture is the sensory and functional manifestation of the structural, mechanical and surface properties of foods detected through the senses of vision, hearing, touch and kinesthetic.” Its objective characterization could not be solely analyzed based on instrumental approaches such as mechanical or microstructural, since certain characteristics related to eating, like moistness, roughness, size, shape and number of food particles are very important to the perception (Hutchins *et al.*, 1988; Nishinari, 2004; Szczesniak, 2002).

In the early 1960s, a group of panelists developed and refined a method to quantify food texture, much as the flavor profile had enabled the quantification of flavor properties (Brandt *et al.*, 1963, Szczesniak *et al.*, 1975). This technique, the Texture

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Profile method, used a fixed set of force-related and shape-related attributes to characterize the rheological and tactile properties of foods and how these changed over time with mastication. These characteristics have parallels in the physical evaluation of food breakdown or flow. Texture profile panelists were also trained to recognize specific intensity points along each scale, using standard products or formulated pseudo-foods for calibration (Lawless *et al.*, 2010).

The usual sequence of texture perception when consuming a food product is visual evaluation of texture followed by direct (with the fingers) and/or indirect with eating utensils such as knife, fork, or spoon) tactile evaluations followed by oral-tactile (with the lips, tongue, palate, saliva) evaluations. Concurrent with the oral-tactile evaluation (and sometimes also when cutting/ stabbing the food with a utensil) are also the aural (sound) evaluations of crunchy, crispy, crackly, etc.

Sensory analysis is widely used to evaluate the perceived characteristics of dry cellular food products like extrudates, chips, or puff cereal products and results often compared to physical measurement (Chaunier *et al.*, 2005). For instance, hard solid foods are foods like crackers, breakfast cereals, dry toast, expanded starch products, biscuits, crispy crusts of bread and fried snacks, hard candies, but also products as fresh carrots, celery, and some fruits. Important sensory attributes for these products are among others “hardness”/”firmness”, “brittleness”, “crispy”/”crunchy” behavior, “splinter formation”, and rate of dissolving (for hard candies) (Van Vliet *et al.*, 2009). And so, consumers are able to describe the differences between crisp and crunch by judging the sound: a crisp sound is short, a crunchy sound more long lasting (Fillion *et al.*, 2002). On table 2, it was exemplified the sensory tests applied to several crispy products in some studies.

Although sensory analysis gives a more complete description of texture of tested products, there has been a great interest in developing instrumental techniques to assess crispness. Instrumental techniques present some advantages, especially in industrial environments where quick and easy-to-use methods are in great demand and economically more profitable. Crispness is being described as a concept with kinesthetic and auditory components, therefore it is not surprising that the instrumental methods developed to evaluate it, have focused on the measurements of these properties singularly or in combination of both properties (Roudaut *et al.*, 2002).

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Table 2 – Methods for sensory evaluation of several crispy products

Product	Type of Test	Panellist characteristics	Measured parameters	Reference
Potato-Soy snacks	Hedonic	Panel 10 judges	Overall Acceptability	Nath <i>et al.</i> , 2007
Potato Crisps	Descriptive	11 Trained	Hardness, crunchiness, sogginess, rate of breakdown, density, etc	Rojo <i>et al.</i> , 2009
Crust bread	Discriminative	Panel	Crispy, dry, tough, brittle, hard, soft	Primo-Martín <i>et al.</i> , 2006
Chips, puff cereals, extrudates	Descriptive	Trained panel	Crunchy, crispy, cracky	Saeleaw <i>et al.</i> , 2011
Apples	Descriptive	Panel	Crispness, juiciness Hardness, crunchiness, mealliness, Overall Texture	Brookfield <i>et al.</i> , 2012; Zdunek <i>et al.</i> , 2010, 2011
Apples	Discriminative	Panel	Crispy, mealy	Ballabio <i>et al.</i> , 2012
Crispy biscuit	Discriminative	Untrained panel	Bite force, hard, crumbly, sticky, drying, gooey, crunchy, sound, etc	Pocztaruk <i>et al.</i> , 2011
Toasted rusk roll	Pairwise comparison	Trained panel	Amount of sound, snapping, crackly, crispy, brittle, hard, tough, etc,	Primo-Martín <i>et al.</i> , 2008
Potato chips	Descriptive	Trained panel	Hardness, crispness	Salvador <i>et al.</i> , 2009
Crackers	Hedonic	15 assessors	Appearance, oiliness, color, crispness, taste, overall acceptability	Wang <i>et al.</i> , 2012

Introduction

Table 2 – Methods for sensory evaluation of several crispy products

Product	Type of Test	Panellist characteristics	Measured parameters	Reference
Battered squid rings	Hedonic	50 consumers	Appearance, color, crispness, favour, Overall Acceptability	Sanz <i>et al.</i> , 2007
Biscuits	Descriptive	2 Trained	Irregularity, development, color of the sole, color of sugaring	Edoura-Gaena <i>et al.</i> , 2007
Chapatti	Hedonic	10 judgments	Appearance, bite, texture, taste, overall acceptability	Shalini <i>et al.</i> , 2007
RTE potato-soy snacks	Hedonic	10 judges	Overall acceptability	Nath <i>et al.</i> , 2008
Roasted almonds	Descriptive	100 consumers	Crispness, hardness, crunchiness, deformability, Overall Texture	Varela <i>et al.</i> , 2008
Chickpea flour-based snacks	Hedonic	10 Trained	Appearance, color, taste, crispness, overall acceptability	Debnath <i>et al.</i> , 2003
Coated turkey breast	Hedonic	24 Untrained 1	Crispness	Iliassafov <i>et al.</i> , 2007
Cornflakes	Descriptive	13 assessors	Sound, brittleness, airiness, crackly	Chaunier <i>et al.</i> , 2007
Corn-lentil extruded snacks	Descriptive	10 Trained	Hardness, crispness, crunchiness, appearance, cohesiveness	Lazou <i>et al.</i> , 2010
Snack food	Discriminative	11 Judges	Crispness	Srisawas <i>et al.</i> , 2003

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Moreover the different approaches to crispness/crunchiness evaluation, both sensory and instrumental, that have been described in several studies, it is important to have well-defined Spanish terms and their correct translation into English (Varela *et al.*, 2008). The Spanish translations presented by Drake were “crocante” or “crujiente” for crunchy and “quebradizo” or “crujiente” (again) for crispy. Another view of crisp and crunchy foods looks at the time-sequence of breakage, the deformation and rupture of the food upon application of force (Szczesniak, 1990).

Crisp foods break in a single stage whereas crunchy foods break in several successive stages. Thus, a crisp food will always be perceived as crisp regardless of the way the breaking force is applied, but a crunchy food may be perceived as crunchy or crisp depending on the applied force (Lawless *et al.*, 2010).

Sensory descriptive test are among the most sophisticated tools used by sensory scientist. Descriptive analysis training for crispness and crackliness evaluation focuses on parameters such as structure of the intact food, sound emitted at fracture, the force needed to crush the food, the collapse of the food at fracture and the appearance of sample observed and perceived following fracture (Roudaut *et al.*, 2002). From the sensory results, Primo-Martín *et al.* (2010) found that panelists perceive a higher sound intensity from crispier foods.

Sensory evaluation provides a direct measure of food crispness but it is not a convenient routine test method. An alternative for food crispness evaluation is to analyze the sounds produced during mechanical load with an instrument. At present instrumental acoustic methods are becoming more and more popular for the investigation of food product properties. Few researches have combined mechanical-sound measurements with sensory evaluation. The use of instrumental analysis is more convenient than that of sensory evaluation. The determination of texture by instrumental tests is easy to perform, simple to reproduce and less time consuming. However, studying sound-mechanical instrumental data correlated with sensory score may help to fully understand crispness perception by individuals (Saeleaw *et al.*, 2011).

II – OBJETIVOS

II.1. –OBJETIVO GENERAL

En un gran número de alimentos, tales como los snacks crujientes, los rebozados, o en algunas verduras y frutas, las texturas crujiente y crocante son de una gran importancia, estando directamente relacionadas con la aceptabilidad del producto por parte de los consumidores. Existen multitud de factores que se relacionan con la mayor o menor intensidad de esta textura, desde la propia composición del producto (contenido en humedad, proteínas, carbohidratos y grasa, tipo de proteína, carbohidrato o grasa), pasando su estructura y microestructura (organización de los componentes, formación de burbujas, estructura de las paredes de las burbujas...) y sin olvidar los aspectos relacionados con su proceso de elaboración y almacenamiento grado de deshidratación, temperatura de fritura, humedad durante el almacenamiento, recalentamiento...). Muchos de estos productos con una textura crujiente son ámbito de la producción industrial, pero existen también innumerables ejemplos de productos que se elaboran culinariamente. Es más, en los últimos años se ha disparado el uso de diferentes tecnologías, tradicionales y avanzadas, para el desarrollo de snacks por parte de cocineros, con el objetivo de poder desarrollar productos con una identidad propia y original. Por ejemplo, se ha propuesto el uso de técnicas alternativas a la fritura, como el calentamiento en microondas, para la elaboración de snacks fritos tradicionales, como las cortezas de cerdo. Es más, mediante la desestructuración de la corteza, se ha sugerido el uso de esa masa desestructurada y tratada en el microondas como una posible cobertura crujiente de diferentes preparaciones. El famoso cocinero Heston Blumenthal desarrolló un método para mejorar la textura crujiente los productos rebozados y fritos mediante la sustitución de parte del agua de la masas por alcohol etílico. Y alguno, han desarrollado un crujiente cuya base es la elaboración de una espuma con diferentes hidrocolóides, que es posteriormente deshidratada mediante desecación. Paralelamente, diferentes cocineros, como Ferrán Adriá, han venido utilizando la liofilización como método para el desarrollo de espumas con una textura ligeramente crujiente y etérea.

Objectives

Resulta por lo tanto de un gran interés conocer los factores que determinan la textura de este tipo de productos, para así poder adecuar sus condiciones de elaboración y almacenamiento, dado que hasta el momento, su desarrollo ha sido básicamente empírico, y se desconocen muchas de sus características estructurales y de composición

La evaluación de sus características puede realizarse mediante análisis sensorial. Este tipo de técnicas implica un adecuado entrenamiento de un panel de catadores, o el uso de paneles de consumidores muy numerosos. Así pues, a la hora de evaluar dichas características de snacks y otros alimentos crujientes, resulta interesante conocer en qué medida otras técnicas instrumentales, más sencillas y objetivas que las del análisis sensorial, proveen de una información útil y valiosa a la hora de evaluar la textura crujiente de distintos productos crujientes.

Así pues los dos grandes objetivos globales de esta tesis doctoral fueron conocer cómo distintos factores se relacionan con la textura de diferentes productos cuya característica más sobresaliente era su textura crujiente, y determinar en qué grado diferentes técnicas de análisis proveen de información valiosa sobre estas características texturales.

II.2 – OBJETIVOS ESPECÍFICOS

El objetivo general de este trabajo se concreto en una serie de objetivos específicos:

1. Conocer las diferencias en los cambios que acaecen durante el proceso de elaboración y en la composición y características de cortezas fritas de cerdo, y de cortezas desestructuradas de cerdo.
2. Establecer en qué medida el grado de deshidratación previo al calentamiento en microondas influye sobre diferentes características físico químicas, estructurales y sensoriales de cortezas de cerdo.
3. Determinar la efectividad de diferentes técnicas analíticas para evaluar las características y estructura de cortezas de cerdo terminadas en microondas
4. Comprobar cómo la sustitución del agua por etanol y la presencia de dióxido de carbono en masas para rebozadas influyen sobre las características físico-químicas, estructurales y sensoriales de la cortezas de calamares rebozados.

Objectives

5. Desarrollar espumas deshidratadas crujientes de yogur mediante el uso de diferentes hidrocolóides y deshidratación mediante desecado y liofilización.
6. Conocer las características físico-químicas, estructurales y sensoriales de dichas espumas y establecer relaciones entre dichas características.

**CHAPTER I - BROMATOLOGICAL ANALYSIS OF
DEHYDRATED SNACKS
("ANÁLISIS BROMATOLÓGICA DE SNACKS DESHYDRATADOS")**

ABSTRACT

There are several studies about meat snacks in order to increase the alternative value of certain meat tissues. Pork skin is a by-product that results from carcass cut and de-boning (6.7% of carcass weight), with a very particular texture, and which use in food industry have been increasing, from gelatine production to traditional fried pork rinds snacks elaboration. Frying is a largely used method to produce these type of snacks since it permits to develop an unique texture and flavour, however due to its fat content, and also to a large demand from consumer for low calories "snacks", with less salt, or with health benefits, it has become necessary the use of some techniques to produce "snacks" without frying. So, dehydration by hot air followed by microwaving may be an option to obtain a similar kind of product. In fact, the combination of hot air dehydration technology followed by microwave application may be also an interesting approach for improving product quality and reduce production costs. In this study, pork skin with 50% of initial moisture was used and it was produced following two different procedures (whole and minced) and subsequently dried at 50°C for 24h. Bromatological analyses were made, namely moisture, ash, protein, fat. Water losses during drying were recorded in both products (whole and minced). Significant correlations between chemical parameters were observed, and there were significant differences in moisture content, protein and fat content either in whole pork rind samples or in minced pork rind samples. In bromatological terms, the whole dehydrated pork rind had higher protein content and lower fat content than minced dried pork rind, and thus it had a higher caloric value.

Keywords: Pork Rind, Dehydration, Mincing, Moisture, Bromatological Analysis

1. INTRODUCTION

With scientific, technological and empirical knowledge, Food Engineering searches new benefits for society such as a longer shelf life, total takings of natural resources and elaboration of new products that attend to consumer's needs and satisfaction. The demand for these convenient foods has led to the development of products based on by-products, which subsequently has resulted in an effective profitability for these by-products (Ferreira, *et al.* 2009). Studies on restructured beef snacks sticks and jerky has focused on increasing the share of the food market held by meat snacks (5%). Beef jerky is a high protein; light-weight and shelf-stable meat snack that has traditionally been made from thinly sliced whole muscles which are brined and dried. Current developments have involved developing snack sticks or restructured meats (Unklesbay *et al.*, 1999). Pork skin is a by-product resulting from pork carcass to bone and cut, with a very special and hard texture, that represents around 6.7% of carcass weight. It has been traditionally used in Spain and South American countries to produce fried puffed snacks, called "couratos" or "cortezas". The skin from the back is the best for obtaining this product, since it presents a strong protein composition, fat and collagen, and it's very uniform in terms of moisture, protein and fat, which permits to the final product to reach unique sensorial characteristics (Ferreira, *et al.* 2009). Collagen is a natural protein with tertiary structure, which constitutes a fundamental part of animal tissues, providing springiness and strength to bones, tendons, skin and so on (Zira, 2008). It is composed by many amino acids, where it's possible to identify the three main: glicin, prolin and hidroxyprolin. Pork skin has major applications in different subjects, but the most usual is on medicine, more precise on surgery, being most useful as support in skin implants in the treatment of serious burns on patients (Ramírez, *et al.*, 2005). Along time pork skin has been used in bags manufacture, belts and wallets and leather articles. Nowadays it's largely used on food industry, namely on gelatine (E441) extraction (Isoldeh, 2009), and in pork rind snacks as aperitifs.

Air-drying, in particular, is an ancient process used to preserve foods in which the solid to be dried is exposed to a continuously flowing hot stream of air where moisture evaporates (Ratti, 2001) and reduces water activity and increases products shelf-life (Zhang *et al.*, 2006). This process has a complex combination of transport phenomena such as hot air application and moisture removal from substrate. This

method is effective in biological products conservation, since it doesn't require a severe thermal process and allows the conservation at room temperature (Schuck *et al.*, 2008). The combined use of convective drying followed by microwaving not only greatly enhances the drying rate, but may also improve final product quality, allowing the product to puff similarly to when it is fried but reducing the caloric content (Torrington, *et al.*, 2001). Microwave energy can successfully be applied in several unit processes in the food industry because of the resulting volumetric heating of the material. Since there is no need for a driving temperature gradient, the practically obtained power densities are generally much higher compared to conventional heat transfer. Successful applications are pasteurising of convenience food and thawing. The high thermal efficiency of microwaves at low moisture contents gives a possibility for decreasing energy consumption in comparison with convective drying process (Nijhuis *et al.*, 1998).

Research on microwave expanded snack foods has increased in recent years because of their prospective convenience for consumers and also the potentially diverse range of shapes and textures that can be achieved at home using pre-made food formulations. Previous studies on microwave expanded snack foods have focused mainly on popcorn (Lin *et al.*, 1988; Pordesimo *et al.*, 1990; Mohamed *et al.*, 1993; Singh *et al.*, 1999) cereal-based pellets such as rice or corn pellets (Chen *et al.*, 2000; Gimeno, *et al.*, 2004).

For these cereal-based snack foods, various compositional variables have been shown to influence the expansion during microwave heating (Gimeno *et al.*, 2004). Moisture content is an important factor as it is thought to change to superheated steam providing the necessary driving pressure that induces expansion during microwave heating (Ernault *et al.*, 2002; Sjöqvist *et al.*, 2005). On microwave heating, the food matrix melts, the temperature of the water in the sample reaches boiling point and vapour bubbles are created. The bubbles then grow within the food matrix causing it to expand as foam (Arimi *et al.*, 2008). Water vapour acts as a plasticizer of the matrix and in addition creates the pressure that induces expansion.

Three major processes were observed to occur during microwave heating. These were firstly a temperature increase, secondly expansion and thirdly weight loss. On continued heating evaporation of water vapour takes place and once sufficient moisture

has been driven off (moisture content < 15%) the matrix appears to set, since it had reached its glassy transition state, and maintains the expanded structure on cessation of heating. If insufficient evaporation occurs and too much moisture (plasticizer) remains in the sample, as is the case for short heating times, the matrix can collapse on cooling. Other components such as fat and protein have been shown to influence the expansion process. Solid fat favours expansion but liquid fat has the opposite effect (Ernoult *et al.*, 2002). Proteins affect expansion through their effect on water distribution in the matrix (Gimeno *et al.*, 2004).

The presence of even a small amount of fat markedly reduces the degree of expansion possibly due to the negative effect of liquid fat on foam stability (Arimi *et al.*, 2008).

Frying is a thermal process largely applied to food production, namely on meat products since it improves its texture, flavour and appearance (Girard, *et al.*, 1991).

Deep fried products form an important group of food products consumed worldwide, and for decades consumers appreciate fried products due to its unique flavour-texture combination, such as in French fries, doughnuts, extruded snacks, fish nuggets and chicken nuggets (Van Vliet, *et al.*, 2007).

However, the increased awareness of consumers to the relationship between food, nutrition, and health has emphasized the need to limit oil consumption, calories originating from fat and cholesterol among others. Today consumers are more interested in healthy products that taste good (Da Silva *et al.*, 2008).

With microwave expansion innovation the principal aim of this study is an attempt to valorise a by-product, which first of all is an important protein, fat, amino acids, minerals and vitamins source (Zhang *et al.*, 2010; Biesalski, 2005), and at the same time, guarantee that during their procedure those snacks will uptake less oil and then will be healthier.

In order to accomplish that aim it was first of all prepared two kinds of pork rind, one in whole strips and the other minced and moulded, then it was done several cycles of 24 hours, in an attempt to optimize pork rind dehydration. In parallel, it was also made a bromatological analysis to both dehydrated whole pork rind and minced pork rind and at last it was made several microwave experiments with the purpose of achieving an expanded snack from the dehydrated whole and minced pork rind.

2. MATERIAL AND METHODS

Two kinds of pork skin (rind) snacks have been studied: “Whole Snacks” and “Minced Snacks”, which steps in laboratory processing, will be described. Raw material used on both final products was the same and it was fresh local pork skin, which was obtained from the local slaughterhouse and stored at 4°C and then manually trimmed into rectangular chunks. However, the two different snacks had different steps during their processing as shown in the following figures. First, pork skins were boiled under pressure in a pressure cooker for different times (40 and 60 minutes) (Figure 3). Afterwards, they were trimmed for visible fat.



Figure 3 - Boiled pork skin under pressure

Before the dehydration step, half the rinds were minced at 60°C in a Thermomix and moulded in silicon moulds (Figure 4) for “Minced Snacks” dehydration. The other half were cut into strips (Figure 5) for “Whole Snacks” dehydration.



Figure 4 – Minced pork rind in silicon moulds



Figure 5 – Pork skin cut into strips

Bromatological Analysis of Dehydrated Snacks

Dehydration phase occurs in a convective hot air dryer STI (figure 6) under controlled temperature (50°C), relative humidity (10%) and air velocity, by use of an anemometer to take register of hot air flow speed.

Dehydration temperature was defined based on some studies related to meat products dehydration (Singh, *et al.* 2000).



Figure 6 – Pork rind dehydration in the convective hot air dryer

After dried stage, the snacks are ready to expansion on a KUNFT microwave in an adequate recipient at 700W power for 1 minute. In figures 7 and 8 was represented both minced and whole pork rind processes.

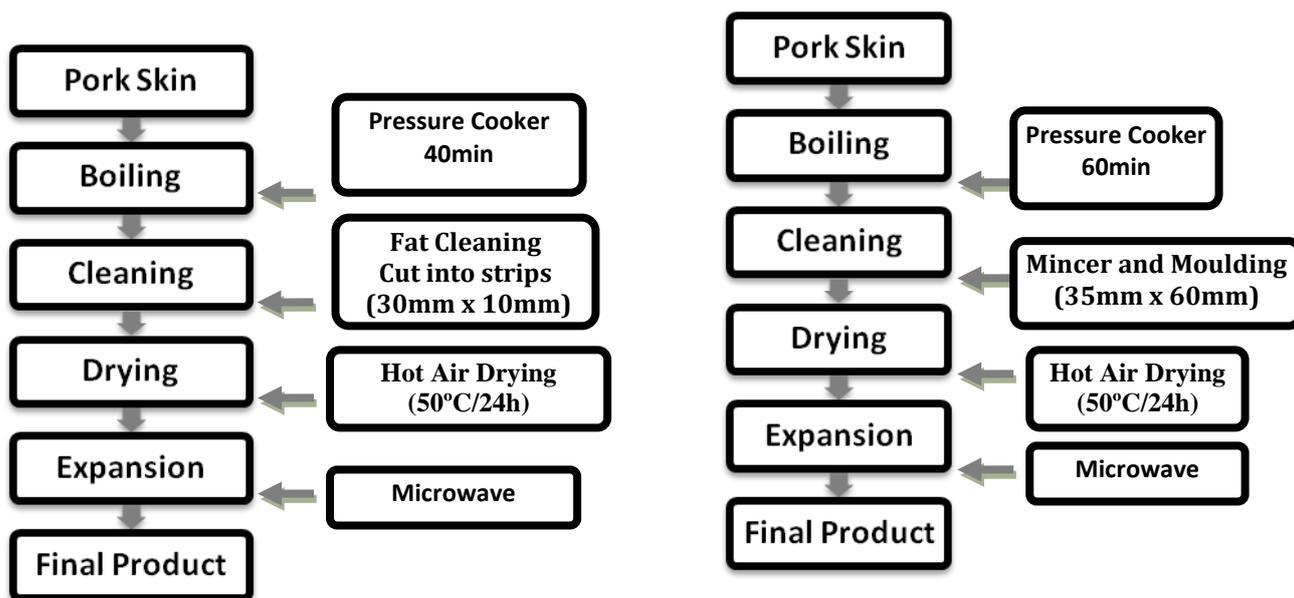


Figure 7. “Whole Snacks” processing

Figure 8. “Minced Snacks” processing

Bromatological Analysis of Dehydrated Snacks

The single difference between both processes was boiling time, which was longer in minced pork rind (60 min) than in whole pork rind (40 min), besides the biometry/structure of the pork rind before and after dehydration phase (figure 9 and 10).

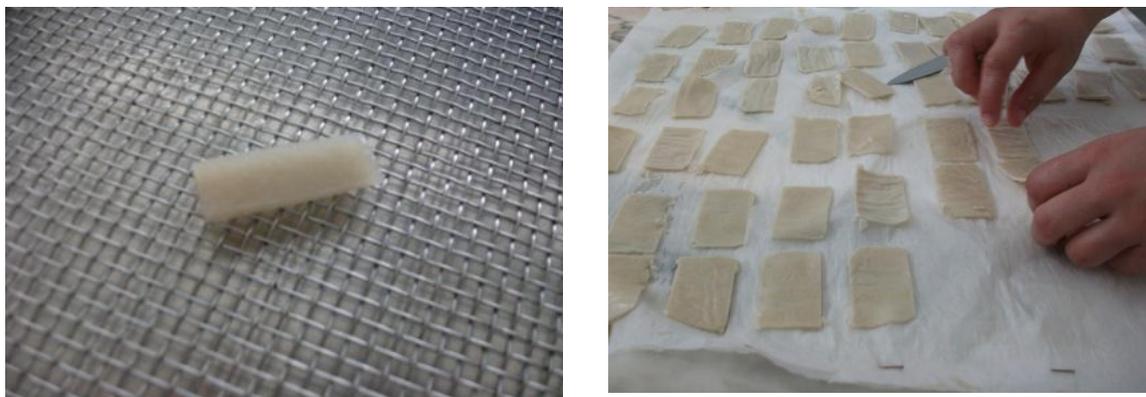


Figure 9 – Pork rind strip before dehydration Figure 10 – Dehydrated minced pork rind

Both types of pork rind dehydrated snacks were subjected to physical and chemical analyses, more precise to moisture content analysis, which was determined by NP 1614/1979, to ash content analysis, which was followed by the method NP 1610/1979, to protein determination, which method was NP 1612/1979, and finally to fat content analysis, which was determined by NP 1613/1979. The microwave expansion was done in several attempts to achieve an expanded final product from the dehydrated whole and minced pork rind.

For sample preparation to physical and chemical analyses, it was necessary to use a mill to assure samples grinding homogeneity, however due to samples hardness/toughness after dehydration, mainly in whole pork rind strips, it was necessary to vacuum seal samples and freeze at -20°C before milling, in order to allow grinding phase and consequently improve sampling uniformity. All analyses were made with three replicas.

The balance was used to estimate the crude carbohydrate content. The physical and chemical parameters were subjected to analysis of variance (ANOVA) to determine statistically significant ($p < 0.05$) differences. Scheffé's test was used to distinguish differences between means. Physic-chemical variables were subjected to Pearson's correlations.

3. RESULTS AND DISCUSSION

Whole pork rind moisture content (%), protein (%) and fat content (%) are shown on the following table, for raw pork skin samples (RPS), boiled pork skin samples (BPS) and for dried pork skin rind strips during drying phase at drying times, from 2h drying (2hD) to 24h drying (24hD), as well as the significant differences between all samples before and during drying phase.

Table 3 - Moisture content (%), protein (%) and fat content (%) on raw pork rind (RPS), on boiled pork rind (BPR) and on dried whole pork rind with 2h drying (2hD), (...) and 24h drying time (24hD)

Samples	Moisture (%)	Protein (%)	Fat content (%)
Raw (RPS)	50.71 ^b (0.82)	35.10 ^h (0.69)	12.70 ^b (0.82)
Boiled (BPS)	55.76 ^a (0.41)	41.30 ^g (0.47)	2.24 ⁱ (0.33)
2hD	44.67 ^c (0.55)	48.70 ^f (0.64)	6.09 ^h (0.65)
4hD	33.25 ^d (0.73)	57.87 ^e (0.73)	7.70 ^h (0.58)
6hD	22.83 ^e (0.84)	66.95 ^d (0.63)	10.03 ^{d,e,f,g} (0.47)
8hD	15.11 ^g (0.78)	72.77 ^b (0.64)	11.80 ^{b,e} (0.72)
10hD	14.45 ^g (0.84)	73.15 ^b (0.69)	12.22 ^b (0.55)
12hD	14.50 ^g (0.69)	69.33 ^c (0.55)	15.25 ^a (0.60)
14hD	13.22 ^{g,h} (0.65)	73.90 ^b (0.65)	12.27 ^b (0.63)
16hD	11.10 ^{i,f} (0.80)	76.59 ^a (0.79)	11.75 ^{b,g} (0.73)
18hD	11.45 ^{h,i,f} (0.73)	75.89 ^a (0.78)	11.89 ^{b,d} (0.77)
20hD	13.03 ^{g,i} (0.83)	76.25 ^a (0.79)	10.16 ^{c,d,e,f,g} (0.55)
22hD	10.95 ^f (0.57)	76.90 ^a (0.76)	11.78 ^{b,f} (0.73)
24hD	10.33 ^f (0.65)	77.27 ^a (0.80)	12.02 ^{b,c} (0.62)

Related to moisture content, it was expected that raw and boiled samples presented significant differences between each other and also with all dehydrated samples, and, in fact, moisture content of raw samples was 50.71%, which was significant lower than moisture content of boiled samples with 55.76%. In other study, moisture content on cooked pork rind was 75.88% (Visessanguan *et al.*, 2005).

Bromatological Analysis of Dehydrated Snacks

Dehydrated samples at the first 8h of drying process had significant differences and the values were 44.67% with 2h dehydration time and 22.83% with 6h of dehydration time. So, it was with 8h dehydration time and until 14h, that the samples presented a constant and with insignificant differences, from 15.11% at 8h and 13.22% at 14h. Then, the last drying period (since 16h until 24h) presented significant differences with this last one (from 8h until 14h), since moisture content was 11.10% with 16h (16hD) and at it was 10.33% with 24h drying time (24hD).

Notice the exception of sample at 20h drying time with a moisture content of 13.03%, and so with no significant differences with samples from 8h (8hD) until 18 h (18hD), and thus presented significant differences with samples 22h (22hD) and 24h (24hD). This fact may be similar to the principle applied in moisture content analysis, which was determine by a official drying method at $\pm 103^{\circ}\text{C}$ until constant mass (NP 1614/1979), and thus this criteria determine the end of the drying process when the last to weights of the sample in analysis were constant or even if it increased a few and then decreased again and maintained the previous constant mass. In fact, this sample with 20h drying time was in the middle of four samples (16hD, 18hD, 22hD and 24hD) that hadn't significant differences between each other.

Protein content revealed an increase with drying time, and thus the higher value was on sample 24hD with 77.27%, but this sample hadn't significant differences with dried samples with 16h (16hD) until 22h (22hD), which protein content was, respectively, 76.59% and 76.90%. However, all these samples presented significant differences with the other dried, boiled and raw samples.

Samples dried for 8h (8hD), 10h (10hD) and 14h (14hD) had no significant differences with each other, and their values were, respectively, 72.77%, 73.15% and 73.90%. All the other dried samples had significant differences with each other and with those mentioned dried samples, but in general, as drying process occurred the protein content increased, as shown by sample 2hD with 48.70% and sample 24hD with 77.27%.

Protein content on raw sample was 35.10%, which was much lower than the value of 70% referred in a study (Bechtel, 2001), and in boiled sample was 41.30%, which is considerably high when compared with 22.79% (Visessanguan *et al.*, 2005) or with 26-27% (Osburn *et al.*, 1997) obtained in other studies with cooked pork rind.

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Fat content was significant lower on boiled sample (BPS) with 2.24% and on dried samples for 2h (2hD) and 4h (4hD) with, respectively, 6.09% and 7.70%. In other study, cooked pork rind had a similar fat content with 2.06% (Visessanguan *et al.*,2005).

Raw pork skin (RPS) presented a fat content of 12.70%, which was much lower than the values referred in other studies with a fat content of 27-28% (Osburn *et al.*, 1997) or 30% (Bechtel, 2001). So, for exception of dried samples at 6h (6hD) and 20h (20hD), which presented the lowest fat content values with 10.03% and 10.16%, respectively, and dried sample at 12h (12hD), that had the highest significant value of fat content with 15.25%, all the other dried samples had no significant differences with each other and with the raw pork skin, and their values were more or less from 11.75% to 1270%.

In resume, it was possible to notice that along the boiling phase, both moisture content and protein increases, and fat content decreases. And all along the process of drying, moisture content decreases approximately 1/5 of initial value, protein content increases at around the double of initial value, and fat content increases and stabilized with values similar to original value.

In reality, the differences possibly were influenced by conditions and methods used in rind preparation, in particular in some phases such as, fat trimming and scalding condition. Indeed, in some studies it was referred that the higher temperature and prolonged scalding time may had possibly leached out protein and lipids (Visessanguan *et al.*,2005), however in this study that possibility was only viable to fat content evolution after scalding phase, since protein content had not decreased in that phase, it seems that, even occurring, during heating, the collagens softening and thus its partial conversion in gelatine, which might be responsible for its leached out, in this study that phenomena was not sufficient to evidence a decrease on protein content when pork rind was cooked.

Dehydrated minced pork rind moisture content (%), protein (%) and fat content (%) are shown on the following table for different drying times, from 2h drying (2hD) to 24h drying (24hD), as well as for raw pork skin samples (RPS) and boiled pork skin samples (BPS), and also the significant differences between all samples before and during drying phase.

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Table 4 - Moisture content (%), protein (%) and fat content (%) on raw pork rind (RPS), on boiled pork rind (BPR) and on dried minced pork rind with 2h drying (2hD), (...) and 24h drying time (24hD)

Samples	Moisture (%)	Protein (%)	Fat content (%)
Raw (RPS)	50.71 ^b (0.90)	35.10 ^j (1.00)	12.70 ⁱ (0.85)
Boiled (BPS)	57.92 ^a (0.85)	24.58 ^l (0.79)	16.99 ^h (0.39)
2hD	32.98 ^c (0.95)	45.25 ⁱ (0.85)	20.79 ^g (0.48)
4hD	28.47 ^d (0.88)	48.35 ^h (0.80)	23.08 ^f (0.39)
6hD	16.67 ^e (0.87)	54.04 ^f (0.78)	29.02 ^e (0.48)
8hD	17.11 ^e (0.78)	47.17 ^{h,i} (0.93)	35.01 ^c (0.67)
10hD	8.19 ^f (0.75)	51.02 ^g (0.48)	40.47 ^b (0.67)
12hD	5.30 ^{g,i} (0.58)	51.80 ^g (0.87)	42.81 ^a (1.00)
14hD	6.13 ^{g,h} (1.00)	58.62 ^{b,c} (1.00)	34.63 ^c (0.89)
16hD	6.32 ^{f,g} (0.59)	57.08 ^{c,d} (0.60)	36.01 ^c (0.78)
18hD	3.50 ^{i,j} (0.77)	56.84 ^{c,e} (1.00)	38.90 ^b (0.93)
20hD	4.19 ^{h,i,j} (0.80)	59.77 ^b (0.81)	35.59 ^c (0.80)
22hD	4.49 ^{g,j} (0.95)	63.48 ^a (0.89)	31.59 ^d (0.98)
24hD	3.88 ^{i,j} (0.89)	56.02 ^{d,e} (0.75)	39.40 ^b (0.72)

Moisture content had presented significant differences between raw pork skin (RPS) with 50.71%, boiled pork skin (BPS) with 57.92% and dried samples until 10h (10hD) drying time. Then, all dried samples since 12h (12hD) until 24h drying sample (24hD) had no significant differences, and moisture content had values between 5.30% (12hD) and 3.88% (24hD), with a significant increase at 16h drying with 6.32%.

The fact that was relevant on moisture content, after observing tables 1 and 2, was the significant decrease between 8h (8hD) and 10h (10hD) on minced pork rind, whereas on pork rind there was no significant decrease in the same drying period. Above all, it was remarkable the great decrease on moisture content on minced pork rind during the 24h drying period, when compared with the whole pork rind, effectively the mincing process had also removed the bounded water, that was bound to the protein fraction, which was also affected by this mincing phase as will be following mentioned.

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Related to protein content, as it was expected this parameter increased during drying process from 45.25% with 2h drying time (2hD) to 56.02% with 24h drying time (24hD), but the highest values were on samples with 22h (22hD), 20h (20hD) and 14h (14hD) with 63.48%, 59.77% and 58.62%, respectively. On cooked pork skin, protein content decreased from 35.10% on raw pork skin to 24.58% after boiling phase, and thus it seems that occurred the phenomena, previously described, that during heating, collagens are softened and partially converted into gelatine, and therefore easily leached out (Visessanguan *et al.*, 2005). Then during drying phase, protein content never reached values similar to those observed on whole pork rind, and that fact might be due to a decrease on protein extraction during the method to quantify protein or to a protein degradation occurred during drying process.

On minced pork rind, the lowest fat content value was observed on raw pork skin, with 12.70%, and even on boiled minced pork rind, the fat content with 16.99% was significant higher than on raw pork skin, probably because this boiled sample was minced immediately after boiling phase and then was analyzed, which had not occurred on whole boiled pork rind samples (table 3), and so lipids leaching out seems to hadn't took place in this case.

The significant higher fat content value was at 12h drying (12hD) with 42.81%, followed by dried samples at 10h (10hD), 18h (18hD) and 24h (24hD) with significant lower fat content values around 39% - 40%, and then by the dried samples at 8h (8hD), 14h (14hD), 16h (16hD) and 20h (20hD), all without significant differences and with approximately 35%.

And the other dried samples, all with significant lower values, but even so, always higher than fat content values on whole pork rind, which fact lead to an hypothesis that, since extraction method of analysis was precisely the same (both with acid hydrolysis preceding extraction phase), probably mincing process contributed to an increase on fat extraction.

Whole and minced pork rind ash (%) are shown on the following table, for raw pork skin samples (RPS), boiled pork skin samples (BPS) and during drying phase for different drying times, from 2h drying (2hD) to 24h drying (24hD).

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Table 5 – Ash (%) on whole and minced raw pork rind (RPS), boiled pork rind (BPR) and on dried pork rind with 2h drying (2hD), (...) and 24h drying time (24hD)

Samples	Whole pork rind Ash (%)	Minced pork rind Ash (%)
Raw (RPS)	0.54 ^b (0.19)	0.54 ^a (0.20)
Boiled (BPS)	0.45 ^b (0.18)	0.51 ^a (0.07)
2hD	0.52 ^b (0.17)	0.08 ^e (0.05)
4hD	0.50 ^b (0.13)	0.11 ^c (0.10)
6hD	0.19 ^d (0.07)	0.25 ^d (0.05)
8hD	0.18 ^d (0.07)	0.19 ^d (0.06)
10hD	0.14 ^d (0.04)	0.15 ^d (0.04)
12hD	0.71 ^a (0.08)	0.09 ^e (0.01)
14hD	0.33 ^c (0.08)	0.60 ^a (0.05)
16hD	0.41 ^c (0.08)	0.54 ^a (0.15)
18hD	0.39 ^c (0.05)	0.39 ^b (0.10)
20hD	0.56 ^b (0.03)	0.46 ^b (0.04)
22hD	0.26 ^c (0.04)	0.42 ^b (0.09)
24hD	0.28 ^c (0.01)	0.45 ^b (0.14)

Either on whole samples or minced samples there were many significant differences on ash content, both raw samples had the same ash content (0.54%), and boiled minced pork rind had a higher ash content (0.51%) than whole pork rind (0.45%), but even so, both boiled samples had a much higher ash content than ash values presented on cooked pork rind in a study with 0.01% (Visessanguan *et al.*, 2005).

Related to correlations, it seems that in whole pork rind, moisture content presented significant correlations with protein and fat content, but with protein content it was a significant negative correlation, since the decrease on moisture content was correlated to an increase on protein content, but on the other hand, it had a significant positive correlation with fat content, since its content also decrease along moisture content decrease. On minced pork rind, moisture content had a significant negative correlation with both protein and fat content, since moisture content decrease implied an increase in both protein and fat content.

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Whole dehydrated pork rind had shown a faster decrease (figure 11) in moisture content (WSM), mainly between boiled sample (BPS) and dried sample for 8 hours (8hD). However, after this period the losses were lower and practically stabilized after 12 hours of drying process at 50°C, when those dried samples (12hD) had 14.50% moisture content. The results for moisture content during the 24 hours of drying period in parallel with studies of other meat products with higher moisture content (74% on beef jerkey) which were dehydrated at 55°C/7h until reaching 24% of moisture content (Konieczny, *et al.* 2006), permit inferring that probably it would be sufficient a drying process of 12 hours, or at most 14 hours, since after this drying time moisture content was relatively constant with approximately between 11% to 10% of moisture content with 16h drying and 24h drying time, respectively.

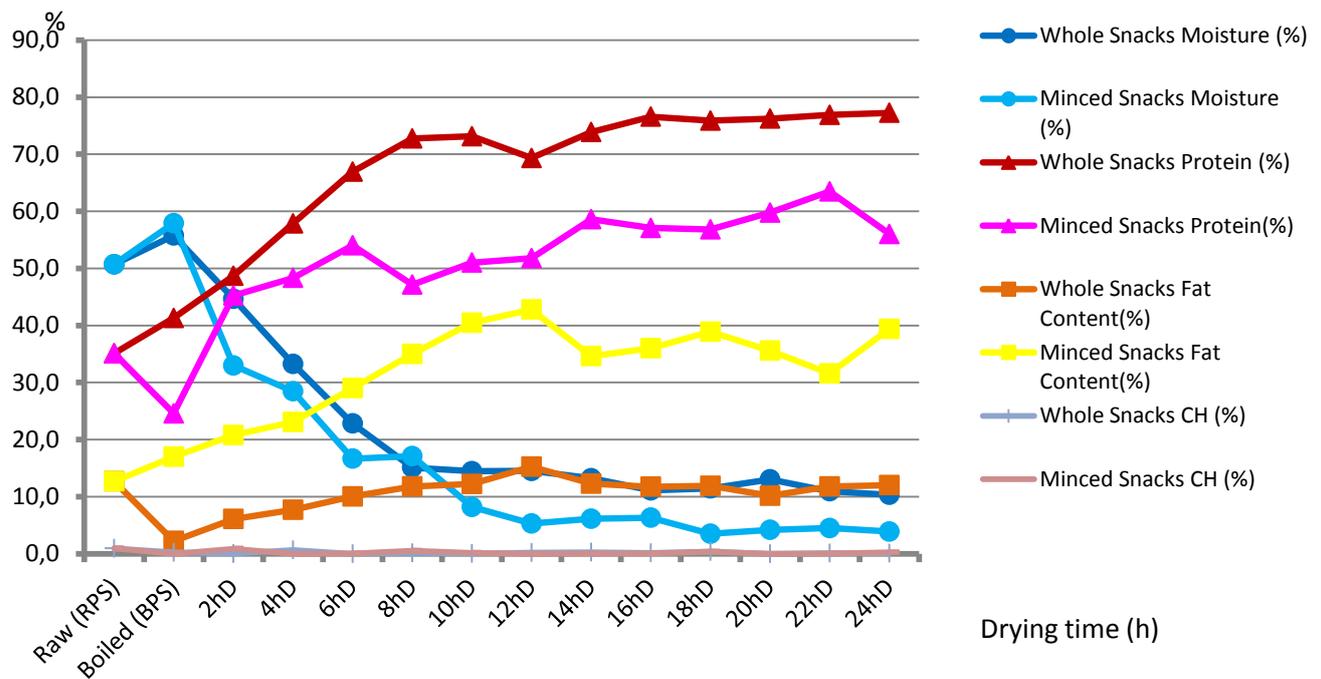


Figure 11 - Whole and Minced Snacks Bromatological Analysis

RPS – raw pork skin; BPS – boiled pork skin; 2hD – boiled pork skin dried for 2h at 50°C (...)
 24hD – pork skin dried for 24h at 50°C; WSM – whole snacks moisture; MSM – minced snacks moisture; WSP – whole snacks protein; MSP – minced snacks protein; WSF – whole snacks fat content; MSF – minced snacks fat content; WSCH – whole snacks carbohydrates; MSCH – minced snacks carbohydrates

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The extent of the moisture content decrease was higher on minced dried pork rind rather than in whole pork rind. Indeed, as it happens with whole pork rind, after the boiling phase, in which the samples gain moisture, there was a moisture decrease throughout drying time and finally, they reached a period, approximately after 12-14 hours drying, on which their moisture content maintains a relatively constant value, to be more exact, 3.5% with 18h drying and 3.9% after 24h drying time. So those facts impelled to establish that the most appropriate drying time for minced pork rind would be around 12 -14 hours.

In other studies with meat products with high moisture contents, such as spent hen meat (67%) (Lee, *et al.* 2002) and goat meat (73%) (Cosenza, *et al.* 2002), it was possible to produce either popped snacks made of spent hen meat mixed with cereal grains with 17% of final moisture content (Lee, *et al.* 2002) or goat snack sticks with final moisture 51%, however this last product is not considered a semi-dry product since its moisture content is slightly higher than 40-50% (Cosenza, *et al.* 2002).

Concerning to protein content, this study had shown that during drying periods there were in global an increase in protein content either in whole pork rind, or in minced pork rind, however the increase was slower and lower in minced pork rind than it was in whole pork rind, which fact might be related to a probable protein degradation during mincing process, as it was previously mentioned. In fact, cooked pork rind is an important source of protein, which form networks and structures and interact with other components. Thus, the gel formation of myofibrillar proteins is responsible for retaining and stabilizing water and fat, and since collagen is the main component of the skin, this protein and other connective tissue protein will play a negligible role in gel formation (Visessanguan *et al.*, 2005). So it seems that all these protein functionality was assured on whole dehydrated pork skin, but on minced dehydrated pork rind the significant lower protein values might have had a cause on protein degradation during mincing.

In snacks from dried meat (beef jerkey) protein content between 4h drying samples and 5h drying samples can exceed 50% (Konieczny, *et al.*, 2006). Others authors referred that snacks made with spent hen meat had 24.36% of protein content (Lee, *et al.* 2002), and in meat goat snacks protein value was more or less 25% (Cosenza, *et al.* 2002). On the other hand, in starchy snacks protein content can only represent 6% (Salvador, *et al.* 2009).

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Related to fat content this study had shown that in whole dried pork rind this parameter decreases with no significant differences during the drying process until a final value of 12.02% in whole pork rind with 24h drying (24hD), in comparison with raw skin with 12.70% of fat content. Nevertheless, in dehydrated minced pork rind, raw sample presented the lowest fat content than dried samples with fat contents from 42.81% (12hD) to 39.40% at the final of 24h (24hD) drying process. In fact, as it was previously mentioned, the gel formation of myofibrillar proteins is responsible for retaining and stabilizing water and fat (Visessanguan *et al.*, 2005), and, it might be that the mincing process will contribute to fat content increase since the fat was retained in the minced structure and, on the contrary of water removal during the drying phase, the fat didn't even exudates, as it happened on the whole dried pork rind samples. Others studies with popped snacks with spent hen meat had shown a fat content of 7.15% (Lee, *et al.* 2002), or 17% in goat meat snack sticks (Cosenza, *et al.*2002). Nonetheless, in starchy snacks fat content showed 34% (Salvador, *et al.* 2009).

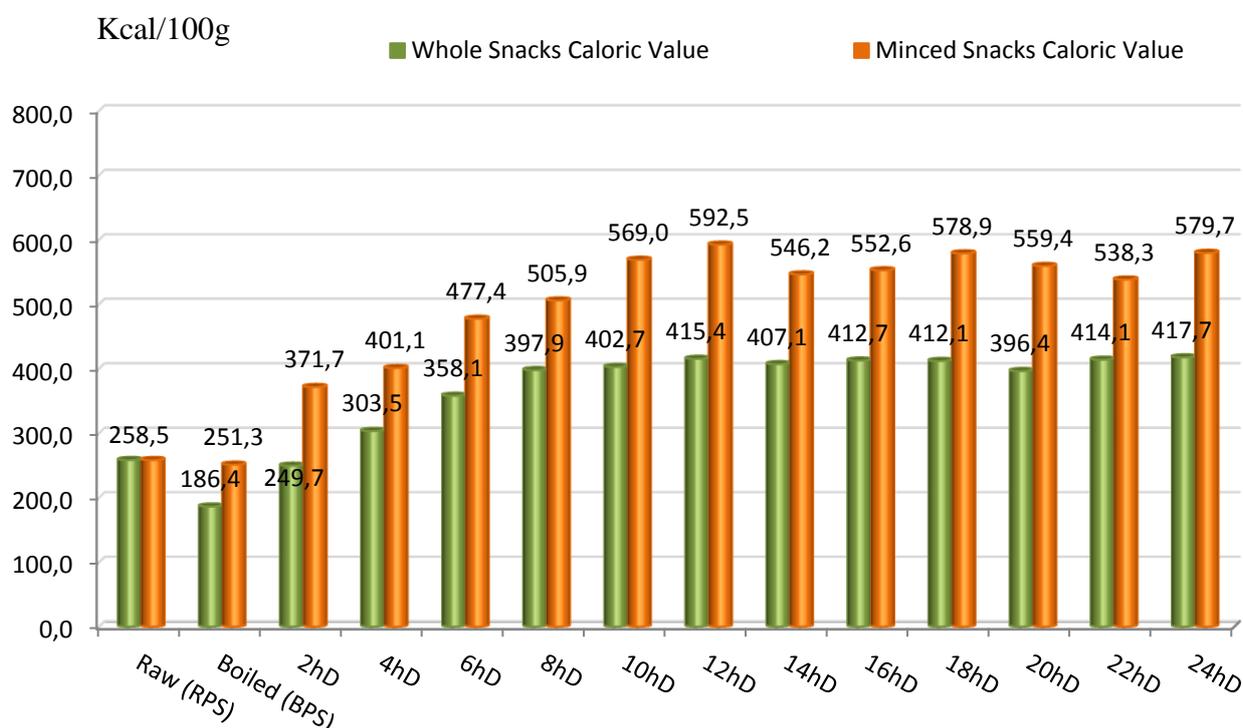


Figure 12 - Whole and Minced Dehydrated Pork Rind Caloric Value (Kcal/100g)

RPS – raw pork skin; BPS – boiled pork skin; 2hD – boiled pork skin dried for 2h at 50°C (...)
24hD – pork skin dried for 24h at 50°C

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In minced dehydrated pork rind, caloric value was higher at the end of drying time, with 579.7 Kcal/100g (24hD) as it was possible to observe on figure 12, since sample 24hD had a higher value than the corresponding dried whole pork rind sample, with 417.7 Kcal/100g. Yet, along drying phase either minced samples or whole samples had of the same value on raw pork rind before boiling phase, however just after this phase, the cooked minced pork rind presented a higher value of 251.3 Kcal/100g (BPS), and along the drying process the caloric values were always higher on minced pork rind. This fact was in agreement with the theory mentioned above about the gel formation due to mincing being responsible for retaining fat, and thus, even with some protein degradation, the fat component is the major contribute to the higher caloric value on minced samples. On whole dehydrated pork rind, the major component responsible for caloric value was proteins.

Finally, the microwave experiments done to the dehydrated whole and minced pork rind were well succeeded on whole pork rind, and thus justifying the following chapter, but the minced pork rind hadn't expanded, and therefore their production ended in this chapter.

Undeniably, in this product processing there is a crushing/grinding phase where collagen structure is broken. Besides that fact, there is an expansion phase during microwave processing and the shrinkage of collagen after heating above 60°C causes a contracture of the collagen sheath and an exudation of moisture from the myofibrils which is responsible for the toughening of the myofibrils between 65° and 75°C (Unklesbay et al., 1999).

4. CONCLUSIONS

Results from this study revealed that the principal aim was achieved: optimize pork rind dehydration from two different preparation of pork rind, one in whole strips and the other minced at 60°C and moulded, and then dried for 24 hours at 50°C. And, thus, we concluded that drying was achieved in around 12-14 hours, in both types of pork rind (whole and minced).

Since this pork skin is an important protein, fat, amino acids, minerals and vitamins source (Zhang et al.,2010; Biesalski, 2005), after drying process the whole pork rinds had a higher protein content than the minced pork rind, which was probably due to protein degradation during mincing phase. Fat content was higher in minced snacks given the fact that mincing contributed to a better posteriors fat extraction during the analysis, and also that gel formation (myofibrillar proteins) also retained fat.

With the aim of this study to valorise the value of a by-product on meat industry, the attempt microwave expansion innovation was well succeeded on whole dehydrated pork rind, since during microwave process only the whole snacks expanded, whereas the minced had not expanded and so compromised their acceptability.

Hence, this type of potential snacks will be furthermore studied in order to guarantee that they could be commercialized as expanded pork rind snacks, instead of the traditionally fried pork rind snacks, since the microwaved will not uptake oil and then will be healthier.

However some facts were not perfectly demonstrated and so the following suggestions are presented as a contribution to optimize this by-product.

So, knowing that skin from loin part is most indicated to obtain this product is preferable to skin from the belie, it's essential to establish a rigorous criteria's to select raw product. Also related with raw product, it seems that subcutaneous fat deposition and its peccary cleaning has strong influence over whole snack's microwave expansion. Besides moisture content data, it would be also very useful water activity (a_w) determination. It seems that during thermal processing (hot air drying and microwave expansion) water loss influences protein interactions and contributes to possible toughness reactions.

Definitely it would be interesting to train panelists with the purpose of familiarize them to these new products textures and appearances. All together do

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instrumental (rheological) tests to snack's texture and correlate it with sensorial evaluation data.

In future perspectives and with the aim of getting a full nutritional information about these innovating puffed snacks it should be completed with whole microwave expanded snack's caloric value.

“Study of Different Methods for Crispness Measurement of a Crispy Snack:
Microwaved Pork Rind”

**CHAPTER II – “STUDY OF DIFFERENT METHODS FOR CRISPNESS MEASUREMENT OF A
CRISPY SNACK: MICROWAVED PORK RIND”**

**(“Estudio De Diferentes Métodos De Evaluación De Crujiencia En Un Snack
Crujiente: Piel De Cerdo Expandida En Microondas)**

ABSTRACT

Consumer demands and global competition are causing the meat sector to embrace new processing technologies and new ingredient systems. Furthermore, several meat industries face with a serious problem: by-products, such as waste tissues, in particular, pork rind. A worldwide concern to develop healthier products containing less fat, absorbed during industrial frying processes, is one of the dominant factors in the latest research trends, prompting studies to alternative technologies, for instance microwave processing. A new approach to investigate the acoustic nature of crispness has recently emerged and it is based on the simultaneous recording of sound and fracture/mechanical events produced during the application of a force to a crisp product, in order to understand and measure sensory properties of foods (i.e., crispness, crunchiness) and establish relationships between emitted sound and structural characteristics of brittle food products. Hence, the objective of this study was to develop a reliable and easy way to implement methodologies for evaluating the sensory crispness/crunchiness of crispy food and how experimental conditions of instrumental sound-texture assessment and microstructure analysis by scanning electron microscope (SEM) could be related to the sensory perception of texture. Boiled rind was cut into strips, dried at 50°C during 6h, 10h and 14h and finally microwave expanded. After production the following analysis were made: moisture before and after microwave, weight loss, degree expansion, instrumental texture analysis (both mechanical and acoustic), image analysis, SEM and sensory analysis. Samples dried 10h and 14h, after microwave had shown higher expansion degree, since both had a lower moisture content (< 15%), than sample dried for 6h, which after microwave had the lowest expansion degree. Even though that in instrumental texture analysis (compression and penetration tests) the samples crispness was not reasonably clear, in both acoustic emission and sensory perception it was fairly clear that it were expanded samples dehydrated for 10h and 14h, those selected as the more crispy.

Keywords: Pork Rind, Microwave, Texture, Sound, Sensory.

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1. INTRODUCTION

Meat and meat product consumption is gradually being considered as causes for increased risks of attracting chronic diseases such as obesity, cancer and stroke (Weiss *et al.*, 2010). While this view often neglects that meat is also an essential factor in maintaining human health since it's a very good source of minerals, vitamins, and contain “complete” proteins (i.e. proteins that in contrast to many plant-based proteins contain all nine of the essential amino acids) (Verbeke *et al.*, 2010). Furthermore, several meat industries face with a serious problem: by-products. There is a need for these major resources to be used more efficiently and profitably. Thus meat and meat-derived products should incorporate concepts and innovations (Madruga *et al.*, 2011; Betoret *et al.*, 2011). In fact, food industry has been manufacturing products with additional acceptable nutritional attributes by means of several processes that added value to by-products such as waste tissues. Generally, these foodstuffs have sensorial disadvantages but, after upgrading, they could be incorporated in human nutrition (Cardoso-Santiago *et al.*, 2001).

For example, some traditional products, such as “Nham”, which is a Thai fermented pork sausage, among typical ingredients used in this product besides minced pork is cooked pork rind, and both include the two major ingredients (90% of the raw mix). Proteins, derived from raw meat and cooked pork rind, exhibit a wide range of functional properties. They are able to form networks and structures, and thus play an important role in textural, sensory and nutritional quality of foods. To be precise, collagen is the main component of the skin and together with other connective tissue proteins can play an important role in gel formation in this type of sausage batters (Visessanguan *et al.*, 2005). However, they are typically included in formulations for improving water binding, processing yield, juiciness and palatability or even blend cost in processes meat (Eilert *et al.*, 1993; Jobling, 1984). As product costs are largely based upon the quantity of meat protein in their formulations, increasing proportion of rind in the formulation can cut down production cost (Visessanguan *et al.*, 2005). Other studies refers the use of pork skin connective gels satisfactorily in reduced-fat “bologna” (Osburn *et al.*, 1997) and also as raw material for meat products (Puolanne *et al.*, 1981; Satterlee *et al.*, 1973).

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Pork rind has been a small niche market in the U.S. snack food industry, but is showing surprising growth (Bechtel, 2001). Pork rinds are a feature in every cuisine where pork is used. Often a by-product of the rendering of lard, it is also a way of making even the tough skin of a pig edible. Pork rinds traditionally are made in a two-step process: pork skin is first rendered and dried, and then fried and puffed (Zeldes, 2010; La Cocina Paso a Paso; The Deep Fryer; Receta de Cortezas de cerdo). These are also called by the Mexican name, “chicharrón”, in reference to the popular Mexican food. Over the past year, “chicharrones” have been embraced by the “mainstream” through the efforts of chefs like Ryan Farr, whose “chicharrones”, are other worldly. They’re incredibly light and when they’re fresh, they snap, crackle, and pop in your mouth (Chicharrones). Nutritionally, 43 % of a pork rind's fat is unsaturated, and most of that is oleic acid, which is the same healthy fat found in olive oil. Another 13 % of its fat content is stearic acid, a type of saturated fat that is considered harmless, because it does not raise cholesterol levels, and in terms of extent of cholesterol oxidation, “chicharrón” only presented 2.3%, which was lower than in other dried and deep-fried food products (Soto-Rodriguez *et al.*, 2008).

A cooking method that enhances the flavour, texture and appearance of food products is frying. In fact, fried products are globally appreciated, although one of the main problems associated with fried food is its high oil content. Therefore, a worldwide concern to develop healthier products that contain less fat, absorbed during industrial frying processes, is one of the dominant factors in the latest research trends in this area (Varela *et al.*, 2011), prompting studies to alternative technologies, for instance microwave processing. Dehydration technology which involves the use of microwaves represents one of the latest advances in this area of food processing (Vega-Mercado *et al.*, 2001). The unique heating mechanisms of microwaves permit dramatic energy saving in many instances since nearly all the microwave energy is absorbed by the material to be heated and it is not expended in heating the air, conveyor or other parts of the equipment (Ala’a *et al.*, 2010).

The sensory properties of food, as well as a large number of non-sensory factors, determine the decisions people make with respect to food (Jaeger, 2006). Texture is the sensory manifestation of the structural, mechanical and surface properties of foods

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detected through the senses of vision, hearing, touch and kinesthetics (Szczeniak, 2002). To take account of the dynamic process of food breakdown in the mouth, since the particular sequence of perceptual events that occur during that phenomena and which triggers swallowing still remains unknown (Lenfant *et al.*, 2009), it is necessary a multidisciplinary approach for studying the relationship between food structure and texture perception, combining sensory research, physiology studies and research into food physicochemical characteristics. A texture judgement can be made by looking at a surface of a food product, and in particular an auditory texture perception results from the sounds food makes when chewed. The hard solid foods like crackers, breakfast cereals, dry toast. Expanded starch products (snacks), biscuits, crispy crusts of bread and fried snacks (Van Vliet *et al.*, 2009) are often called crisp or crunchy (Wilkinson *et al.*, 2000). The architecture of a crispy product can mostly be characterized as an open cellular structure that, in physical terms, is stiff, brittle and fractures and disintegrates abruptly and completely during biting and chewing with accompanying sound emission (Primo-Martin *et al.*, 2008).

A method for evaluating the sensory crispness of snack food products based on direct application of frequency domain spectra of acoustic signals and the use of neural network models was developed with the purpose of measuring the perception of air-conducted sounds and their correspondence with the sensation of crispness (Srisawas *et al.*, 2003). A new approach to investigate the acoustic nature of crispness has recently emerged and it is based on the simultaneous recording of sound and fracture/mechanical events produced during the application of a force to a crisp product. In order to do so an Acoustic Envelope Detector (AED) was attached to a Texture Analyser (Salvador *et al.*, 2009). Indeed, at present, acoustic methods of investigation of food properties are becoming more and more popular. In food science, acoustic techniques have been used as an objective texture measurement, in particular, sound emission has mainly been used to understand and measure sensory properties of foods (i.e., crispness, crunchiness) and establish relationships between emitted sound and structural characteristics of brittle food products (Juodeikiene *et al.*, 2004). Some authors found a very good connection between some recorded sound parameters, the instrumental texture measured, and the sensory (Chen *et al.*, 2005; Varela *et al.*, 2006). Although sensory analysis gives a more complete description of texture of tested

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products, there has been a great interest in developing instrumental techniques to assess some attributes/properties (Roudaut *et al.*, 2002). In fact, since a food sample is a three-dimensional physical structure (Gao *et al.*, 1996), image processing techniques have been used to characterize the internal structure of expanded products (Smolarz *et al.*, 1989; Tan *et al.*, 1994). In addition to internal structure, surface characteristics that are highly correlated with the internal structure have also been shown to convey important information (Tan *et al.*, 1994). These geometric structures can be described with image features that effectively predict the mechanical properties of corn puff (Gao *et al.*, 1996). Therefore, to clearly understand the sensations of crispness and crunchiness in relation to product properties, there is a need for examining spatiotemporal characteristics of sample fracture under dynamic conditions (Dan *et al.*, 2007). Also, structural properties like porosity, characterize the texture of dehydrated products, and thus information on porous formation in foods during processing influences mechanical properties (Koc *et al.*, 2008; Rahman, 2001). Besides, image analysis and porosity, various microscopic techniques are available for characterizing surfaces and internal structures, in particular, scanning electron microscopy (SEM) is often used to study the morphology of the cell walls more closely (Marzec *et al.*, 2007). So, structure has a large impact on the sounds produced when biting into products with crisp, crunchy and crackly textures (Duizer, 2001). Thus the acoustic emission during deformation (or eating) of food products with a crispy/crunchy crust depends on the structure of the food (Primo-Martín *et al.*, 2010), and future work in this area should combine acoustics and microstructure.

Hence, the objective of this study was to develop a reliable and easy way to implement methodologies for evaluating the sensory crispness/crunchiness of crispy food and how experimental conditions of instrumental sound-texture assessment, images analysis and scanning electron microscopy (SEM) could be related to the sensory perception of texture.

2. MATERIAL AND METHODS

The experimental procedures were carried out in different places: (1) Laboratory of the “Área de Tecnología de Alimentos de la Facultad de Veterinaria de la

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Universidad de Extremadura” in Caceres (Spain) – optimization of microwave process by calculating the correct time and power for snack expansion, its moisture and Kramer rheological analysis; (2) factory of the Technology and Applied Sciences Department in “Escola Superior Agrária” of “Instituto Politécnico de Beja” – definition of the conditions of boiling process of the samples, also the optimization drying process by assuming two different temperatures (50°C and 60°C) along 24 hours, and with different sample biometry; and determination of physical and chemical properties, texture analysis and sensorial profile in the Laboratories of Meat Technology, Food Rheology and Sensorial Analysis of Technology and Applied Sciences Department in “Escola Superior Agrária” of “Instituto Politécnico de Beja” in Beja (Portugal); (3) Laboratory of “Instituto de Agroquímica y Tecnología de Alimentos -CSIC” in Valencia (Spain) – texture and sound characterization with a spherical probe and sound recording. In all measurements the number of replica depended from the parameter in analysis, and the scanning images analyses (SEM) were made in "Servicio de Análisis y Caracterización de Sólidos y Superficies de la Universidad de Extremadura" (Badajoz).

Pork skin was removed from the backfat of the slaughtered pigs, was soaked in distilled water overnight and later used for the preparation of rind. Backfat on the skin was trimmed before and after scalding. Scalding in hot water at 61°C lasted 5 min (Abiola *et al.*, 2001) to ensure proper removal of pig hair from follicle. Boiling phase occurred under pressure during 20 min. Boiled rind was cut into strips with different biometrics (10x20mm; 30x65mm; 15x30mm). If not immediately used it was frozen at -20°C for no more than 1 week.

Then, the drying experiments were performed at 50°C during three different periods of time: 6h, 10h and 14h. These times were defined according to the attempt of optimization of the drying process for pork rind, described on the previous chapter.

Microwave expanded snacks were processed from the dehydrated rind strips in a Kunft microwave at 700W during 90 seconds.

The following analysis were made by a specific order: moisture (NP 1614 – 1979) before microwave (bMW) processing and after microwave (aMW), weight loss, degree expansion, instrumental texture analysis (both mechanical and acoustic), image analysis, scanning electron microscopy (SEM) and sensory analysis.

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Moisture Content

Moisture was determined by keeping the sample in an oven at $103^{\circ}\pm 2^{\circ}\text{C}$ until reaching a constant weight (standard technique, official method NP-1614/1979 for meat and meat products). Four replicates were performed for each kind of dehydrated (bMW) and microwave-heated snack (aMW).

Weight Loss

For determining the weight loss during the microwave heating of already boiled and dehydrated pork rinds the following expression was used (Arimi *et al.*, 2008):

$$\% \text{ Weight Loss} = (\text{initial weight} - \text{final weight})/\text{initial weight} * 100$$

The weight loss of the samples during microwave heating was expressed as showed in the previous expression where the initial weight (g) corresponds to the weight of the samples before heating and final weight (g) corresponds to the weight of the samples immediately after microwave heating. Three replicates were performed for each kind of microwave-heated snack.

Degree Expansion

The degree of expansion of the microwave-heated samples was determined as the percentage change in volume between unheated and microwave-heated samples.

Volume was measured by a modified displacement method (AACC, 2000) using millet seeds (figure 13) and the degree of expansion was calculated according to the following expression:

$$\text{Degree of Expansion (\%)} = (\text{Final Volume} - \text{Initial Volume})/\text{Initial Volume} * 100$$

In the previous expression, initial volume (ml) is considered the volume before microwave heating and final volume (ml) is the volume after microwave heating (Arimi *et al.*, 2010). Three replicates were performed for each kind of microwave-heated snack.

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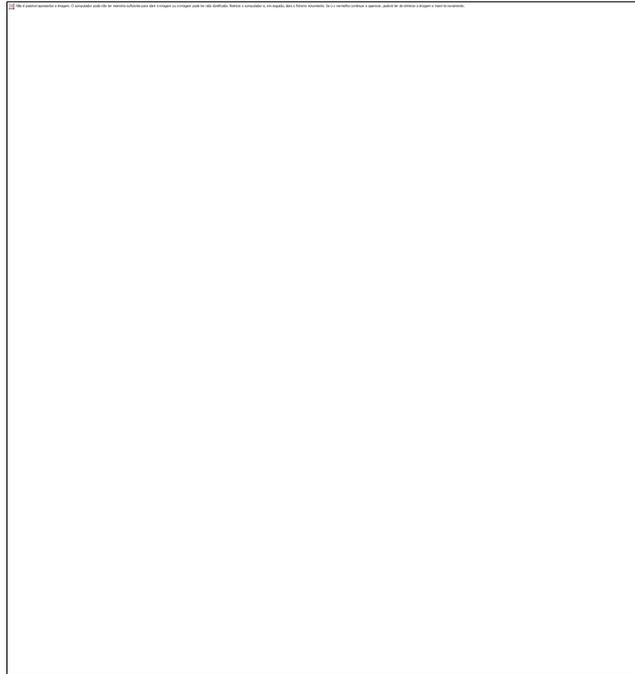


Figure 13 – Measurement of degree of expansion

Instrumental Texture Analysis

A TA-HDi Texture Analyser (Stable Micro Systems, Godalming, UK) was used for the compression tests and a TA-XT plus Texture Analyser was used for penetration tests, both with a 25kg load cell, and the tests settings were the following:

a) Compression Tests

The TA-HDi was used with the Kramer Shear Cell (HDP/KS5) with the following settings: test speed 2mm/s, trigger force 4N, travel distance of the blades 15mm and acquisition points per second 200pps. Ten replications with approximately 12g were performed for each test.

The following parameters were calculated from the force *versus* time curves (figure 14): force and distance of the first breakdown event, displacement (mm) since the first breakdown event until maximum force, maximum force and number of peaks of the curve or fracture events or crispness (drop in force higher than 0.049N).

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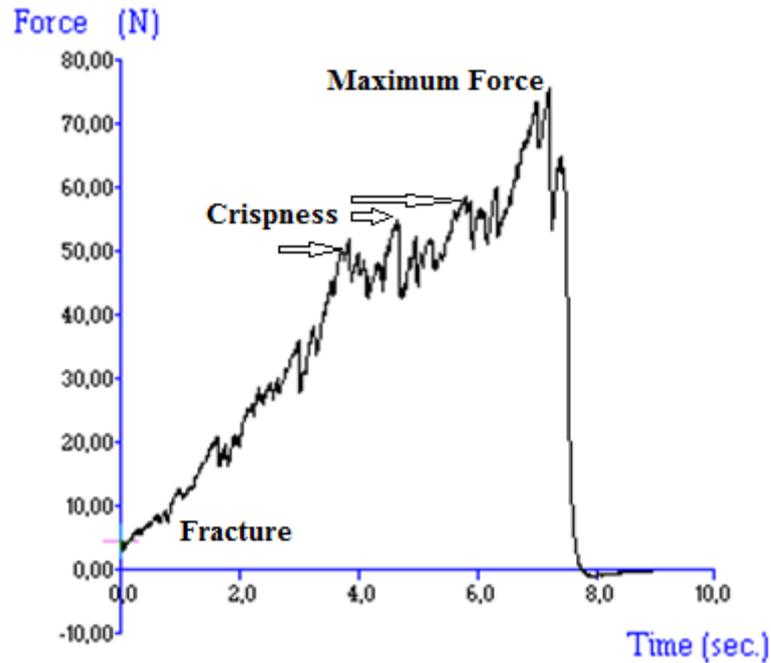


Figure 14 - Force *versus* time Kramer curve showing first breakdown event until maximum force, maximum force and number of peaks of the curve (crispness)

b) Penetration Test and Sound Emission

With the intend for instrumentally assessment of synchronized sound/texture measurements, penetration tests were made in order to imitated a rupture with a tooth-like probe: Volodkevich tooth rig (imitating human biting) (Varela *et al.*, 2009).

A spherical probe (P/0.25S) was used in this test and samples were placed on the Crisp Fracture Support Rig (HDP/CFS) and corresponding platform (SMS). The test speed were 1mm/s, trigger force 5g, travel distance of the probe 3 mm.

An Acoustic Envelope Detector (AED) (figure 15) was used for sound recording with the corresponding software (Texture Exponent 32). The gain of the AED was set at one. A Bruel and Kjaer free-field microphone (8mm diameter), calibrated using an Acoustic Calibrator Type 4231 (94dB and 114 dB SPL-1000Hz) was positioned at 4 cm distance with an angle of 45° to the sample.

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Ambient acoustic and mechanical noise was filtered by the use of a high pass filter of 1 kHz. A low pass filter set the upper calibrated and measured frequency at 16 kHz. The AED operates by integrating all frequencies within the band pass range generating a voltage proportional to the sound pressure level (SPL).

All tests were performed in a laboratory with no special soundproof facilities at room temperature (Salvador *et al.*, 2009). Twenty replications were performed for each kind of expanded sample. The data acquisition rate was 500 points per second (pps) for both force and acoustic signals.

Force/displacement curves were simultaneously plotted. From the force curve the following parameters were extracted: area below the force curve, number of force peaks (drop in force higher than 0.049N), and gradient (slope of the curve up to the first major peak). From the sound curves, the number of sound peaks (drop in sound pressure level higher than 10dB) and the sound pressure level (average of the ten higher peaks, SPL_{max10}) (Varela *et al.*, 2008).



Figure 15 - TA-XT plus Texture Analyser with spherical probe and Crisp Fracture Support Rig and an Acoustic Envelope Detector (AED)

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Images Analysis

Images of each sample slice were acquired by scanning them cut and blue stained surface (Crowley *et al.*, 2000; Datta *et al.*, 2007; Esteller *et al.*, 2006) on a flat-bed scanner (Benq Corporation, 5000 Colour Scanner, USA). Brightness and contrast parameters of the scanner program (MiraScan6, Benq, Taipei, Taiwan) were kept in the default value (zero) for all samples (Pérez-Nieto *et al.*, 2010). The scanned images were saved as TIFF-24bit files and retrieved later for subsequent analysis. Images were cropped, pre-processed (change to grey-level) and segmented (manual thresholding from the histogram of frequencies of grey-level) and pore size measurements were carried out using the same program (MiraScan6, Benq, Taipei, Taiwan).

All the image analysis operations were made using Matlab R2012 and its “Image Processing Toolbox” (The MathWorks Inc., MA, USA). After acquisition, each sample image were isolated from the others, pre-processed to obtain the greyscale image (rgb2gray function) and segmented to a binary image using a threshold value (im2bw function). This value was optimized to give the maximum number of pores in each image having a final mean value of 0,446 for the described working conditions.

The data of pore distribution (number, individual area and roundness) for each sample was obtained from this binary images (bwboundaries function) and exported to Excel for further analysis.

Shapes are physical dimensional measurements that characterize the appearance of an object. Shape features can be measured by combining size measurements, and are relatively simple to determine using image analysis techniques. One of the most widely used shape features with combinations of size measurements for food products is roundness. Roundness gives the ratio of the area of the object and that of a circle with the same perimeter, its value is between 0 and 1, with 1 being a perfect circle (Aguilera *et al.*, 2001; Hicsasmaz *et al.*, 2003; Arellano *et al.*, 2004; Fernandez *et al.*, 2005; Mayor *et al.*, 2005; Riva *et al.*, 2005; Zhu *et al.*, 2005; van Buggenhout *et al.*, 2006).

Roundness is given by the following formula:

$$\text{Roundness} = 4\pi \text{Area}/(\text{Perimeter}^2)$$

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Scanning Electron Microscopy (SEM)

Sensory Analysis

A panel of 15 assessors with experience in the descriptive evaluation (NP ISO 8586-1, 2001) was used to evaluate the three type of microwave expanded pork rinds (dehydrated for 6, 10 and 14 hours). Panellists were trained during months, first in group sessions in order to define descriptors to these kind of products (puffed snacks) and to reach a consensus among panellists on the meaning of every attribute, and then in three individual sessions with different puffed snacks with the aim of training them to recognize specific texture descriptors (hardness, crispness, crunchiness and friable character). To achieve this, it is recommended in handbooks to provide assessors with a definition (Roudaut *et al.*, 2002).

Testing was carried out in a sensory laboratory equipped with individual booths (NP ISO 8586-2, 2001). The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9).

To evaluate hardness the instruction was to bite the whole sample with the incisor teeth until fracture and score the material resistance (Vincent, 2004). To score crispness the instruction was to evaluate altogether during mastication (first three bites), amount and quality of the sound produced (Chaunier *et al.*, 2005; Lazou *et al.*, 2010). To evaluate crunchiness the instruction was to score during mastication the number of layers with incorporated air (Dijksterhuis *et al.*, 2007). Friability was evaluated by assessing the amount of tiny pieces (vitreous state) in which the snack could break during mastication, which was the key on time to subsequent deglutition (Chaunier *et al.*, 2005).

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The samples were served in random order, each on a separate glass tray, identified with a letter random code. Panellists were instructed to rinse their mouths with water between sample evaluations.

Statistical analyses were conducted using a commercial statistical package, Design Expert version 6.01 (Statease Inc., Minneapolis, USA). Statistical significant difference between values was evaluated at $p < 0.05$ level with a comparison test using SPSS 12.0.1 (SPSS Inc., Chicago, Illinois, US).

3. RESULTS AND DISCUSSION

Moisture content, weight loss (%) and degree of expansion (%) on dried samples before microwave (bMW) processing and after microwave heating expansion process (aMW) with different drying times (6h, 10h and 14h) is shown in table 6.

Table 6 - Moisture content, on dried samples before microwave (bMW) processing and after microwave heating expansion process (aMW), together with weight loss (%) and degree of expansion (%) during microwave heating.

	6H	10H	14H	SEM	p
Moisture content (%) (b MW)	18.96 ^a	12.71 ^b	9.76 ^c	0.72	0.001
Moisture content (%) (a MW)	2.44	2.79	2.23	0.11	0.121
Weight Loss (%)	23.75 ^a	12.42 ^c	16.81 ^b	1.68	0.001
Degree of Expansion (%)	344.10	634.66	452.52	55.52	0.073

It was observed that moisture content before microwave expansion (bMW) presented significant differences between all three samples with different drying times ($p=0.001$); more precise samples less dried (6H) had 18.96% moisture content, which was significantly different from samples with 10 h (10H) or 14 h (14H) drying time, with respectively 12.71% and 9.76% moisture content.

In microwave expanded samples, moisture content (aMW) did not show significant differences, and the final values were 2.79% in samples with 10 h drying

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(10H), 2.44% in samples with 6 h drying (6H) and 2.23% in samples with 14 h drying (14H).

Significant differences were also observed for weight loss as a consequence of different drying time: samples dried for 6 hours showed 23.75% of weight loss during microwaving, which was significantly different from samples dried for 14 h and 10 h (10H) (16.81% and 12.42% respectively).

The microwave expanded samples did not present significant differences on degree of expansion between means, but the p value in the ANOVA for the drying time shown a trend ($p = 0,073$) to a significant effect. The final values were 634.66% in samples with 10 h drying (10H), 452.52% in samples with 6 h drying (6H) and 344.10% in samples with 14 h drying (14H). It was also relevant the SEM (standard error mean) value, which was due to the fact that in this parameter were used few samples ($N = 3$) and also this parameter was intrinsically more variable.

Generally, during microwave heating three major processes take place: first there is a temperature increase, second, the product expands and third, water evaporates giving rise to weight losses. The time course of the above processes may be divided into three distinct phases; heating up, moisture loss + expansion and moisture loss + burning. On continued heating evaporation of water takes place and once sufficient moisture has been driven off (moisture content less than 15%) the matrix appears to set, like if it had reached its glassy transition state, and maintains the expanded structure on cessation of heating (Arimi *et al.*, 2008).

In fact, on heating, the temperature of the water in the sample reaches boiling point, bubble nucleation is initiated and vapour bubbles are formed. These bubbles then grow within the product matrix causing it to expand as a foam. But since water vapour acts as a plasticizer and in addition creates the pressure that induces expansion, if insufficient evaporation occurs and too much moisture (plasticizer) remains in the sample, as is the case for short heating times or for samples with higher moisture, the matrix can collapse on cooling. So, this explains the difference between expansion degree in samples 10H and 14H, which had a lower moisture ($< 15\%$), when compared with 6H sample expansion degree, that was the lowest.

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Table 7 shows the parameters from the force *versus* time Kramer curves. More precisely, the force and distance of the first breakdown event (fracture) and distance to fracture), displacement (mm) since the first breakdown event until maximum force, maximum force and number of peaks of the curve (fracture events) as sense of the instrumental crispness, on microwave heating expansion samples dried at different drying times (6h, 10h and 14h).

Table 7 - Parameters from the force *versus* time Kramer curves: force and distance of the first breakdown event, displacement (mm), maximum force and number of peaks of the curve (fracture events) on microwave heating expansion samples dried at different drying times (6h, 10h and 14h)

	6H	10H	14H	SEM	p
Fracture (N)	13.84	17.63	19.05	1.18	0.180
Distance to Fracture (mm)	2.22 ^b	3.37 ^a	2.82 ^{ab}	0.19	0.039
Maximum Force (N)	92.63	77.01	101.98	4.44	0.063
Crispness (number of peaks †)	94.20	87.80	88.70	1.57	0.203
Displacement (mm)	11.97	10.80	11.62	0.24	0.117

In the above table, it was showed that all three samples dried at different drying times (6h, 10h and 14h) did not show significant differences between each other, with respectively 13.84N (6H), 17.63N (10H) and 19.05N (14H), but still this parameter presented a high SEM value.

The only parameter that was significantly affected by the drying time was the distance to fracture. Thus, samples with 10h drying time (10H) and 14h drying time (14H) did not show significant differences between each other, with respectively 3.7mm and 2.82mm, but on the other hand, samples with 10h drying time (10H) showed significant differences with samples dried for 6h (6H), with 2.22mm of distance to fracture. Nonetheless, samples with 6h drying time (6H) and 14h drying time (14H) were not significantly different.

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As shown in the preceding table, all three samples dried at different drying times (6h, 10h and 14h) did not evidenced significant differences in maximum force between means, although the P value of the ANOVA revealed a trend ($P=0.063$), and in fact samples 14H presented a much higher maximum force (101.98N), probably due to their lower moisture, followed by 6H (92.20N), perhaps owed to its collapse on cooling, as it was mentioned above, which contributed to a rubbery state. In this parameter it was also noteworthy its high SEM value. Samples dried for 10H showed the lowest value (77.01).

Crispness did not show significant differences between each other, with 94.2 for samples dried 6h (6H), followed by 88.7 for samples dried 14h (14H) and 87.8 for samples dried 10h (10H).

Displacement refers to the displacement since the first breakdown event until maximum force; besides it was not significantly influenced by the drying time ($P=0.117$); in fact, it was expected to behave similar to maximum force and crispness parameters. Indeed, samples 6H presented the highest value with 11.97mm, followed by samples 14H with 11.62mm and finally by samples 10H with 10.80mm.

Accordingly to compression Kramer texture parameters, and more particularly to measurement of peak force, it seemed that these parameters were not good indicators of crispness in this present work, yet had been already defined as a good crispness marker in compression tests (Dogan *et al.*, 2005).

Table 8 shows the parameters from the force curve obtained in a simple test with a spherical probe and Crisp Fracture Support Rig (HDP/CFS), in which the samples were placed. At the same time, sound recording were performed by an Acoustic Envelope Detector (AED). The following data were recorded force curve: area below the curve (Force Work), number of force peaks and gradient or slope of the curve up to the first major peak. From the sound curves, the number of sound peaks (drop in sound pressure level higher than 10dB) and the sound pressure level (average of the ten higher peaks, SPL_{max10}) were also measured on microwave expanded samples dried for different times (6h, 10h and 14h).

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Table 8 - Parameters from the force curve obtained with the Crisp Fracture Support Rig and an AED sound recorder: area below the force curve, (Force Work), number of force peaks, gradient, the number of sound peaks and SPLmax₁₀ on microwave heating expansion samples dried at different drying times (6h, 10h and 14h)

	6H	10H	14H	SEM	p
Gradient (N/s)	18.17 ^a	13.56 ^{a,b}	13.05 ^b	0.89	0.032
Force Work (N.s)	82.70 ^a	46.08 ^b	59.93 ^b	4.12	0.001
Number of Force Peaks	47.62 ^b	50.45 ^b	67.8 ^a	2.47	0.001
Count Peaks Acoustic	120.24 ^b	132.73 ^b	215.00 ^a	8.30	0.001
SPLmax₁₀ (dB)	93.16	93.13	95.41	0.42	0.039

SPLmax₁₀: sound pressure level (average of the ten higher peaks)

The parameter gradient or slope was significantly affected by drying time, and had shown significant differences between average values of samples 6H and samples 14H, with respectively 18.17 N/s and 13.05 N/s. However, neither samples 6H and 10H, nor samples 10H and 14H had shown significant differences. The force work was significantly different in samples 6H, with 82.70 N.s, as compared to samples 10H and 14H, that had shown 46.08 N.s and 59.93 N.s., respectively.

In summary, these two force parameters (slope and work force) were considerably higher in sample dried for 6h, most likely due to the fact that its moisture before microwave heating was 18.96% and since insufficient moisture hasn't been driven off during drying (moisture content higher than 15%), the matrix didn't maintained the expanded structure on cessation of heating (Arimi *et al.*, 2008) since water vapour acted as a plasticizer and in addition the matrix collapsed on cooling, giving rise to a more elastic structure, which caused these high values on penetration test.

The number of peaks force (thresholding 0.049N) showed significant difference between samples dried 14h (14H) with 67.8 peaks and samples 6H and 10H, with 47.62 and 50.45 respectively, than as drying time was higher, so it was the number of peaks force. This means that a longer and more intense drying before microwaving would lead to the number of fractures during chewing (REF), which in turn seems to be related to crispness (REF).

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In fact, some studies with crispy products had shown that crispness is negatively affected by increasing water content (Primo-Martin *et al.*, 2008), which might support the lowest crispness of samples dried for 6H obtained in this instrumental texture test, since its respective moisture content was the highest in all three samples after dehydration with 18.96%. As for the previous parameter, water would act as a plasticizer, increasing the elasticity of the product, and thus, decreasing the number of fracture events.

In relation to sound parameters, acoustic peaks (with sound pressure level higher than 10dB) in samples dried for 14h were significantly higher ($P=0.001$) than those from samples dried for 6h and 10h, (215 vs. 120.24 and 132.73 respectively). These results were in reasonable agreement with the results obtained for count peaks force, and would reflect the lower moisture content of samples dried for 14h and the role of water in microwaved pork rinds as a plasticizer.

SPL_{max10} represents the sound pressure level, which is given by the average of the ten higher peaks. Although the ANOVA detected a significant influence ($P=0.039$) of drying time on this parameter, the Tukey tests was not able to find significant differences between the average values of all three type of samples. The trend was to higher values for SPL_{max10} with longer dehydration time, and so samples with 14h drying time had 95.41dB and samples with less drying time (6H and 10H samples) had more or less 93 dB. This behaviour was therefore similar to that found for the previous variables, with higher moisture level leading to more elastic and less crispy microwaved product, probably due to the effect of water as a plasticizer.

Added to the eventual production weakness during expansion process, some references showed that upon adsorption of water a crispy product becomes pliable, ductile and soft and loses its crispness and consequently its acceptability by the consumer (Primo-Martin *et al.*, 2008).

In resume, in this instrumental texture analysis, it was clearly shown that samples dried for 14h were the crispiest, considering the higher values for sound parameters and count force peaks (crispness), followed by 10H samples, which did not show significant differences with 14H ones in terms of acoustic emission, and the less crispy were those dried for 6h.

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Thus, we have shown in this work that the instrumental penetration method to evaluate texture, particularly, crispness parameter, was more sensitive for detecting differences in crispness than the previous one using the Kramer compression method.

In effect, it had given the impression that these texture parameters in penetration test were good crispness markers, in addition to the fact that crispness weren't well detected on other instrumental methods (Varela *et al.*, 2009); in the present work it had measured somewhat well the relation between this texture parameters and moisture content. However, one must be careful in order to understand the implications of the interpretation in a combined analysis of fracture behaviour and acoustic emission. It is necessary to understand some of the basics of fracture mechanics (Saeleaw *et al.*, 2011).

The following figure shows a blue stained sample on the surface of a flat-bed scanner, after microwave heating expansion. On the scanned image analysis of a microwave expanded sample with 10h drying time, presented on figure 16, it was possible to identify the distribution of pore, in order to determine the percentage of pores at each category of pores sizes (mm^2), the number of pores and their roundness.

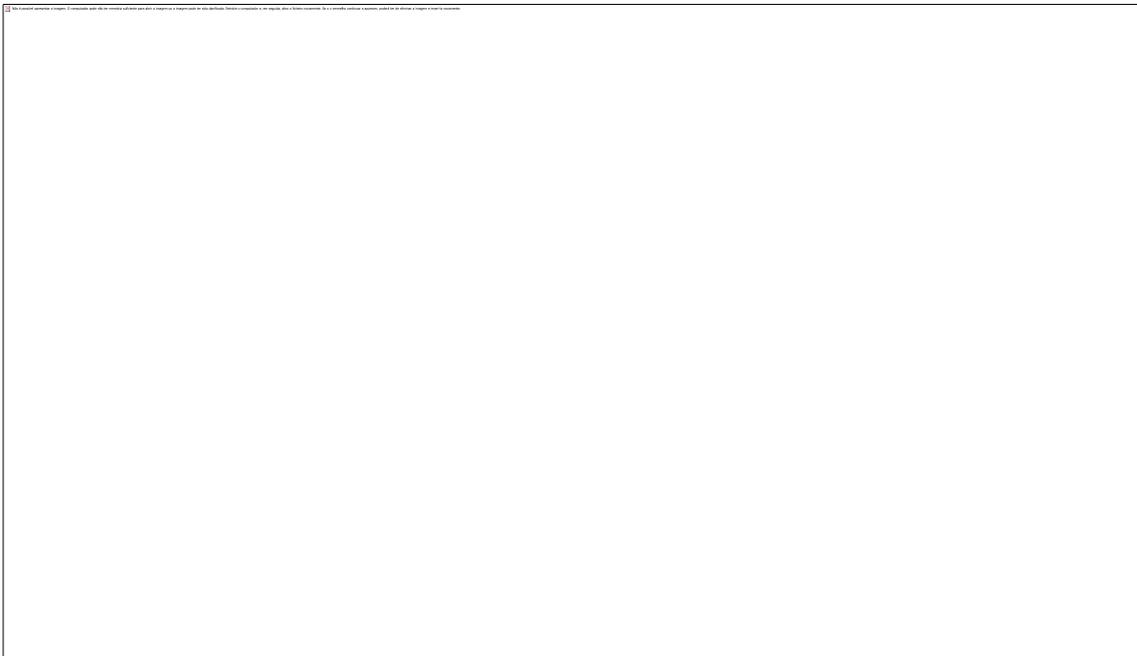


Figure 16 – Picture of scanned samples dried for 10h (10H)

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Scanned image analysis (distribution of pore sizes and pore and roundness) are presented on table 9 for samples dried for three different times (6h, 10h and 14h) and after microwave heating expansion. The results were expressed in percentage of pores at each category of pores sizes (mm^2) and roundness.

It was notable that, as a general trend, for most categories of pore sizes there were no significant differences between all three samples. Nevertheless, for categories of pores with higher diameters (pore size between 3.99 - 4.43 and pores greater than 4.43 mm^2) there was respectively a significant difference ($P=0.020$) and a significant trend ($P=0.089$) as a consequence of drying time. In both cases, samples dried for longer times showed a higher proportion of this type of larger pores. Thus, besides the fact that all samples mainly showed a majority of small size pores, as drying time was longer, there was a trend to a higher proportion of larger pores.

The development of such an aerated structure takes place during microwaving, when the moisture within the product is rapidly heated, leading to formation of steam, which increases the pressure inside cells of water surrounded by degraded connective tissue (gelatin). As a consequence, bubbles are formed throughout the structure.

As long as gelatin is hydrated enough, the structure is elastic and bubbles grow. In the moment in which water content is too low, a glassy structure of proteins is formed and bubbles set.

If water content was very low before microwaving, then the amount of steam formed could be not enough to expand the bubbles. If water was too high, then the bubbles may collapse after heating, since the bubble walls still could be elastic after microwaving.

In other studies (Vincent, 2004), images analysis had shown that crispness was related to the number of air cells and their size, since more and smaller cells result in less crisp texture, and it was also associated to the thickness of the walls between cells, and therefore, in view of the fact that cells collapse on cooking, so larger cells result from the fusion of smaller ones and the cell walls adhere, increasing the thickness, and thus thicker walls give crisper texture.

The roundness of pores was also significantly affected by drying time ($P=0.031$), the longer the drying time, the higher the roundness values for pores in the image analysis.

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Table 9 – Pores size (mm²) on microwave processed samples dried at different drying times (6h, 10h and 14h)

Pore size (mm ²)	6H	10H	14H	SEM	p
0.00 - 0.44	87.8%	86.4%	86.5%	0.004	0.309
0.44 - 0.89	5.1%	5.0%	4.2%	0.003	0.431
0.89 - 1.33	2.6%	2.5%	2.4%	0.002	0.944
1.33 - 1.77	1.4%	1.2%	1.2%	0.001	0.816
1.77 - 2.22	0.6%	0.6%	0.6%	0.001	0.963
2.22 - 2.66	0.4%	1.1%	0.7%	0.001	0.084
2.66 - 3.10	0.3%	0.3%	0.5%	0.001	0.225
3.10 - 3.55	0.1%	0.5%	0.4%	0.001	0.141
3.55 - 3.99	0.2%	0.2%	0.5%	0.001	0.172
3.99 - 4.43	0.2% ^b	0.2% ^b	0.7% ^a	0.001	0.020
>4.43	1.2%	1.9%	2.2%	0.002	0.089
Roundness	0.458 ^a	0.473 ^{ab}	0.485 ^b	0.004	0.031

In other studies about dehydration influence on physical changes, in particular, in porosity and shape (roundness) factors, it was stated that roundness decreased during the dehydration, since the tissues suffer deformations as a consequence of the water removed in the material (Mayor *et al.*, 2005; 2011; Fernandez *et al.*, 2005; Riva *et al.*, 2005).

However, in this study, during microwave expansion, if water content before microwaving was too high, then after heating, the structure will collapse since the bubble walls still could be elastic after microwaving, and thus determine a lower roundness, when compared with the samples with lower water content before microwaving, which had higher roundness due to a glassy structure of proteins that was formed and so the bubbles set and define the roundness.

The alterations of porosity as a function of drying time on microwaved expanded samples a visualized with SEM photographs presented in Figures 17 (a), (b) and (c), for samples 6H, 10H and 14H, respectively.

“Study of Different Methods for Crispness Measurement of a Crispy Snack:
Microwaved Pork Rind”

Samples 10H and 6H have like bubbles of steam puffing the structure, whereas these were not seen in sample 14H, possibly due to the fact that this hadn't enough moisture.

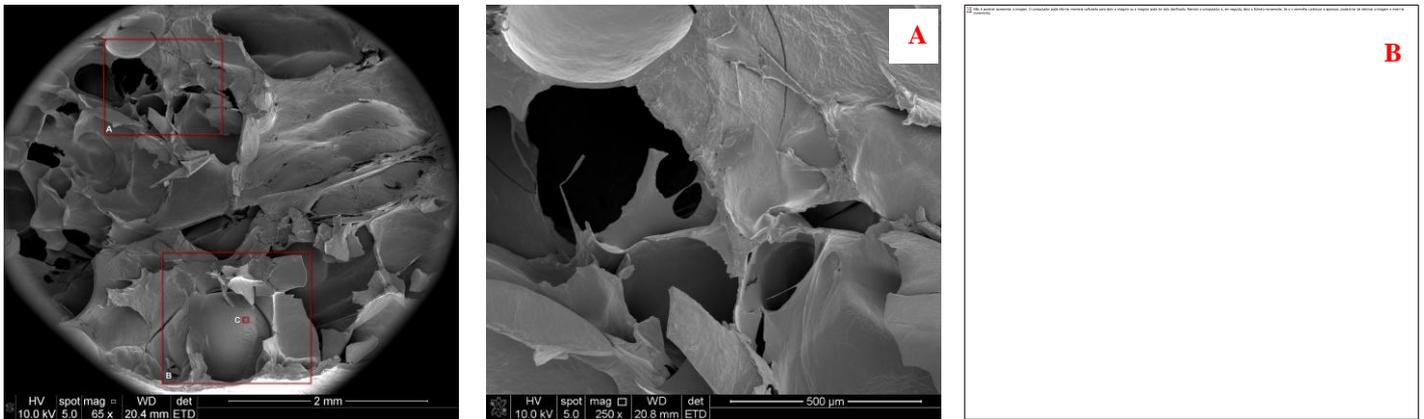


Figure 17 (a) – SEM photography of microwaved expanded sample dried 6H

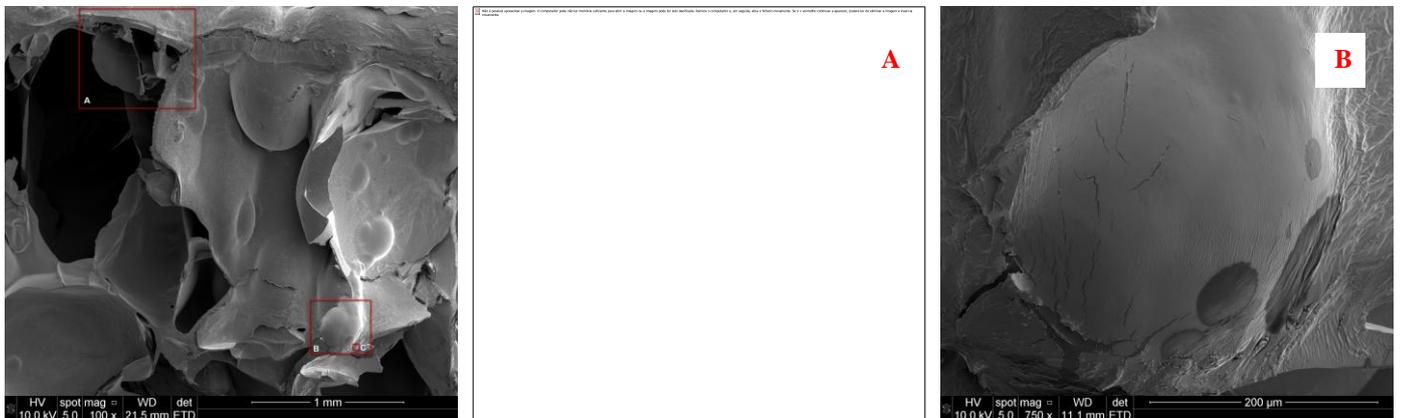


Figure 17 (b) – SEM photography of microwaved expanded sample dried 10H

It was possible to see that structure is closer in 14H, perhaps because of the initial amount lower water content before microwaving. Besides that, it was also visible that their walls are thinner.

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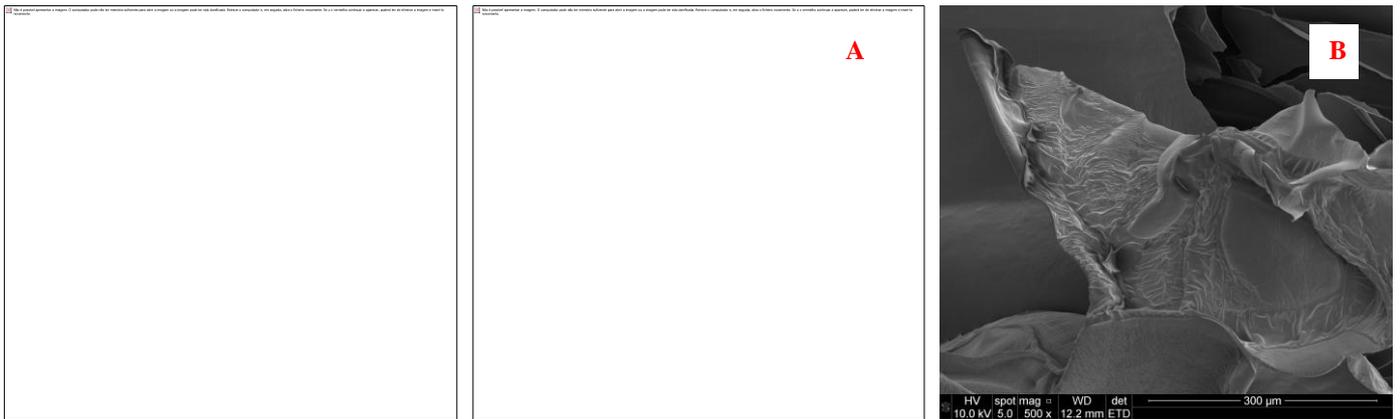


Figure 17 (c) – SEM photograph of microwaved expanded sample dried 14H

The results on table 10 were obtained with a panel of 15 assessors with experience in descriptive evaluation, who were asked about seven traits of microwave expanded snacks and also performed an acceptability question about these samples.

In each session panellists evaluated the three type of samples for the specific texture descriptors (hardness, crispness, crunchiness and friable character), descriptors related to samples appearance (number of holes and holes size) and, finally, samples overall acceptability.

The results showed that except for flavour ($p > 0.05$), all attributes had significant differences between all three samples.

In the number of holes, samples 14H and 10H, with respectively 6.27 and 5.58 scores, had significant differences with sample 6H with 4.92. For holes size, the scores were 6.09 and 6.00, respectively for 14H and 10H samples, and with significant difference 3.76 for sample 6H.

So, as it was mentioned above in table 4, these two samples (10H and 14H) had less proportion of small pores (holes) and higher proportion of bigger pores (holes), which is in agreement with sensory parameters related to holes (pores) appearance. Also, this fact was basically in agreement with the results obtained in the image analysis, where the samples dried for longer times (10H and 14H) had shown a higher proportion of bigger pores.

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Table 10 – Sensory attributes on microwave expanded samples dried at different drying times (6h, 10h and 14h)

	6H	10H	14H	SEM	p
Number of Holes	4.92 ^b	5.58 ^a	6.27 ^a	0.12	0.003
Holes Size	3.76 ^b	6.00 ^a	6.09 ^a	0.40	0.000
Hardness	7.38 ^a	4.83 ^b	4.82 ^b	0.39	0.000
Crispness	6.67 ^a	6.39 ^b	6.44 ^{a,b}	0.05	0.042
Crunchiness	5.40 ^b	6.22 ^a	6.04 ^a	0.13	0.001
Friability	5.23 ^a	4.33 ^b	4.45 ^b	0.12	0.016
Flavour (toasted)	4.69	4.17	4.82	0.08	0.065
Overall Acceptability	4.00 ^a	5.25 ^b	4.82 ^b	0.22	0.000

The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9)

In terms of hardness, there was a significant difference between sample dried for 6h (7.38) and the other two type of samples (10H and 14H), that scored, respectively, 4.83 and 4.82. This fact wasn’t stunning in view of the values obtained for the parameters slope (N/s) and force work (N.s) in the Kramer compression and penetration tests, which were greater in 6H samples than in the others.

This was probably due to the matrix collapse after microwave expansion, due to the higher moisture content of the pork skins before microwaving, that could have led to a predominant rubbery state, which in turn could produce bubbles collapse and more elastic and hard structure.

In figure 18 are shown the following sensory parameters: number of holes and holes size, hardness, crispness, crunchiness, friability, flavour (toasted) on microwave processed samples dried at different drying times (6h, 10h and 14h).

“Study of Different Methods for Crispness Measurement of a Crispy Snack:
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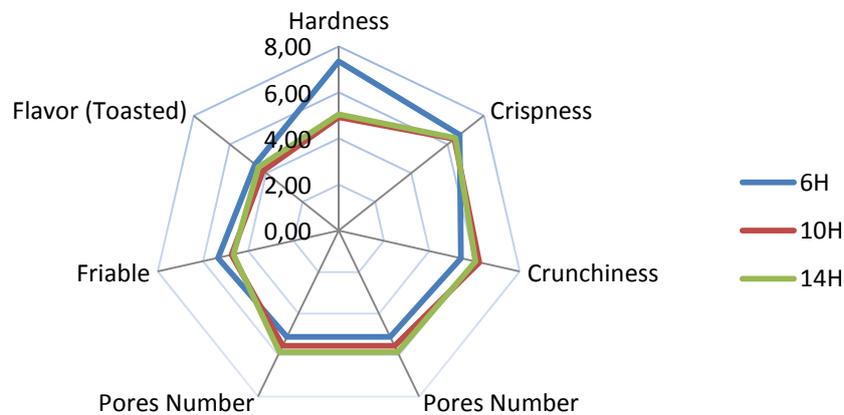


Figure 18 - Sensory parameters: number of holes and holes size, hardness, crispness, crunchiness, friability, flavour (toasted) on microwave processed samples dried at different drying times (6h, 10h and 14h)

The perception of sensory crispness presented significant differences between samples dried for 6h (6H), which scored 6.67, which was the highest value, and samples with 10h drying time (10H) with the lowest score of 6.39. On the other hand, these two samples (6H and 10H) hadn't shown significant differences with sample dried for 14h (14H), with a score of 6.44.

This was surprising, since most instrumental parameters pointed out to higher crispness in samples dried for longer times. It could be that part of the crispness detected by assessors was due to the hardness, counteracting some other parameters related to the physical structure (number of pores, sound during compression, number of failure peaks). Crispness is, as other textural parameter, a very complex sensory parameter, and thus, it results very difficult to assign it to a single objective measurement.

Sensory crunchiness in samples dried for 6h (6H) had shown significant differences with a score of 5.40 with both the other two type of samples (6.22 and 6.04 for 10H and 14H, respectively).

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This attribute was expected to be in accord with count peaks force in penetration test, and in fact samples dried for 6h (6H) were those that presented minor values in such instrumental texture test. This was most likely due to the reasons exposed above, related to the matrix collapse which produces a less aerated structure and thus, less layers to be broken during chewing, which is the structure behind a crunchy sensation (REF).

In addition, a low number of force and acoustic events normally are taken as an index of low crispness and crunchiness; however, a careful observation and analysis of the fracture pattern is necessary (Salvador *et al.*, 2009), since sometimes the force-displacement curves profile may show larger or smaller fracture events, suggesting that the horizontal axis of these spectra can be divided up into a series of regions each characteristic of a type of texture, progressing from crumbliness through crispness and crunchiness to hardness. If indeed these textures are distributed along a mechanical continuum, it is not surprising that they should be difficult to distinguish by a sensory panel (Vincent, 2004).

The fact that crispness and crunchiness didn't show significant differences might be related with an old subject, which is the question whether crispy and crunchy refer to the same sensory concept. The strong positive correlation observed in some studies where crispness and crunchiness were estimated by the same panel, on the same products favours the hypothesis of two labels for an unique concept. However, other works suggested those attributes has different concepts (Roudaut *et al.*, 2002).

The friability shows significant differences between samples dried for 10h and 14h, (respectively 4.33 and 4.45) and samples dried for 6h (5.23).

There was a trend ($P=0.065$) for the effect of drying time on the sensory flavour. Thus, 14H samples presented the highest value (4.82) while samples dried for 10h had shown the lowest score (4.17).

Finally, in terms of overall acceptability, samples dried for 10h and 14h were significantly different to samples dried for 6h (5.52 and 4.82 vs 4.00). It was noteworthy the panellists preference for expanded samples dried 10h (10H) and 14h (14H). As it was predictable due to its texture lacks, the lower overall acceptability value for expanded snacks with 6h dehydration (6H).

4. CONCLUSIONS

When a crispy food is broken, fractured or crushed, characteristic sounds are produced due to the brittle fracture of the cell walls. Measuring acoustic and force peaks during a penetration test using a spherical probe seems to be more sensitive to detect the physical events when microwaved pork rinds are broken than textural parameters obtained during compression using a Kramer cell. In fact, the former provide information that fit with the moisture content and structure of the product.

Scanned image analysis method, by information of distribution of pore sizes, pore and roundness permit to conclude the fact that as drying time was longer, and thus, moisture content lower, there was a trend to a higher proportion of larger pores. So, during the breakdown event of fracturing these solid structures of microwave expanded snacks, besides the instrumental acoustic and texture characteristics, the structure moreover constitutes an important instrumental tool to predict the sensory perception of crispy/crunchy behaviour

Above and beyond the fact that most instrumental parameters pointed out to higher crispness in samples dried for longer times, sensory crispness had an inverse behaviour, being higher in sample dried for a shorter time. It could be that part of the crispness detected by assessors was due to the hardness, counteracting some other parameters related to the physical structure (number of pores, sound during compression, number of failure peaks). In addition, results indicated that a certain degree of sensory hardness is necessary for crispness perception. And so, crispness is, as other textural parameter, a very complex sensory parameter, and thus, it results very difficult to assign it to a single objective measurement.

Sensory crunchiness was in accord with instrumental penetration test, since samples less dried were those that presented minor values in such instrumental texture test. This was most likely due to the matrix collapse which produces a less aerated structure and thus, less layers to be broken during chewing, which is the structure behind a crunchy sensation. At last, it was noteworthy the sensory preference for expanded samples with longer drying times; perhaps due to the former afford information on texture characteristics and physical structure.

CHAPTER III – “IMPROVEMENTS IN BATTERED FOOD”
(INOVACIÓN EN ALIMENTOS REBOZADOS”)

Abstract

The aim of this study was to study the effect of different batter formulations with uncommon ingredients (CO₂ and ethanol) besides water and flour on different physical, chemical and sensory characteristics of fried battered squid freshly produced and after 48 H refrigeration and subsequent oven reheating. Moisture and fat content, instrumental color, instrumental texture analysis and sensory characteristics after frying process (day 0) and after oven reheating (day 2) were analysed. Substitution of water with ethanol in batters led to significant changes in water and fat content; the highest values for these two parameters were on batters produced with water, either without CO₂ (43.36% and 36.33% respectively) or with CO₂ (38.88% and 29.44% respectively), while in those produced substituting the water by ethanol the contents were 11.63% and 18.38%, without CO₂ and with CO₂, respectively. Instrumental texture parameters, namely, crispness and hardness, were significant different on water batters and on ethanol batters; and thus hardness was higher on water batters, and crispness was higher on ethanol batters, and during storage, CO₂ incorporation increased ethanol batter crispness. In terms of color, there were not significant differences between all four battered samples, even after regeneration on oven (day 2). Adherence of batter had significant differences at day 2, mostly on water formulation without CO₂, with the lowest score. Sensory hardness was associated to textural parameters, in particular to maximum force, work force and slope, since water formula without CO₂ had the highest values in these three texture parameters, which fact was confirmed by panellists, whom definitely establish this sample as the least crispy and crunchy in the face of the most spongy and elastic. Overall acceptability in water batter without CO₂ had the lowest value and ethanol batter without CO₂ had the highest value.

Keywords: Frying, Oven, Batters, Kramer Texture, Sensorial Crispness

1.INTRODUCTION

“Battering and frying” is a traditional method for preparing foods throughout the world; empiricism has dominated its application for decades (Fiszman *et al*, 2003). Undeniably, coating preserves and enhances food quality (Varela *et al*, 2011; Dogan *et al*, 2005), since fried batters improve food sensory quality (Shih *et al*, 2010) and are used for adding value to some food products by improving their texture, flavour, weight and volume (Baixauli *et al*, 2003). In parallel with those facts, convenience foods are in great demand due to social and cultural changes in recent years and one of the most important foods in this group is battered products (Albert *et al*, 2009). These products are common both in high-convenience consumer societies and in developing countries (Varela *et al*, 2008).

Batters are covered on the surface of food products to form the crust during deep-fat-frying. The crusts of the fried products can provide the crispy texture, golden yellow color and can act as barrier against moisture loss by protecting the natural juices of foods (Chen *et al*, 2009). There are some physical and chemical changes occurring inside the food material during frying. The rapid evacuation of moisture during this thermal processing creates pores in the product (Barutcu *et al*, 2009) and so porosity increases during frying (Dogan *et al*, 2005). The fact that these products are tender and moist on the inside with a porous crunchy crust provides an increase in palatability that is responsible for the great acceptance of these foods (Freitas *et al*, 2009).

Crispness is a highly valued textural characteristic; in particular, in breaded and battered foods like fish, seafood, poultry, cheese or vegetables (Dogan *et al*, 2005). Appearance, colour, texture, adhesion and flavour are important factors in consumer perceptions of coated foods and crispness is the most critical property that determines consumer acceptance, as the crisp outer layer contrasts with soft core (Loewe, 1993; Maskat *et al*, 2002; Freitas *et al*, 2009). In fact, one of the most important functions that an edible film performs is to resist the migration of moisture (Varela *et al*, 2011) and thereby ensuring a final product that is tender and juicy on the inside and at the same time crisp on the outside (Fiszman *et al*, 2003).

The crust formed during deep frying of battered products is primarily appreciated for its crispness, which is a key driving factor for consumer preference

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(Primo-Martín *et al*, 2010; Primo-Martín *et al*, 2011). Indeed, crispness is one of the most important quality aspects of battered food and extensive research has been conducted to investigate the influence of batter ingredients on crispness development (Sanz *et al*, 2007). To achieve the desirable texture of crust in fried battered products, design of appropriate ingredients with wide-ranging functionalities is available (Chen *et al*, 2008). In fact, different tempura-type batter mixtures vary widely, depending on the type of food being batter-coated and the specific characteristics desired for the final products (Salvador *et al*, 2005).

A batter can be defined as a liquid mixture composed of water, flour, starch, salt, leavening, and other minor ingredients (Baixauli *et al*, 2003; Suderman, 1993), into which food products are dipped prior to cooking (Albert *et al*, 2009). Flour functionality in batter systems depends largely on the two major constituents of all flours, starch and protein. The proteins in batter provide structure and increase the coating pick-up values and final yield in the fried products (Fiszman *et al*, 2003). Gluten is a tough, elastic substance that acts as a net, trapping and holding air bubbles in batter and it is traditionally associated with great adhesion and crispness in final products (Breuil, 2001). However, depending on the quantity and quality of gluten and the available water, the resultant structure can be as firm as bread dough or as flowing as batter, thus determine the products final texture (Loewe, 1993). Therefore, batter formula, frying temperature, frying time, heating method, product shape, and frying oil will influence the quality of crust (Chen *et al*, 2009; Baixauli *et al*, 2003; Altunakar *et al*, 2004).

Blumenthal (2007) developed a batter, which principle is based the use of alcohol instead of water, more precise, vodka and beer, despite the flour. Batter preparation was made in a siphon with CO₂, in order to achieve a batter with more bubbles, and therefore, a coater less thickener and more crispy. Alcohol, promotes gluten rapid dissolution, avoiding its characteristic elastic network and also reduces water amount absorbed by starches granules. Finally, during frying, alcohol evaporation precedes water evaporation, and thus, after frying, the coater dries quickly and becomes golden and crispy, avoiding seafood to loss water content and also during overheating, it will protect proteins muscle from shrinkage, and, hence, from an increase on toughness. Most of all, this coater remains crispy after reheated on oven.

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Other cooking techniques such as conventional and microwave ovens can be used, but with the latter, the final texture is normally unsatisfactory (Albert *et al*, 2009). Some studies proved that baking could be used to prepare battered food, mainly in reheated processing in an oven (Jackson *et al*, 2008). The microwave oven is now a well-established method for cooking or heating food, primarily because of its quickness. In fact, current trends seek battered foods without a detrimental effect on crispness (Primo-Martín *et al*, 2011).

Thus, the objective of this study was to study the effect of different batter formulations with uncommon ingredients (CO₂ and ethanol) besides water and flour on different physical, chemical and sensory characteristics of fried battered squid freshly produced and after 48 H refrigeration and subsequent oven reheating, and to implement methodologies for evaluating the sensory crispness/crunchiness of fried battered squid and oven reheated fried battered squid. And also determine instrumental texture assessment that could be related to the sensory perception of texture.

2. MATERIAL AND METHODS

The experimental procedures took place in the Laboratory of the “Departamento de Producción Animal y Ciencia de los Alimentos de la Facultad de Veterinaria de la Universidad de Extremadura” in Caceres (Spain) – definition of each four formulations for batter, definition of the conditions of frying process of the different formulations, also the optimization of reheated process in oven, in the factory of the Technology and Applied Sciences Department in “Escola Superior Agrária” (ESA) of “Instituto Politécnico de Beja” (IPB) – production of each four formulations for batter, and frying process of the different battered squid rings, also the reheated process in oven after 48 hours in refrigeration at 7°C, and determination of physical and chemical properties, texture analysis and sensory profile in the Laboratories of Meat Technology, Food Rheology and Sensorial Analysis of Technology and Applied Sciences Department in ESA – IPB.

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Experimental Design

Frozen squid rings were purchased in a local supermarket and were thawed (figure 19) before battered with four different formulations, which were produced with flour specific for fried battered products. The four batches corresponded to the use of either mineral water (groups named as “H₂O”) or ethylic alcohol (named as “ethanol”) for food use (National Distillery, Riachos, Portugal), and the incubation of the batter in a siphon with CO₂ (Isi- Consumer Products, Vienna, Austria) (groups named respectively as “without “CO₂“ and with “CO₂”).



Figure 19 – Thawed squid rings

Battered squid rings were deep-fried on sunflower oil at $190^{\circ} \pm 5^{\circ}\text{C}$ during 2 min and 30 seconds in a domestic fryer (Philips) (figure 20). Half of production was covered in a glass dish with transparent film at 4°C for 48h. After this time, the four different battered type of samples were reheated at $130^{\circ} \pm 5^{\circ}\text{C}$ during 15 min in a conventional domestic convection oven (UFESA).

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Figure 20 – Deep-frying of battered squid rings

Immediately after cooking (figure 21) crust and core were separated in order to proceed to some analysis to the crust, namely the physical and chemical analysis and instrumental texture analysis. Sensory analysis was made with the battered squid ring (crust and core squid). In both cases, temperature of crust or battered squid rings were continuously controlled with the intention of never being less than $45^{\circ} \pm 5^{\circ}\text{C}$.



Figure 21 – Fried battered squid rings

(**A** – H_2O ; **B** - $\text{H}_2\text{O} + \text{CO}_2$; **C** – ethanol; **D** – ethanol + CO_2)

Laboratorial Procedures

After battered squid rings production the following analysis were made by a specific order: moisture, color analysis, degree expansion, rancidity index (TBA) and fat content. Instrumental texture analysis and sensory analysis were also made just after frying process (day 0) or oven thermal reheating process (day 2).

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Moisture Content

Moisture was determined by keeping the sample in an oven at $103^{\circ}\pm 2^{\circ}\text{C}$ until reaching a constant weight (standard technique, official method NP-1614/1979 for meat and meat products). Five replicates were performed for each kind of formulated batter.

Color Analysis

Color measurements were made on each of four randomly selected battered squid rings from each formulation for each storage time (0 and 2 days). The Commission Internationale d’Eclairage (CIE) L^* , a^* , b^* , chroma and hue values were determined using a chroma meter model CR-300, Minolta Camera Company, (Osaka, Japan) that was calibrated with a standard white calibration plate (figure 22). The CR 300 chroma meter uses a xenon lamp as the light source. Five replicates were performed for each kind of battered squid ring.



Figure 22 – Minolta CR 300 chroma meter

Fat Content

After cooking (deep-frying or oven convection), crust was separated from core to analyze crust fat content. The total oil content of the crusts was determined by extraction with petroleum ether using a Soxtec Avanti 2050 extraction system (Foss Tecator, Sweden), previous hydrolysis was done with HCl and calculated as a percentage. Three replicates per sample were used.

Instrumental Analysis

A TA-HDi Texture Analyser (Stable Micro Systems, Godalming, UK) was used for the compression tests with a 25kg load cell, and the tests settings were the following mentioned. Kramer analysis was made just after cooking (deep-frying or oven convection) process, in order to assure crust temperature $45^{\circ} \pm 5^{\circ}\text{C}$.

The TA-HDi was used with the Kramer Shear Cell with 5 blades (HDP/KS5), with the following settings: test speed 2mm/s, trigger force 4N, travel distance of the blades 15mm and acquisition points per second 400pps. Ten replications with approximately 10g were performed for each test.

The following parameters were calculated from the force *versus* time curves: maximum force (N) (Sanz *et al*, 2007), respective work force or area (Ns), number of peaks of the curve or crispness or fracture events (drop in force higher than 0.049N), and initial slope (N/s) or gradient (slope of the curve up to the first major peak).

Figure 23 exemplifies the texture profile (force (N) *versus* time (sec) of the probe plots) in a fried battered sample at day 0 and at day 2.

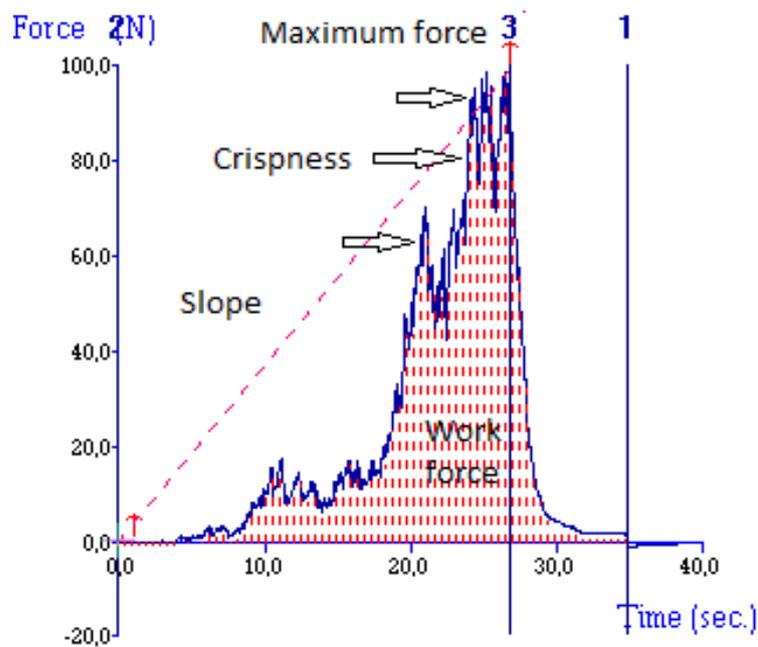


Figure 23 – Force – time curve of a Kramer test

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Sensory Analysis

A panel of 15 assessors with experience in the descriptive evaluation (NP ISO 8586-1, 2001) was used to evaluate the four samples of battered squid rings at production day and with 48h at reheated oven samples.

Panellists were the same that were trained during months for puffed snacks. But before definitive sessions a first group session were made in order to define descriptors to these kinds of products and to reach a consensus among panellists on the meaning of every attribute with the aim of training them to recognize specific texture descriptors (hardness, crispness, crunchiness). Testing was carried out in a sensory laboratory equipped with individual booths (NP ISO 8586-2, 2001).

The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9).

To evaluate hardness the instruction was to bite the whole sample with the incisor teeth until fracture and score the material resistance (Vincent, 2004). To score crispness the instruction was to evaluate altogether during mastication (first three bites), amount and quality of the sound produced (Chaunier *et al.*, 2005; Dijksterhuis *et al.*, 2007). To evaluate crunchiness the instruction was to score during mastication the number of layers with incorporated air (Dijksterhuis *et al.*, 2007). Oiliness was also assessed by the panellists (Prakash *et al.*, 1999), as well as toasted flavour intensity. Finally, panellists were asked to evaluate overall acceptability.

The samples were served in random order, on a plastic tray, identified with a letter random code (fig 24). Panellists were instructed to rinse their mouths with water between sample evaluations.

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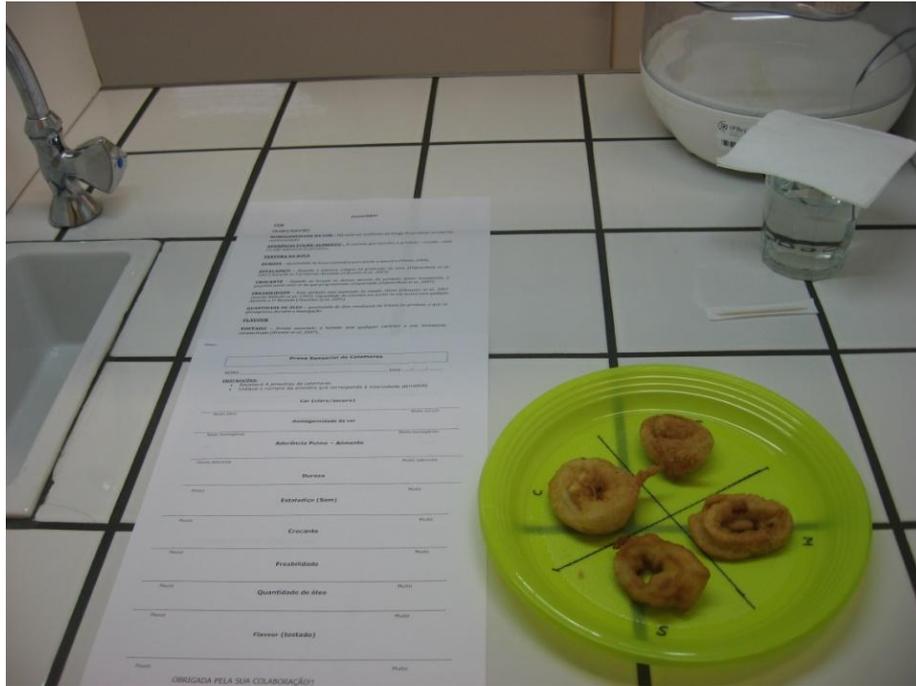


Figure 24 – Sensory analysis of Calamari

Statistical significant differences between values was evaluated at $p < 0.05$ level with a comparison test using SPSS 12.0.1 (SPSS Inc., Chicago, Illinois, US) using the Tukey pair-wise comparison to determine the differences between treatment means and it was also used Microsoft Excel 2002 (Microsoft Corporation, Sacramento, USA).

3. RESULTS AND DISCUSSION

Moisture content on fried battered samples (day 0) and on regenerated oven battered samples (day 2), after 48h in refrigeration, in samples processed with four different formulations (either with H₂O or with ethanol and with or without CO₂ incorporation) are shown on table 11.

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Table 11 - Moisture content on fried battered samples (day 0) and on regenerated oven (Day 2) battered samples processed with four different formulations (with H₂O or with ethanol with or without CO₂).

		H ₂ O	H ₂ O	Ethanol	Ethanol	SEM	P _{H₂O}	P _{CO₂}	P _{H₂O + CO₂}
		Without CO ₂	With CO ₂	Without CO ₂	With CO ₂				
Moisture Content (%)	Day 0	43.36 ^a	38.88 ^b	11.63 ^d	18.38 ^c	3.07	0.001	0.026	0.001
	Day 2	40.90 ^a	34.65 ^b	15.11 ^c	14.93 ^c	2.56	0.001	0.001	0.001
	P time	0.001	0.001	0.001	0.001				
Fat Content (%)	Day 0	12.87 ^b	18.06 ^b	31.72 ^a	31.97 ^a	2.11	0.001	0.151	0.159
	Day 2	16.28 ^d	25.80 ^c	29.44 ^b	36.33 ^a	1.61	0.001	0.001	0.011
	P time	0.150	0.001	0.010	0.001				

^{abcd} For the same row means without a common letter are significantly different

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Substitution of water with ethanol, CO₂ incorporation and their interaction significantly affected moisture content of batters. Samples produced with just water showed the highest moisture content (43.36% and 40.90%), while those elaborated with ethanol had shown much lower levels (11.63% and 18.38%, respectively, with no CO₂ and with CO₂ groups). CO₂ seemed to affect differently moisture content of batters made with water or with ethanol, since in the former the mean values were 43.36% for no CO₂ ones, and 38.88% for CO₂ ones, while in ethanol samples the trend was just the opposite (11.63% for no CO₂ and 18.38% for CO₂).

The same trend was shown for reheated samples after 2 days of refrigerated storage, with samples produced with water showing higher moisture contents (40.90% and 34.65%, respectively, with no CO₂ and with CO₂ groups) and with a different effect of CO₂ in water and ethanol batters (15.11% and 14.93%, %, respectively, with no CO₂ and with CO₂ groups), although in this case, there were no significant differences between ethanol samples as a consequence of CO₂.

All four samples had significant differences from day 0 to day 2, in particular, through an increase in moisture content all along time, in ethanol batter without CO₂, from 11.63% to 15.11%, respectively, at day 0 and day 2, and by a decrease in moisture content in water samples with or without CO₂, and also in ethanol batters with CO₂.

In fact, during refrigeration the water content in foodstuff trends to migrate to the environment in order to equalize vapour water tension, which might explain the similar decrease (~4.0 %) in those samples moisture content. Furthermore, ethanol formulations with or without CO₂ had shown lower moisture content than water formulations, which probably is due to the fact that ethanol formulations water content was only conferred by the flour moisture content (11.25%) used on batter preparation, in part by water present in ethanol, and yet from some ethanol remaining in the batter, and nonetheless from the squid itself, since the values were, respectively, 11.63% without CO₂ and 18.38% with CO₂.

As far as the effect of storage and reheating, different hydration during storage could affect ethanol samples differently, regardless the CO₂ content. The presence of CO₂ on ethanol samples influenced the different thickness of the coating, and therefore, as result of more bubbles, it had difficult water evaporation during frying process, due

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to more layers. In fact, alcohol, promotes gluten rapid dissolution, and also reduces water amount absorbed by starches granules flour components, and then during frying, alcohol evaporation precedes water evaporation, and consequently, after frying, the coater dries quickly and turn out to be more crispy, avoiding the squid rings to loss water content, and, hence, during overheating, it will protect proteins muscle from shrinkage, and thus from increasing core (squid) toughness. Most of all, this coater remains crispy after reheated on oven.

Related to fat content, table 11 had shown that at day zero (day 0) neither water formulations samples nor did ethanol formulations samples have significant differences between each other. In addition, in specific for water formulations samples, besides p value correlated to water factor were less than 0.05, the other p values (p_{CO_2} and $p_{H_2O + CO_2}$) had more influence over p_{H_2O} ($p=0,001$), and therefore, in the end there were no significant differences on fat content between water formulations samples, but still the water samples with CO_2 had a higher value (18.06%) when compared with water samples without CO_2 (12.87%).

Fat content on regenerated samples (day 2) presented significant differences between all four different batter formulations. Moreover, ethanol formulations samples in addition to significant differences between each other had shown higher fat content values (36.33% and 29.44% on ethanol formulations samples with and without CO_2 , respectively). And water formulations samples, had presented the lowest fat content values 25.80% and 16.28% on water formulations with and without CO_2 , respectively. These facts were in agreement with moisture content data, since a decrease or increase on water content were expected to be achieved in parallel with an increase or decrease, respectively, on fat content.

Undeniably, deep-fat frying process involves heat transfer by convection from the surrounding oil to the surface of the product and then heat conduction into the interior of the piece (core). Also, mass transfer occurs due to release of water and by oil uptake. After immersion into the hot oil, the temperature of the surface layers rises rapidly; water starts to boil at $100^\circ C$ and is released from the surface as bubbles. As frying proceeds, the thickness of the developed crust continues to increase and the number of steam bubbles is reduced (Miranda *et al.*, 2006).

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In relation to formulations after 48h refrigeration and regenerated on oven (day 2), water samples without CO₂ were the only ones that had not presented significant differences along time storage, and had the lowest fat content (16.28%). On ethanol formulation samples without CO₂ or without CO₂ and on water samples with CO₂ there were significant.

On the other hand, fat content had the same trend as water content, since on water formulations and on ethanol batter with CO₂ there were an increase on fat content, which was in accord with moisture content evolution observed on these three formulation samples: all of them had a decrease on moisture content during storage. On the opposite, ethanol batter without CO₂ had a decrease on fat content.

In resume, apart from water migration during cooking, during deep-fat frying, crust formation also depends on other physical phenomena. Physical processes such as oil migration from the frying oil to the product or fat from the product to the outside also influence plasticization and are interrelated with moisture loss (Varela *et al.*, 2008), which facts are in consonance to associated moisture/fat contents that were obtained in this study.

Force *versus* time Kramer parameters are shown on table 12, more precise the maximum force (N), area (Ns), number of peaks of the curve (fracture events or crispness) and initial slope on fried battered samples (day 0) and on regenerated oven (day 2) battered samples, after 48h in refrigeration, and all these samples were processed with four different formulations (with H₂O or with ethanol with or without CO₂).

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Table 12 - Parameters from the force *versus* time Kramer curves: maximum force (N), area (Ns), number of peaks of the curve (crispness) and initial slope on fried battered samples (day 0) and on regenerated oven (day 2) battered samples processed with four different formulations (with H₂O or with ethanol with or without CO₂).

		H ₂ O	H ₂ O	Ethanol	Ethanol	SEM	P _{H2O}	P _{CO2}	P _{H2O + CO2}
		Without CO ₂	With CO ₂	Without CO ₂	With CO ₂				
Maximum Force (N)	Day 0	1.97 ^a	1.54 ^b	1.35 ^b	1.44 ^b	0.06	0.020	0.041	0.022
	Day 2	1.93 ^a	1.44 ^b	1.42 ^b	1.27 ^b	0.06	0.001	0.001	0.081
	P time	0.827	0.394	0.885	0.108				
Work Force (Ns)	Day 0	10.01 ^a	7.28 ^b	8.50 ^{ab}	8.63 ^{ab}	0.33	0.898	0.035	0.048
	Day 2	10.83 ^a	6.66 ^b	10.46 ^a	9.42 ^a	0.37	0.031	0.001	0.005
	P time	0.269	0.311	0.110	0.326				
Crispness (number of peaks +)	Day 0	100.20 ^b	114.40 ^b	243.30 ^a	220.40 ^a	11.53	0.001	0.877	0.392
	Day 2	80.60 ^b	44.08 ^c	217.20 ^a	247.30 ^a	14.11	0.001	0.707	0.001
	P time	0.148	0.001	0.524	0.196				
Slope (N/s)	Day 0	10.51 ^a	8.24 ^{ab}	6.17 ^b	7.35 ^b	0.46	0.001	0.335	0.026
	Day 2	12.27 ^a	6.94 ^b	6.45 ^b	6.05 ^b	0.46	0.001	0.001	0.001
	P time	0.348	0.148	0.784	0.110				

^{abcd} For the same row means without a common letter are significantly different

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Maximum force and its work (area) had shown significant differences only in water formulations samples with and without CO₂ in both days (day 0 and day 2). In fact, at day 0, the higher maximum force (1.97 N) and work force (10.01 N.s) were observed on water samples without CO₂, and at day 2, it was also those samples that presented the highest maximum force (1.93 N) and the work force (10.83 N.s).

Related to factor time, neither of water or ethanol battered samples with or without CO₂, had shown significant differences in these force parameters. In general, ethanol samples had shown a decrease in maximum force and an increase in work force during storage, and water samples without CO₂ had the same trend, however water samples with CO₂, had a decrease in maximum force, but it had also a decrease in work force. In fact, usually, a brittle product will exhibit a large hardness, low work and a sudden drop in the force as the crack propagates rapidly (Miranda *et al*, 2006).

Slope was also expected to had a behavior similar to the above mentioned force parameters, since this slope or gradient is established up to the maximum force, and indeed, only water samples with and without CO₂ at day 2 had shown significant differences, with the highest value (12.27 N/s) on water sample without CO₂, as well as it had presented the highest work force value (10.83 N.s) among all other samples, and the lowest slope value (6.94 N/s) was presented in water samples with CO₂ at day 2, as well as the lowest work force value (6.66 N.s). Ethanol batters had no significant differences in both days, and even during storage, and the lowest slope values.

In crispness parameter, despite ethanol samples had not shown significant differences, these samples had presented the highest values in both days, when related to water samples. More precise, ethanol sample without CO₂ had shown the higher value (243.30) in day 0, followed by ethanol sample with CO₂, (220.40), and finally water samples had the lowest crispness values (100.20 and 114.40, respectively without and with CO₂, groups), and these last ones had significant differences with ethanol samples, but not with each other at day 0. After storage, none of ethanol samples and also water samples without CO₂ had shown significant differences from day 0 to day 2, only water samples with CO₂, presented significant differences during storage time, with the lowest crispness value (44.08). During storage stage, both water samples, and ethanol samples without CO₂ had the same trend to decrease crispness, but ethanol

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samples with CO₂ crispness increased and had the highest value (247.30) of all samples in both days. Water samples with and without CO₂ only had significant differences at day 2, with the lowest values of all four samples in both days, and more exact with 44.08 on water samples with CO₂ and 80.6 on water samples without CO₂ at day 2. SEM values on this parameter were extremely high in both days.

Indeed, since during frying, alcohol evaporation precedes water evaporation, and ethanol batters with CO₂, will have more bubbles, thus, after frying, the coater dries quickly, becomes less thickener and more crispy, even after reheated on oven.

An important quality factor in fried battered products is the retention of crispness for some time after frying (Bauxilli *et al.*, 2003), and this texture parameter was strongly and negatively influenced by moisture content, in particular, on water samples, which had determined its elastic nature observed during instrumental texture analysis, and that it was well seen on force-time Kramer data. In fact, a loss of brittleness is detected in instrumental testing as a change in the pattern and in the value of parameters derived from the force-deformation curve, and, as water activity is increased, a shift from brittle fracture to plastic flow and ductile fracture is observed (Miranda *et al.*, 2006).

In general, other studies refer preferably puncture methodologies for instrumental texture evaluation of fried battered or non battered foods (Miranda *et al.*, 2006), rather than Kramer shear compression, but even in both tests the force-deformation (or force-distance) curves are widely used as objective measurements of textural properties of foods.

Color parameters from Lab CIE – L*, a*, b*, Chroma and Hue are shown on table 13, for fried battered samples (day 0) and regenerated oven (day 2) battered samples, after 48h in refrigeration, and all these samples were processed with four different formulations (with H₂O or with ethanol with or without CO₂).

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Table 13 – L*, a*, b*, Chroma and Hue color parameters from Lab CIE on fried battered samples (day 0) and on regenerated oven (day 2) battered samples processed with four different formulations (with H₂O or with ethanol with or without CO₂).

		H ₂ O Without CO ₂	H ₂ O With CO ₂	Ethanol Without CO ₂	Ethanol With CO ₂	SEM	P _{H₂O}	P _{CO₂}	P _{H₂O + CO₂}
L*	Day 0	66.88	57.34	57.83	57.06	1.02	0.018	0.007	0.021
	Day 2	60.85	57.08	52.44	55.18	0.90	0.001	0.685	0.018
	P time	0.136	0.789	0,019	0.147				
a*	Day 0	-2.75	-1.77	-0.36	-0.79	0.25	0.004	0.613	0.435
	Day 2	-1.23	-0.98	1.04	-0.24	0.27	0.001	0.207	0.067
	P time	0.074	0.185	0.061	0.418				
b*	Day 0	22.68	23.54	20.34	19.31	0.58	0.007	0.468	0.603
	Day 2	21.62	23.65	17.95	19.73	0.67	0.002	0.076	0.904
	P time	0.545	0.952	0.206	0.923				
Chroma	Day 0	0.34	0.41	0.35	0.34	0.01	0.072	0.045	0.122
	Day 2	0.36	0.41	0.34	0.36	0.01	0.055	0,046	0.202
	P time	0.417	0.932	0.822	0.765				
Hue	Day 0	-0.12	-0.08	-0.02	-0.04	0.01	0.011	0.665	0.358
	Day 2	-0.06	-0.04	0.06	-0.02	0.01	0.004	0.200	0.054
	P time	0.109	0.183	0.076	0.405				

^{abcd} For the same row means without a common letter are significantly different

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Related to L^* , although $p < 0.05$ in all four different sample formulations, there weren't detect significant differences between means, probably due to the fact that Tukey test wasn't enough to perceive it, even so on water sample formulation without CO_2 it was noticed the highest value (66.88) and in the other three samples the lowest values, in specific on ethanol sample with CO_2 (57.06). In relation to time factor, only ethanol sample without CO_2 had significant differences in this color parameter, with a decrease from 57.83 on day 0 to 52.44 on day 2, revealing the lowest value on day 2, yet water sample without CO_2 had still the higher value (60.85) on day 2. On day 2, even though both $p_{\text{H}_2\text{O}}$ and $p_{\text{H}_2\text{O} + \text{CO}_2} < 0.05$ it seemed that the fact that $p_{\text{CO}_2} > 0.05$ had a greater influence over H_2O factor and even $\text{H}_2\text{O} + \text{CO}_2$ factor, and thus Tukey test wasn't enough to distinguish significant differences on L^* means in all four samples. In summing up, water samples formulations without CO_2 in both days were the brightness ones.

In other study with battered squid rings (Salvador *et al.*, 2005), color parameter L^* showed comparable values with water battered sample without CO_2 incorporation, more precise $L^* = 67.0$ after a heat impact treatment (microwave oven), but those samples were then frozen and a week later fried at 190°C , thus presenting a decrease on L^* values, that were more similar to ethanol samples and even water samples with CO_2 , particularly after oven reheating process. However, other study (Sanz *et al.*, 2004) had shown lower L^* values in fried battered squid rings (50.19), but still higher a^* and b^* values.

Color parameters a^* and b^* showed $p_{\text{H}_2\text{O}} < 0.05$ in both days (Day 0 and day 2), but even so it wasn't enough for Tukey test to sense significant differences. In these parameters, the other factors $p_{\text{H}_2\text{O} + \text{CO}_2}$ and $p_{\text{CO}_2} > 0.05$, and hence all samples hadn't significant differences in both days. Nonetheless, ethanol sample formulation without CO_2 at day 2 was the redness sample (1.04), and water sample with CO_2 was the yellowness (23.65). In relation to time factor, only ethanol batter without CO_2 L^* value had significant differences from day 0 to day 2, because all the other samples hadn't significant differences in neither color parameter.

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In ending these color parameters, it was expected that redness and yellowness were higher in samples at day 2, since two thermal processes (frying and oven) had higher influence in color than just one single thermal process (frying).

In the previous mentioned study with battered squid rings (Salvador *et al.*, 2005), color parameters a^* and b^* in final fried product were higher than all four final battered samples in this study, only comparable with the pre-heated battered squid rings used in that study, which had a^* values negatives (-1.00) and similar b^* values (18.6), because after fried process those same samples had a^* value higher (6.8) and b^* value even higher (35.9).

The other two color parameters – chroma and hue – hadn't significant differences, although some p values less than 0.05, but still those few values, mostly on pH_2O values wasn't enough to be detected by Tukey test. The highest chroma values (0.41) were noticed on water samples with CO_2 in both days and the highest hue value (0.06) was detected on ethanol samples without CO_2 .

In fact, these parameters couldn't present significant differences since these color characteristics derived from $L^*a^*b^*$ parameters, which hadn't shown significant differences.

Sensory parameters related to appearance (color white or dark and color homogeneous) and texture, namely adherence batter-food, hardness, crispy (sound) and crunchiness both on fried battered samples (day 0) and on regenerated oven (day 2) battered samples, after 48h in refrigeration are presented in table 14, and all these samples were processed with four different formulations (with H_2O or with ethanol with or without CO_2).

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Table 14 – Sensory parameters of appearance and texture on fried battered samples (day 0) and on regenerated oven (day 2) battered samples processed with four different formulations (with H₂O or with ethanol with or without CO₂).

		H ₂ O Without CO ₂	H ₂ O With CO ₂	Ethanol Without CO ₂	Ethanol With CO ₂	SEM	P _{H2O}	P _{CO2}	P _{H2O + CO2}
Color (White/Dark)	Day zero	4.14	3.72	5.17	5.19	0.30	0.051	0.727	0.694
	Day 2	4.54	4.84	5.66	5.04	0.21	0.142	0.703	0.293
	P time	0.663	0.232	0.454	0.691				
Color Homogeneous	Day zero	5.47 ^{ab}	6.31 ^a	5.22 ^{ab}	4.78 ^b	0.20	0.008	0.451	0.035
	Day 2	5.83	5.51	4.82	5.12	0.20	0.109	0.977	0.446
	P time	0.597	0.154	0.333	0.384				
Adherence Batter-Food	Day zero	4.83	4.33	4.85	4.61	0.13	0.574	0.187	0.627
	Day 2	3.70 ^b	4.32 ^{ab}	4.85 ^a	5.03 ^a	0.18	0.001	0.068	0.287
	P time	0.007	0.982	0.986	0.178				
Hardness	Day zero	3.44 ^b	3.92 ^b	5.41 ^a	5.28 ^a	0.27	0.001	0.461	0.219
	Day 2	3.75 ^b	4.32 ^{ab}	5.42 ^a	5.41 ^a	0.25	0.002	0.405	0.390
	P time	0.563	0.381	0.987	0.414				
Crispy (Sound)	Day zero	3.06 ^b	4.12 ^b	7.05 ^a	6.78 ^a	0.53	0.001	0.181	0.039
	Day 2	2.71 ^b	2.97 ^b	5.75 ^a	5.35 ^a	0.43	0.001	0.805	0.276
	P time	0.401	0.062	0.049	0.005				
Crunchiness	Day zero	3.00 ^c	4.59 ^b	6.66 ^a	6.83 ^a	0.49	0.001	0.007	0.019
	Day 2	2.79 ^b	3.03 ^b	5.64 ^a	5.23 ^a	0.41	0.001	0.812	0.374
	P time	0.667	0.025	0.098	0.003				

The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9); ^{abcd} For the same row means without a common letter are significantly different

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In color, there were no significant differences between all four battered samples, even after regeneration on oven (day 2). Water samples formulations were noticed to be the whiteness samples, specifically water sample formulation without CO₂ at both days, which scored 4.14 at day 0 and 4.54 at day 2. Undeniably this sensory parameter was expected to be in consonance with L* parameter (table 3) which had shown water samples formulations without CO₂ in both days the brightness ones. It was also made known that ethanol samples with and without CO₂, were the darkness color formulations with 5.17 score at day 0 and at day 2 scored 5.66, the highest value in all samples. Considering the color homogeneity, at day 0 water samples formulations with CO₂, scored 6.31, the highest value in all samples, had significant differences with ethanol samples formulations with CO₂ (4.78). At day 0, all other battered samples hadn't significant differences between each other. At day 2, all four samples hadn't shown significant differences, yet the water samples were those that had higher scored values, mainly without CO₂, with 5.83 and ethanol samples had lower scored values, specifically without CO₂, with 4.82.

The sensory parameter related to the adherence of batter to food had presented significant differences at day 2, mostly in water formulation without CO₂, with the lowest score (3.70) of all four battered samples, even in both days as it was supported by time factor ($p < 0.05$). Besides the other samples had not shown significant differences in both days, the highest scored value (5.03) was noticed on ethanol samples with CO₂ at day 2, since oven thermal process seemed to contribute to an increase on this appearance sensory parameter at least in this battered formulation (ethanol plus CO₂), and despite the constant values in both days on water formulation with CO₂, and on ethanol formulation without CO₂, this thermal process at day 2 wasn't enough to at least maintain the batter adherence, moreover contributed to decrease this appearance parameter.

Definitely, during immersion frying process, a moving boundary is produced within the fried product separating the forming crust from the core that is being cooked (Singh, 2000; Tangduangdee *et al.*, 2003). As dehydrated crust is formed the temperature of the outer surface reaches equilibrium with oil temperature, and a steep temperature gradient sets in through the crust into the movingcrust/core. Heat transfer through the crust is dissipated by further heat conduction into the interior of the fried

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product, eventually cooking it into a mealy core (Miranda *et al.*, 2006). So, this phenomenon would have influenced adherence of fried batter to squid core in this study, which might had been decisive to distinguish the higher scored values in this sensory parameter in both ethanol battered samples (with less moisture content) with the lower scored values in both water batter samples (with higher moisture content).

Related to sensory hardness, this parameter had presented significant differences between ethanol formulations and water formulations, since these last ones had shown significant lower scored values, mostly water formula without CO₂ with a score of only 3.44 compared to the ethanol formula without CO₂, with the highest score (5.41) after frying process. Notice that in this day, either water formulas or ethanol formulas hadn't shown significant differences with CO₂ incorporation. At day 2, simply water formula batter without CO₂ had shown significant differences with the ethanol batter samples, with a score of 3.75, the lowest after oven thermal process. Effectively, it had given the impression that this formula had a constant low sensory hardness even after oven thermal process; however CO₂ incorporation had some contribute on increasing hardness in these water batter formulas.

When sensory hardness was associated to textural parameters, namely maximum force, respective work force and slope, this water batter without CO₂ incorporation had significant differences with the other formulas, and furthermore it had the highest values in these three texture parameters, which fact was confirmed by panellists, whom definitely establish this samples as the least crispy and crunchy in the face of the most spongy and elastic.

At both days, sensory crispness had significant differences between water batters and ethanol batters; however either in water formulas or in ethanol formulas, CO₂ incorporation hadn't shown significant differences. In this parameter, the highest values were scored on ethanol samples (7.05 without CO₂ and 6.78 with CO₂) and the lowest scores were detected on water samples, mostly without CO₂ with 3.06 at day 0 and 2.71 at day 2. It was also noticed that on ethanol battered samples, crispness had significant differences from day 0 to day 2 ($p < 0.05$), with a significant decrease in scored values, above all on ethanol batter with CO₂, which had merely scored 5.35. However, on water battered samples with CO₂ incorporation, time factor ($0.1 > p > 0.05$) revealed a trend in

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spite of not showing significant differences, but nonetheless at day 0 this samples scored 4.12 and after oven thermal process it had scored 2.97.

In comparing these sensory crispy values with texture crispness (table 12), water samples had significant differences with ethanol samples, presenting the lowest crispness values, and in addition at day 2 the water samples with CO₂ incorporation had significant differences when correlated with those same produced by frying thermal process (day 0).

In point of fact, these water battered formulas were the less crispy (in both sensory and textural analysis) since the prime wheat nature of flour used in batters, which gluten was predominantly dissolved on water than on ethanol, and hence contributed to its elastic network on water samples than on ethanol samples. Therefore, these facts were resolutely to distinguish a notorious sensory and textural crispness on ethanol battered formulas and an astonishing lack of sensory and textural crispy attribute on water battered formulas.

On table 14, sensory crunchiness values had presented more heterogeneity in view of the fact that at day 0 water samples had significant differences with ethanol samples, but even because water samples with and without CO₂ incorporation had significant differences between each other. So, the higher scored values were on ethanol battered samples, mainly with CO₂ incorporation which scored 6.83, and this samples had no significant differences with ethanol samples without CO₂ incorporation which scored 6.66. On water samples, those produced with CO₂ incorporation had the higher scored value with 4.59 and samples without CO₂ incorporation had shown significant differences, with the lowest score of 3.00.

Another clearly fact was the significant differences on water and ethanol samples with CO₂ incorporation from day 0 to day 2, which had shown a decrease in crunchiness from day 0 to day 2, as well as it was noticed in crispness, even with the increase in hardness. So, after oven thermal process at day 2, water samples presented the lowest values and ethanol samples the highest values. However CO₂ incorporation seemed to had influenced only water samples at day 0 ($p_{CO_2} < 0.05$), probably due to a more aggressive thermal process (frying), which might had promoted CO₂ diffusion and effectively pushed and puffed wheat gluten matrix contributing to a higher scored value

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on water battered samples with CO₂ (4.59) than without CO₂ (3.00). This CO₂ diffusion theory on ethanol samples hadn't the same effect than on water samples since ethanol diffusion also occurred in parallel and thus justified the fact that both ethanol samples had similar crunchy values.

Other sensory parameters are presented in table 15, namely friable coater, oily character, flavour and overall acceptability on fried battered samples (day 0) and on regenerated oven (day 2) battered samples, after 48h in refrigeration, and all these samples were processed with four different formulations (with water or with ethanol with or without CO₂). In the following table, it was possible to observe that at day 0 the sensory parameter friable coater had shown significant differences between ethanol samples and water samples; and in addition water samples with and without CO₂ also had significant differences. Ethanol samples had presented the highest values, above all in samples with CO₂ incorporation, which scored 5.97, followed by water samples with CO₂ incorporation, with a score of 3.60 and then the lowest scored value (2.72) on water samples without CO₂. After regeneration on oven, ethanol samples with and without CO₂ incorporation, which scored higher values (4.78 with CO₂ and 4.56 without CO₂) had significant differences with water samples with and without CO₂, that had the lowest scored values (2.50 with CO₂ and 2.81 without CO₂). In addition, and related to time factor both water and ethanol samples with CO₂ incorporation had significant differences, both had shown a decrease in scored values, and the lower friable coater values were presented on water samples. However, on ethanol battered samples without CO₂ incorporation, time factor ($0.1 > p > 0.05$) revealed a trend to significant differences, with a score of 5.84 at day 0 and after oven thermal process it had scored 4.78.

Yet concerning to the sensory parameter friable coater, it seemed important to become aware of two evidences. The first was the detail that to panellists were told to associate friable to glassy state, and so a more friable coater would have more tiny dry pieces (like glass fragments) during bolus formation in mouth, that easily would be swallowed, and so those should be known as more friable coaters. Otherwise, it should be considered to be a less friable coater, and that was associated to a moister coater which would be more difficult to swallow.

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Table 15 – Sensory parameters and overall acceptability of fried battered samples (day 0) and on regenerated oven (day 2) battered samples processed with four different formulations (with H₂O or with ethanol with or without CO₂).

		H ₂ O	H ₂ O	Ethanol	Ethanol	SEM	P _{H₂O}	P _{CO₂}	P _{H₂O + CO₂}
		Without CO ₂	With CO ₂	Without CO ₂	With CO ₂				
Friable Coater	Day zero	2.72 ^c	3.60 ^b	5.84 ^a	5.97 ^a	0.43	0.001	0.007	0.029
	Day 2	2.50 ^b	2.81 ^b	4.78 ^a	4.56 ^a	0.33	0.001	0.891	0.414
	P time	0.587	0.024	0.067	0.009				
Oily	Day zero	4.47 ^b	5.76 ^a	5.68 ^a	5.97 ^a	0.19	0.001	0.001	0.008
	Day 2	5.11	5.66	5.54	5.70	0.11	0.242	0.094	0.339
	P time	0.012	0.692	0.645	0.322				
Flavour	Day zero	3.33 ^b	3.81 ^{ab}	5.75 ^a	5.72 ^a	0.39	0.002	0.661	0.622
	Day 2	3.57 ^b	4.10 ^{ab}	5.47 ^a	5.23 ^a	0.27	0.002	0.675	0.270
	P time	0.736	0.743	0.482	0.275				
Overall Acceptability	Day zero	2.92 ^b	4.60 ^a	6.00 ^a	5.67 ^a	0.39	0.001	0.001	0.011
	Day 2	3.04 ^b	3.14 ^b	4.99 ^a	5.01 ^a	0.31	0.001	0.815	0.875
	P time	0.661	0.071	0.036	0.113				

The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9)

^{abcd} For the same row means without a common letter are significantly different

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The other piece of information was related to the fact that the four battered samples moisture content (table 11) probably would influence this sensory parameter. As a result of the samples moisture content, the most friable coaters were ethanol battered samples and in fact those had the lowest moisture contents, and water battered samples without CO₂ incorporation had the highest moisture content (43.36%) and the lowest friable coater score (2.72).

After regeneration on oven process, all four samples had shown a decrease on sensory friability, which fact didn't revealed astonishing since the eventual initial glassy state conferred by frying process was unsure due to the natural lost of glassy state during the cooling phase that preceded the sensory analysis. Moreover that cause, the initial moisture content would also contribute to the speed lost of glassy state and in parallel to definition of rubbery state. While ethanol battered samples during refrigeration had a lack of glassy state, after oven thermal process these samples recovered some glassy state, water battered samples didn't had in frying thermal process a defined glassy state and thus had never recovered from rubbery sate after oven thermal process.

Related to sensory oily or fatty sensation, it was observed in table 15 that water battered samples without CO₂ incorporation had significant differences from all other three battered samples, and its scored value was the lowest of all with 4.47 after frying (day 0), and thus the other samples, namely ethanol battered samples had higher oily scored values.

At day 2, none of four battered samples had presented significant differences between each other, and the most oily was ethanol battered sample with CO₂ incorporation (5.70) and the least oily was water battered sample without CO₂ incorporation (5.11), which fact together with time factor ($p < 0.05$), exposed that this water samples without CO₂ incorporation had shown significant differences from frying (day 0) to oven process (day 2).

Indeed, ethanol battered samples were those that presented higher fat contents (Table 11), while water battered samples had shown in table 1 the lowest fat contents, mostly water sample without CO₂ incorporation with 12.87% fat content at day 0 and 16.28% at day 2. Evidently, in frying kinetics it was normal that a matrix with more

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water absorbed less oil, like it had happened to water battered samples without CO₂ that had 43.36% moisture content, only had scored 4.47 in oily appearance whereas ethanol battered sample with CO₂ incorporation with 18.38% moisture content and 31.97% fat content, had scored 5.97 in oily attribute.

Concerning to flavour it was observed in table 15 that at both days, water battered samples without CO₂ incorporation had significant differences only with both ethanol battered samples, and either these ethanol samples or water samples didn't show significant differences among each other. In both days, ethanol samples scored the highest values, specifically after frying (day 0), and water samples scored the lower values, to be exact the formula without CO₂ incorporation had the lowest score of 3.33 after frying (day 0). It was curious that water samples flavour increased after oven thermal process, and ethanol samples flavour decreased after the same thermal procedure, which was probably due to the fact that frying (at 185-190 °C) enhanced more potentially Maillard reactions than oven process (130-135 °C), but even so on water samples it was possibly the decrease on moisture content what might had contributed to enhanced a few more flavour at day 2 when related with day 0.

In fact, the rules of physical chemistry indicate that there will be an increase in volatile components being released from a sample as it is heated (Delwiche, 2004; Atkins *et al.*, 2002). It is thought that as a result, odours become more intense as a given sample is heated, and a possible consequence of such a phenomenon would be that a sample might contain volatile compounds that are below threshold levels at lower temperatures, but that are detectable as the sample is warmed (Delwiche, 2004). Given this phenomenon, it was surprising the significant differences that water samples without CO₂ incorporation had with the other three samples, since all samples were given to the panellists at the same time and temperature.

However, some studies results indicate that individuals associate certain flavours (and odours) with specific colours and when the colours are altered, the flavour/odour identification is decreased; the stronger the colour-flavour/odour association, the greater the impact of colour (Delwiche, 2004).

Finally, regarding overall acceptability it was noticed that water battered samples without CO₂ incorporation had significant differences with all three other

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battered samples, with the lowest scored value (2.92), and followed by water battered sample with CO₂ incorporation (4.60) and then ethanol battered samples had the highest scored values. Although $p_{CO_2} < 0.05$, water battered samples had shown significant differences due to CO₂ incorporation, but on ethanol battered samples it seemed that Tukey test wasn't enough to detect significant differences ($p = 0.011$).

Related to time factor, it was obvious that ethanol battered samples without CO₂ incorporation had shown significant differences from day 0 to day 2, what was probably due to the synergy occurred during frying process and CO₂ incorporation. Water battered samples with CO₂ incorporation had shown a trend to significant differences from day 0 to day 2, since $0.1 > p > 0.05$, with oven samples had scored 3.14 and frying samples with a score of 4.60.

4. CONCLUSIONS

Substitution of water with ethanol in batters promoted significant changes in water and fat content; contributing to a decrease on water content, and thus after frying, the coater dries quickly and becomes crispy. On the other hand, water content increase led to a fat content increase. This ethanol coater remains crispy after reheated on oven.

CO₂ incorporation in water and ethanol batters had been responsible for more bubbles, and therefore, for a coater less thickener and more crispy

Instrumental crispness and hardness, were significant different on water batters and on ethanol batters; and thus water batters were harder, however, ethanol batters, were crispier and during storage, CO₂ incorporation led to an increased on ethanol batter crispness.

Coater color was not affected on either water or in ethanol batters by CO₂ incorporation, and all coaters were golden, more or less bright, even after reheating process.

Ethanol coaters had higher adherence, after frying or oven reheating, and CO₂ incorporation influenced water batter adherence, mostly after reheating process.

Sensory hardness was well correlated with textural parameters, since water batter without CO₂ was harder in both instrumental and sensory analysis.

Ethanol coaters were crispy and crunchy, not revealing by panellists CO₂ influence like it hadn't on instrumental crispness, but this component had determine on water coaters, after frying, an improvement on crunchiness, however less than on either ethanol coaters.

Ethanol coaters, after frying or reheating, had a greater overall than water coaters, however in this last ones, CO₂ incorporation influenced positively panellists' acceptability, mostly after frying.

CHAPTER IV – “FREEZE-DRIED AND DEHYDRATED
YOGURT FOAMS”

(ESPUMAS DE YOGUR LIOFILIZADAS Y DESHYDRATADAS)

Abstract

As a rule, freeze drying produces the highest quality food product obtainable by any drying method. A prominent factor is the structural rigidity afforded by the frozen substance at the surface where sublimation occurs, which prevents collapse of the solid matrix remaining after drying and, thus, the result is a porous and nonshrunken structure. The shape of the pores, the pore size distribution, and pore connectivity of the porous network of the dried layer formed by the sublimation of the frozen water during the primary drying stage depend on the ice crystals that formed during the freezing stage, which fact will influenced significantly the porous structure of the dried layer. The formula to prepare the whipped foam to freeze-drying or drying processes included yogurt, water, salt, isomalt, maltodextrin, metilcellulose and xanthan gum. For the freeze-drying process, this foam was quickly frozen at -80°C for 24h, and after this period at -50°C and under vacuum conditions in a freeze-dryer. In parallel, whipped foam was placed on the dryer at 57°C for 24h. After freeze-drying and drying processes, to both freeze-dried and dried samples were made the following analyses: moisture, water activity (a_w), weight loss, colour analysis, instrumental texture analysis (compression test), sensorial analysis and image analysis (SEM). Water activity had showed significant differences with higher value on dried foams than on freeze-dried foams. Freeze-dried foams had a significant lower maximum force (hardness) than dried foams. The count of the number of peaks of the curve, which meant the crispness, it was noticed the highest value on dried foams than on freeze-dried foams, since these foams had a higher number of fracture events. The more lightness foams were freeze-dried samples and dried foams were the more yellowness. SEM images evidences that on the freeze-died foams weren't any sign of collapse and on dried foams an enormous collapse in all structure. Sensory results indicated that a certain degree of hardness is necessary for crispness perception, as those values had a similar behaviour, and were higher on dried foams than on freeze-dried foams, as well as it was overall acceptability.

Keywords: Freeze-drying, Drying, Hydrocolloids, Scanning Electron Microscopy

1. INTRODUCTION

After the Second World War, freeze-drying applications in the food industry started and many important developments took place in the 1950s and 1960s (Niranjan 2002). During the 1960s it was expected that the process would have wide application and considerable resources were devoted to developing novel equipment and methods, but in the USA and Europe, highly efficient distribution and storage systems for frozen and chilled food were firmly established, restricting the market for freeze-dried products (Snowman, 1997). Freeze drying is a dehydration process where water vapor is removed by sublimation from frozen materials, usually under conditions of low pressure and temperature (Khalloufi *et al*, 2004). The advantage of this process is mainly related to the high final quality of products (Khalloufi *et al*, 2004; Sagara *et al*, 1994; Krokida *et al*, 1997; Sablani *et al*, 2007).

Water is a small and dynamic molecule that has a heterogeneous spatial distribution within foods, and that exhibits significant variations in properties and reactivity depending on location. At a microstructural level, where colloidal phenomena predominate, the role of water is critical in the formation of droplets (e.g., emulsion), crystals (e.g., ice formation), air cells (e.g., foams), etc. (Aguilera *et al*, 1999; Vittadini, 2007).

In freeze-drying, water, representing more than 80% of food products, is frozen and therefore cannot serve as a solvent reactant throughout the sublimation process (Khalloufi *et al*, 2004). As the solvent (ice) sublimates, the sublimation interface (plane of sublimation), which started at the outside surface recedes, and a porous shell of dried material remains. The vaporized solvent (water) vapor is transported through the porous layer of dried material. During the primary drying stage, some of the sorbed water (nonfrozen water) in the dried layer may be desorbed. The time at which there is no more frozen layer (that is, there is no more sublimation interface) is taken to represent the end of the primary drying stage. The secondary drying stage involves the removal of solvent (water) that did not freeze (this is termed sorbed or bound water). The secondary drying stage starts at the end of the primary drying stage, and the desorbed water vapor is transported through the pores of the material that is dried. In any food material, some nonfrozen water will almost unavoidably be present during freeze drying, but there is very often a rather sharp transition temperature (T_g) for the still wet region during

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drying below which the product quality improves markedly. This improvement shows that sufficient water is frozen to give the beneficial product characteristics of freeze drying (Liapis *et al.*, 2002). The glass transition temperature (T_g) is a very important physical parameter, and it is defined as the temperature at which the material changes from the glassy to the rubbery state for a given heating rate (Perdomo *et al.*, 2009).

As a rule, freeze drying produces the highest quality food product obtainable by any drying method. A prominent factor is the structural rigidity afforded by the frozen substance at the surface where sublimation occurs. This rigidity to a large extent prevents collapse of the solid matrix remaining after drying. The result is a porous, nonshrunken structure in the freeze-dried product (Liapis *et al.*, 2002).

Drying of foods is a process involving simultaneous interface transfer of heat and mass (water vapor). In a typical air drying operation, a moist solid is placed in a closed environment in which hot air is circulated, by convection causing evaporation of water from the body (Aguilera *et al.*, 1999). A dehydrator allows making something that is not crisp, crisp, and, subsequently, to get a variety of textures, as says Chef Talbot (Leschin-Hoar, 2008). During drying microstructural changes occur due to several phenomena, such as, loss of structural structure, shrinkage, changes in macromolecules and changes in sugars. For instance, shrinkage affects the rate of drying as well as physical and functional properties of the product, and thus the shrinking behaviors of different food materials results in singular particle shapes and microstructures.

According to some authors, a shrinkage phenomenon is related to the glass transition of the matrix of soluble components, mostly sugars, but also proteins. The view that the glass-rubber transition explains structural changes during air drying appears to be based on studies of freeze-drying of sugars and polysaccharide solutions. Indeed this process of freeze-drying induces minimal product shrinkage unless the temperature of the dry matrix exceeds a “collapse temperature (T_c) (Aguilera *et al.*, 1999; Harnkarnsujarit, *et al.*, 2012). Some authors postulated that collapse is T_g -governed phenomena, and a close correlation was found between T_g and T_c for maltodextrins (e.g., T_c occurs 40°C to 70°C above T_g , depending on the moisture content) (Levine *et al.*, 1986; Tsourouflis *et al.*, 1976), since this additive serve as cryoprotector or cryostabilizer on freezing processes (Diniz-Mendes *et al.*, 1999; Yu *et al.*, 2012).

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As moisture content decreases during the latter part of drying, several reactions occur at the macromolecular level. Among the most important are crystallization of polysaccharides and aggregation of proteins (Aguilera *et al.*, 1997). In fact, this process generates a variety of stresses, such as low temperature stress, formation of ice crystals (Kiani *et al.*, 2011) and dehydration stress, which can destabilize proteins, and so to diminish those consequences many types of stabilizers have been used (Srirangsan *et al.*, 2010; Arakawa *et al.*, 2001; Wang, 2000). Another important factor on removal water content processes is the fact that rate of drying can affect the final state of a sugar, in view of the fact that, typically, rapid drying is associated with an amorphous rather than a crystalline state. For instance, hygroscopic whey powders contain mostly amorphous or glassy lactose whereas in nonhygroscopic powders most of the lactose is crystallized. As hygroscopic particles take up moisture from the air, mobility is increased and lactose molecules rearrange themselves into regular crystal lattices at a rate that depends on a_w and temperature.

Foaming is a process by which liquid or semi solid foods are whipped to form foams. Many foods, such as egg white, beef extract and milk, naturally contain soluble proteins, which can be converted into stable foams when being whipped (Thuwapanichayanan *et al.*, 2008). Foams and gels have gained notoriety among modern chefs for their light and exquisite textures. Aerated gels are gastronomic concepts in between the creations of two of the most reputed chefs in the world the “airs” of Ferrán Adrià and the “jellies” of Heston Blumenthal (This, 2006).

Aerated liquids are thermodynamically unstable; bubbles must be stabilized at their air-liquid interface usually by surface active agents such as proteins and emulsifiers or solid particles, like fat crystals. Furthermore, drainage and bubble coalescence is retarded by increasing the viscosity of the liquid in lamellae between the bubbles. If bubbles become physically entrapped in a gel network the food will be stable (Boom, 2007). The type of gas influences structure and stability of aerated products. Foams containing N_2 or air bubbles will coarsen slower than those consisting of CO_2 bubbles, since diffusion is largely determined by the solubility of the gas (Zúñiga *et al.*, 2008). Liquid foams comprise the dispersion of a gas (i.e., air, nitrogen, carbon dioxide) into a liquid continuous phase where the particular life-span of the foam ranges from seconds to several days (Pernell *et al.*, 2002). Their stability has been shown to be dependent on different factors such as bubble size distribution, volume fraction of air,

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beating time, protein type and concentration, the presence of small molecular weight surfactants and the viscosity of the continuous phase (Campbell *et al.*, 1999; Dutta *et al.*, 2002; Pernell *et al.*, 2002; Lau *et al.*, 2005; Allais *et al.*, 2006). The size of a bubble is a primary parameter determining its behavior and contribution to food structure and texture. Often there is a wide range of bubble sizes, with some sizes contributing more to appearance and others to texture (Lau *et al.*, 2005; Foegeding *et al.*, 2006).

Therefore, the preparation of emulsion or foam in the first place requires some kind of emulsifying or foaming agent. A class of emulsifying agent used in food processing are surface-active food biopolymers, mainly dairy and egg proteins, but also including some hydrocolloids (Dickinson, 2007). Stabilizing agents may be soluble proteins (McClements, 2005), soluble polysaccharides, which play several roles in contributing to the stability of foams and emulsions, namely slow down film drainage and enhance the life-time of foams, such materials as xanthan (Morris, 2007), or even, dispersed particles (fat crystals, casein micelles). Most soluble proteins have a strong tendency to adsorb at oil-water and air-water interfaces to form stabilizing layers, thereby fulfilling both the emulsifying or foaming role as well as the colloidal stabilizing role (McClements, 2007). The main stabilizing action of the hydrophilic hydrocolloids is via “thickening” and “structuring” of the aqueous continuous phase by means of viscosity modification and/or gelation (Dickinson, 2007).

Foams prepared from egg white and milk proteins are often used in foods as structuring materials. This means that the foam microstructure created prior to further processing (e.g., heating) will determine the final mechanical and textural properties of the food material. Liquid foams such as cappuccino, mousse, soufflé, sponge cake, meringue and beer head are, for the most part, protein-stabilized systems that may or may not be exposed to further processing (mostly heating) after aeration (Goff *et al.*, 2007). Combination of appropriate techniques for foam formation and drying indeed forms a basis for producing crisp food products (Thuwapanichayanan *et al.*, 2012).

The aim of this study was to produce freeze-dried and dried foams in order to evaluate each of these products had the most crispy structure, and also the complexity of the pore formation mechanism needs further study, and with these purposes were made some analysis, namely moisture content, water activity (a_w), weight loss, color analysis, texture analysis, sensorial analysis and scanning electron microscopy (SEM).

2. MATERIAL AND METHODS

Experimental Procedure

The experimental procedures occurred in three different places: (1) in the Laboratory of the “Departamento de Producción Animal y Ciencia de los Alimentos de la Facultad de Veterinaria de la Universidad de Extremadura” in Caceres (Spain) – definition of each two formulations for freeze-dried and dried whipped foams, definition of the conditions of freeze-drying process and convective drying process, and determination of physical and chemical properties; (2) in the Laboratory of the Technology and Applied Sciences Department in “Escola Superior Agrária” of “Instituto Politécnico de Beja” in Beja (Portugal) – texture analysis, namely Kramer rheological analysis in the Laboratory of Food Rheology and sensorial profile in the Sensorial Analysis Room, and the scanning images analyses (SEM) were made in "Servicio de Análisis y Caracterización de Sólidos y Superficies de la Universidad de Extremadura" (Badajoz).

The formula to prepare whipped foams for both processes: freeze-drying and convective drying is presented in table 16.

Table 16 – Ingredients percentage to prepare whipped foams formula

Ingredients	%
Natural Yogurt	42
Mineral Water	23.6
Isomalt “Cargill”	4.4
Maltodextrin “Cargill”	2.2
Metilcellulose 4000cp “Shin Etsu”	1
Xanthan Gum “El Majuelo”	0.2
Salt	0.2
Mineral Water	26.5
TOTAL	100

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The yogurts were dipped and mixed with half volume of water. Then the other half water was to dissolve isomalt, maltodextrin and salt and boiled for a few time until the mixture was well done. After boiling phase, metilcellulose and xanthan gum are stirred. After this, the yogurt was added and well mixed to the previous preparation. Finally, and still hot, that mixture was put in a 1L stainless steel foaming canister (“Ibili”), turn to seal tightly and loaded with two NO₂ cartridges and it was immediately put in a water/ice bath for 90 min.

Towards it was maintained under refrigeration along 4 days. Final foam yield was 3 and ½ yogurts to produce 1L, which allowed producing 56 foamed samples. After this resting period, the foam was put in silicon shapes and frozen at -80°C for 24h, and this phase as to occur very quickly so that the foam doesn’t breakdown. Following the freeze-drying process of the frozen foam at -50°C and under vacuum conditions in a Freeze-dryer (Telstar LyoQuest).

In parallel, another foam with 4 days refrigeration was also set in silicon shapes and placed on the dryer Excalibur at 57°C for 24h.

Both freeze-dried and dried samples were placed into boxes with silica until moisture content, water activity (a_w), weight loss, colour analysis, texture analysis, sensory analysis and image analysis (Scanning Electron Microscopy).

Laboratorial Procedures

After freeze-drying and drying processes the following analyses were made by a specific order: moisture content, water activity (a_w), weight loss, color analysis, instrumental texture analysis, sensory analysis and image analysis, more precise scanning electron microscopy (SEM).

Moisture Content

Moisture was determined by an oven at 103^o±2°C to constant weight (standard technique, official method NP-1614/1979 for meat and meat products). Ten replicates were performed for each kind of samples: freeze-dried (FD) and dried (D).

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Weight loss

For determining the weight loss of the freeze-dried and dried samples the following expression was used:

$$\% \text{ Weight Loss} = (\text{Foam initial weight} - \text{Freeze-dried or Dried Foam final weight}) / \text{Foam initial weight} * 100$$

The weight loss of the samples during freeze-dried and dried processes was expressed as shown in the previous expression where the initial weight (g) corresponds to the weight of the samples before freeze-dried and dried processes and final weight (g) corresponds to the weight of the samples immediately freeze-dried and dried processes. Ten replicates were performed for each kind of sample.

Color Analysis

Color measurements were made on each of selected freeze-dried or dried foam samples. The Commission Internationale d'Eclairage (CIE) L*, a* and b* values were determined using a chroma meter model CR-300, Minolta Camera Company, (Osaka, Japan) that was calibrated with a standard white calibration plate. The CR 300 chroma meter uses a xenon lamp as the light source. Fifteen replicates were performed for each freeze-dried or dried foam samples.

Instrumental Texture Analysis

A TA-HDi Texture Analyser (Stable Micro Systems, Godalming, UK) was used for the compression tests with a 25kg load cell. The probe used was the Kramer Shear Cell (HDP/KS5) at the following settings: test speed 2mm/s, trigger force 4N, travel distance of the blades 15mm and acquisition points per second 200pps.

Approximately ten to fifteen replications were performed for each kind of samples. Since dried samples had different weight and height, thus volume from freeze-dried foams, it was introduced into Kramer cell for each test two dried samples and one freeze-dried sample by test.

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The following parameters were calculated from the force *versus* time curves: maximum force (N), respective work force or area (Ns), number of peaks of the curve or crispness or fracture events (drop in force higher than 0.049N), slope (N/s) or gradient (slope of the curve up to the major peak) and displacement (mm), which means the all distance since the first breakdown event until maximum force.

The following figure shows a Kramer plot force/time of dehydrated foam with representation of maximum force (N) and number of peaks of the curve or crispness.

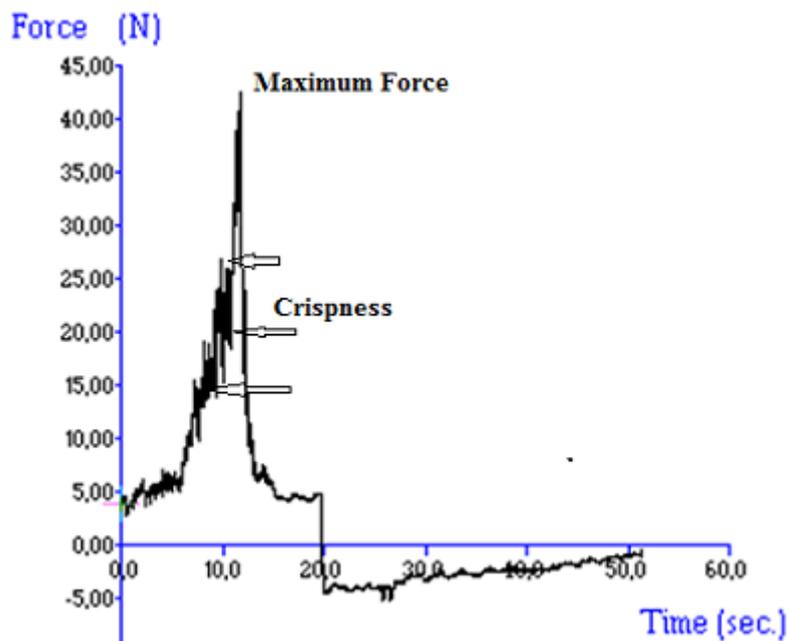


Figure 25 - Force *versus* time Kramer curves of dehydrated foams with representation of maximum force (N) and number of peaks of the curve or crispness

On figure 26 it was possible to observe a Kramer plot force/time of freeze-dried foam with representation of maximum force (N) and number of peaks of the curve or crispness.

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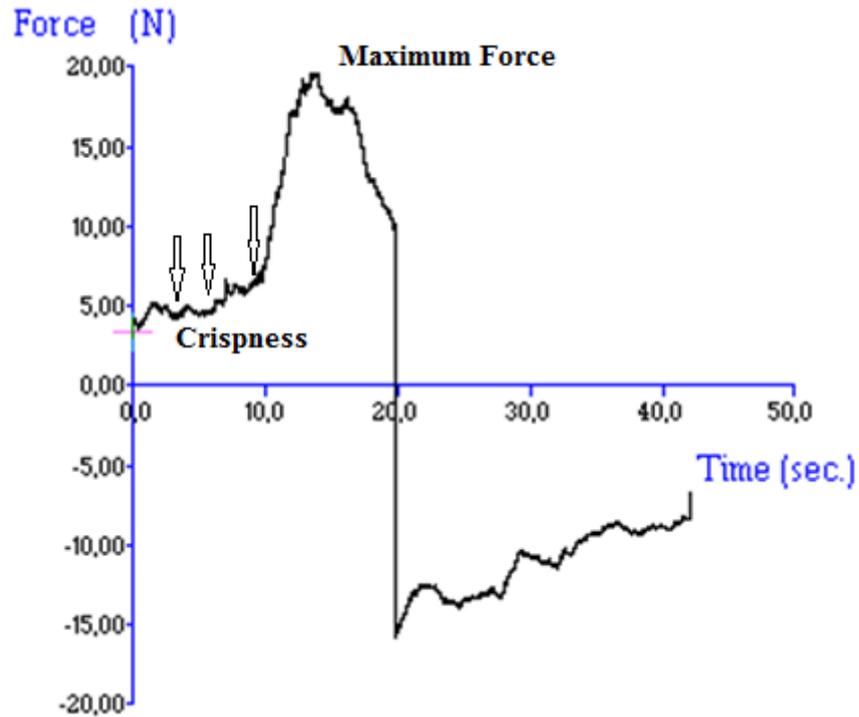


Figure 26 - Force *versus* time Kramer curves of freeze-dried foam with representation of maximum force (N) and number of peaks of the curve or crispness

Microstructure Determination

A scanning electron microscope (JEOL, JSM-5600LV, Tokyo, Japan) was used to study the microstructure of freeze-dried and dried foams. A sample was placed

SEM micrographs were taken at an accelerating voltage of kV and a magnification of...times.

Sensory analysis

A panel of 15 assessors with experience in the descriptive evaluation (NP ISO 8586-1, 2001) was used to evaluate the two samples of foams (freeze-dried and dried). Panellists were previously trained during months, first in group sessions in order to define descriptors to these kind of products (puffed snacks) and to reach a consensus among panellists on the meaning of every attribute, and then in six individual sessions with different puffed snacks with the aim of training them to recognize specific texture descriptors (hardness, crispness, crunchiness and friable character).

To achieve those, it is recommended in handbooks to provide assessors with a definition (Roudaut *et al.*, 2002). Testing was carried out in a sensory laboratory equipped with individual booths (NP ISO 8586-2, 2001). The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9).

To evaluate hardness the instruction was to bite the whole sample with the incisors until fracture and score the material resistance (Vincent, 2004). To score crispness the instruction was to evaluate altogether during mastication (first three bites), amount and quality of the sound produced (Chaunier *et al.*, 2005; Lazou *et al.*, 2010). To evaluate crunchiness the instruction was to score during mastication the number of layers with incorporated air (Dijksterhuis *et al.*, 2007). Friable was evaluated by instruction that during mastication the snack could break in numerous tiny pieces (vitreous state) which was the key on time to subsequent deglutition (Chaunier *et al.*, 2005). Pasty attribute was instructed to evaluate, during mastication process, the ability of the product to easily become a paste, like a chestnut puree. Toasted flavour was associated to non raw and inclusive comparable to slightly toffee (Bruwer *et al.*, 2007).

The samples were served in random order, each on a separate glass tray, identified with a letter random code. Panellists were instructed to rinse their mouths with water between sample evaluations.

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Statistical Analysis

Statistical analyses were conducted using a commercial statistical package, Design Expert version 6.01 (Statease Inc., Minneapolis, USA). Statistical significant difference between values was evaluated at $p < 0.05$ level with a comparison test using SPSS 12.0.1 (SPSS Inc., Chicago, Illinois, US) using the Tukey pair-wise comparison to determine the differences between treatment means and it was also used Microsoft Excel 2002 (Microsoft Corporation, Sacramento, USA).

3. RESULTS AND DISCUSSION

In the following table are shown the moisture content (%), water activity (a_w), weight loss (%) and degree of expansion (%) on freeze-dried (FD) and dehydrated (D) yogurt foam samples.

Table 17 - Moisture content, water activity (a_w), weight loss (%) and degree of expansion (%) on freeze-dried (FD) and dehydrated (D) yogurt foam samples

	FD	D	SEM	p
Moisture (%)	5.57	4.52	0.22	0.091
a_w	0.17	0.21	0.01	0.029
Weight Loss (%)	87.06	86.93	0.02	0.038

In table 17, it was possible to observe that besides $0.05 < p < 0.1$ on moisture content had showed a trend, Tukey test wasn't enough to detect significant differences between freeze-dried and dried foams, yet freeze-dried samples had a higher moisture content (5.57) than dried foams with 4.52%. However, water activity had showed significant differences with higher value on dried foams (0.21) than on freeze-dried foams (0.17). These parameters were done at the same time, but even maintaining samples well conditioned into recipients with silica, perhaps it wasn't enough to avoid a certain rehydration, in particular on freeze-dried samples, which might explain the insignificant higher difference in moisture content. However, since lactose glass is metastable and tends to sorb water, which results in plasticization, then water plasticization increases the molecular mobility (Omar *et al.*, 2007), and thus might justified the difference in those water parameters (moisture content and water activity) on freeze-dried foams.

Related to water activity, it was expected that this parameter would be lower on freeze-dried foams, since some nonfrozen water will almost unavoidably be present during freeze drying, but there is very often a rather quick transition temperature (T_g)

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for the still wet region, and thus the immobility of water molecules was higher in freeze-dried foams, than on dried foams.

Although there were significant differences on weight loss between the two foams, with higher value on freeze-drying foams (87.06), both values were high, which were in consonance with the lower moisture content values in both samples.

In the following table are shown the parameters from the force *versus* time Kramer curves, in order to achieve the following data: the maximum force (N), the area below the force curve (Force Work) (N.s), gradient or slope of the curve up to the major peak(N/s), displacement (mm) since the first breakdown event until maximum force, and number of peaks of the curve (fracture events) as sense of the instrumental crispness, on freeze-dried (FD) and dehydrated (D) yogurt foam samples.

Table 18 - Parameters from the force *versus* time Kramer curves: force and distance of the first breakdown event, displacement (mm), maximum force and number of peaks of the curve (fracture events) on freeze-dried (FD) and dehydrated (D) yogurt foam samples.

	FD	D	SEM	p
Maximum Force (N)	16.05	43.64	4.57	0.002
Slope (N/s)	1.65	267.08	0.35	0.001
Force Work (N.s)	78.27	13.50	12.86	0.003
Crispness	71.77	156.56	17.06	0.001
Displacement (mm)	7.76	3.18	0.73	0.027

In table 18, it was noticed that in all texture parameters both freeze-dried and dried foams had significant differences, as it was expected, since these two samples besides had in common the same formulation at the end of process were quite different.

Related to maximum force, freeze-dried foams had a significant lower force (16.05 N) than dried foams with 43.64 N, which were in fact rather hard on hand touch, and so the slope of the curve up to the major peak had also a significant higher value (267.08 N/s) on dried foams than on freeze-dried foams (1.65 N/s).

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However, the area below the curve or work force had a significant higher value on freeze-dried foams with 78.27 N.s, than on dried foams with 13.50 N.s, which possibly was influenced by a spongy nature on freeze-dried foams, which was notable in the force-time curve (figure 27a), and at a glassy nature on dried foams (figure 27b), but given also the fact that it was chosen a height criteria to determine the same volume of foams to set on Kramer cell.

A brittle object will exhibit a large hardness, low work to fracture and a sudden drop in force as the crack propagates rapidly (Miranda *et al.*, 2006). Therefore, on dried foams work force was lower than on freeze-dried foams, and these last samples of yoghurt freeze-dried foams had showed lower hardness, which was in consonance with some references to freeze-dried yoghurt that revealed a poor texture (Venir *et al.*, 2007). However, in table 18, SEM value on force work was relatively high.

The displacement, which means the all distance since the first breakdown event until maximum force, was also lower on dried foams with 3.18mm, than on freeze-dried foams with also a higher value of 7.76mm.

Related with the count of the number of peaks of the curve, which meant the crispness, it was noticed the highest value (156.56) on dried foams than on freeze-dried foams (71.77), since these foams had a higher number of fracture events (drop in force higher than 0.049N) as it was possible to see in force-time curve. However, standard mean error (SEM) value on crispness was rather high.

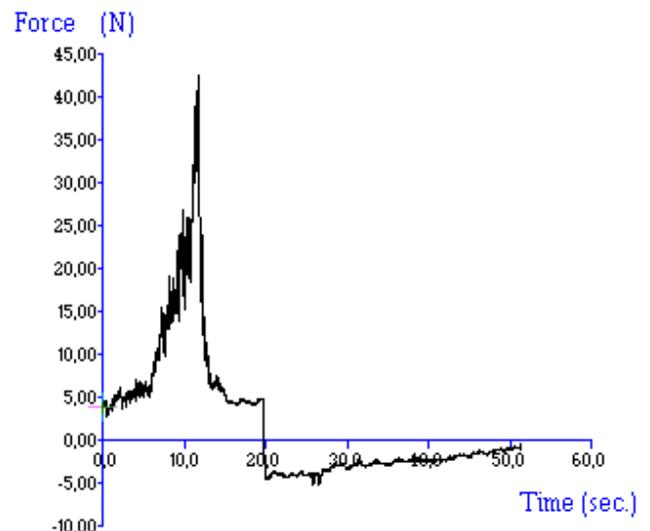
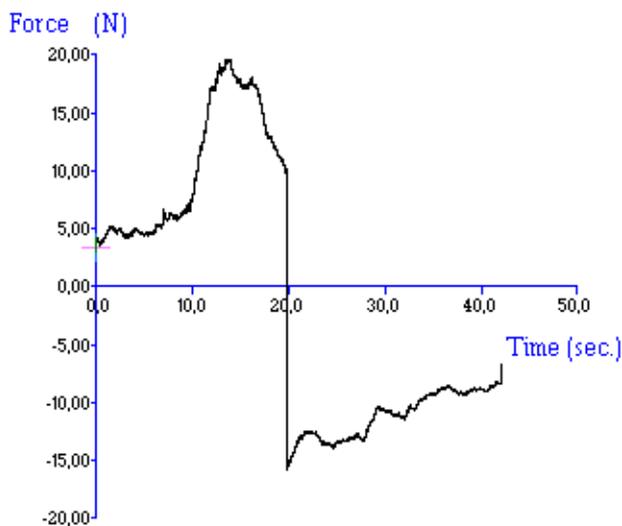


Figure 27 (a) - Force-time curve on Freeze-dried Foams

Figure 27 (b) - Force-time curve on Dried Foams

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In table 19 are shown the L*, a*, b*, Chroma and Hue color parameters from Lab CIE on freeze-dried (FD) and dehydrated (D) yogurt foam samples.

Table 19 – L*, a*, b*, Chroma and Hue color parameters from Lab CIE on freeze-dried (FD) and dehydrated (D) yogurt foam samples.

	FD	D	SEM	P
L*	93.33 ^a	79.18 ^b	1.10	0.001
a*	-0.99	-1.41	0.07	0.595
b*	3.17 ^b	4.79 ^a	0.29	0.001
Chroma	0.04 ^b	0.06 ^a	0.00	0.001
Hue	-0.30 ^a	-0.42 ^b	0.03	0.001

In color parameters, for parameter a* exception, all other parameters had shown significant differences between freeze-dried and dried foams. The more lightness foams were freeze-dried samples with a L value of 93.33, than dried foams with a L value of 79.18.

Dried foams were also the more yellowness samples with a b* value of 4.79, and freeze-dried foams had a significant lower b* value of 3.17, and in fact these last samples were less yellow since their thermal process (either freezing or freeze-drying) used always low temperatures (-80 to -50°C). In both foams, a* parameter had showed negative values, which was in agreement that their formulation had none raw material to contribute to a posterior redness, even after thermal process of freeze-drying or convective drying.

The scanning electron microscopy images of freeze-dried foams and dried foams are shown in figure 28 (a) and (b), respectively.

On freeze-dried foams, the image evidences the holes from ice crystals, and thus the fact that freeze-drying process occurred in good conditions since the structure maintained, without any sign of collapse. In effect, slow freezing produces big crystals and leaves large pores (porosities of most freeze-dried products range from 0.65 to 0.90).

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However, if freezing produces many small isolated ice crystals surrounded by a solid matrix, then the vapour must diffuse through the solid, and usually the matrix is cracked (Miranda *et al.*, 2006). Thus, these facts explain the non collapse structure with small evidences of holes. In fact, a major factor, during freeze-drying processing, is the structural rigidity afforded by the frozen substance at the surface where sublimation occurs, which fact prevents collapse of the solid matrix remaining after drying stage. Consequently, the result is a porous, nonshrunken structure in the freeze-dried product (Liapis *et al.*, 2002).

So, on freeze-drying processing, the water loss from the matrix contributes to the creation of pores in the resultant cellular solid (Rassis *et al.*, 1998) and the shape of the pores, pores size distribution, and pore connectivity of the porous network of the dried layer formed by the sublimation of the frozen water during the primary drying stage depend on the ice crystals that were formed during the freezing stage (Liapis *et al.*, 1982).

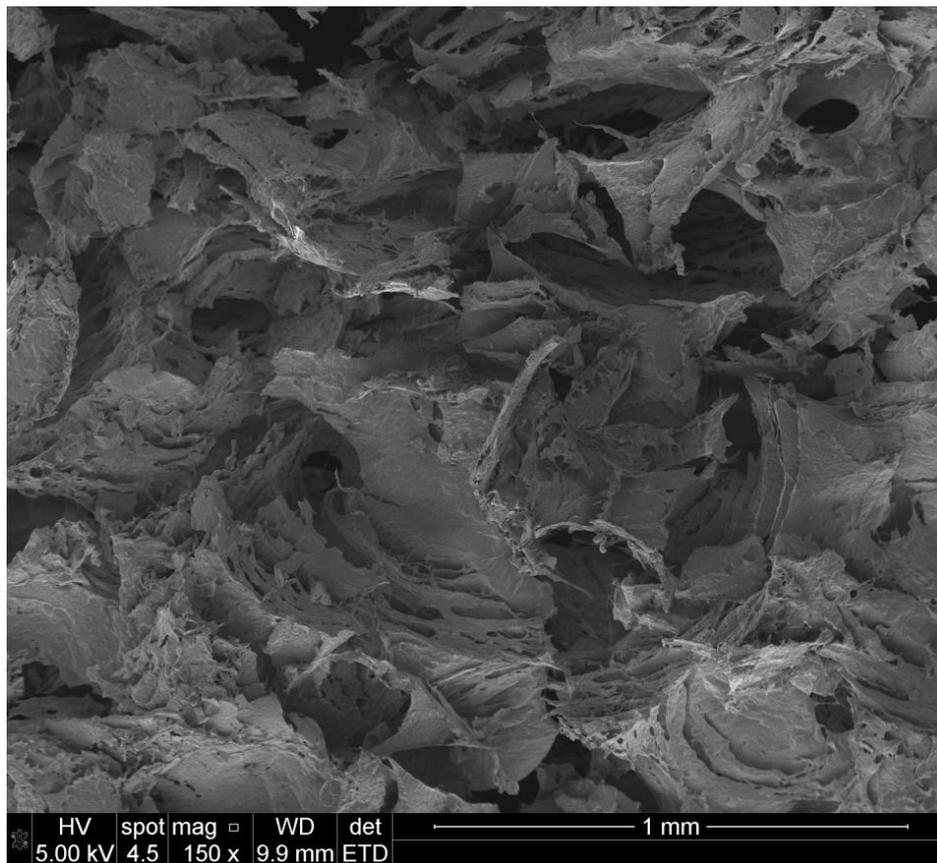


Figure 28(a) - Freeze-dried Foams SEM image

“Freeze-Dried and Dehydrated Yogurt Foams”

Dried foams image had shown a clearly absence of holes and an enormous collapse evidence in all structure, given the predominant visible plane area. In fact, during drying processing, microstructural changes occur such as loss of structural structure and shrinkage (Aguilera *et al.*, 1999) and hence induces structure collapse

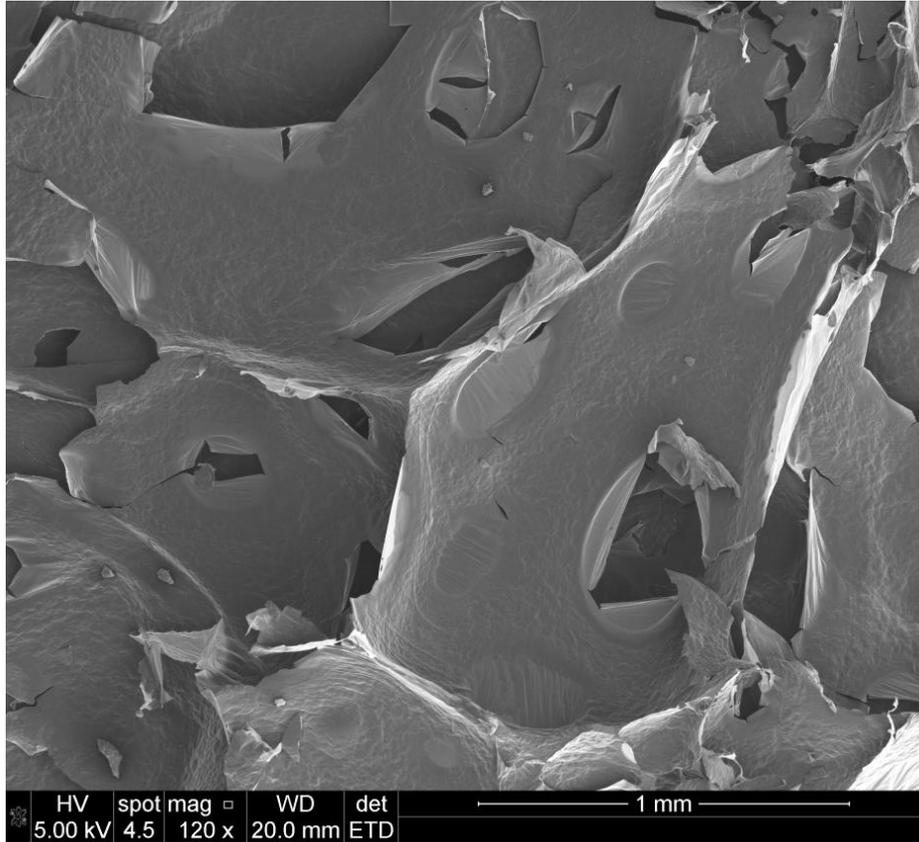


Figure 28 (b) - Dried Foams SEM image

The results on table 20 were obtained with a panel of 15 assessors with experience in the descriptive evaluation, who were used to evaluate the two yogurt foam samples processed by freeze-drying or by dehydration methods. Panellists evaluated at the same time for each of the six sessions both two samples in order to evaluate the specific descriptors: color (white and dark), hardness, crispness (sound), crunchiness and friable character, pasty sensation, sweat, salty or acid taste, flavour and, finally, samples overall acceptability.

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Table 20 – Sensory parameters: color, hardness, crispy (sound), crunchiness and friable character, pasty sensation, sweat, salty or acid taste, flavour and overall acceptability on freeze-dried (FD) and dehydrated (D) yogurt foam samples.

	FD	D	SEM	p
Color	1.90	4.91	0.47	0.004
Hardness	1.72	6.01	0.67	0.091
Crispy (Sound)	2.11	6.75	0.71	0.060
Crunchiness	3.09	5.50	0.39	0.264
Friable	2.61	5.96	0.52	0.561
Pasty	4.81	2.46	0.37	0.510
Sweat Taste	3.64	3.67	0.09	0.336
Salty Taste	2.73	3.00	0.07	0.362
Acid Taste	3.82	4.16	0.14	0.351
Flavour	2.11	4.03	0.32	0.764
Overall Acceptability	3.53	5.60	0.33	0.215

The intensities of sensory attributes were scored on 9 cm unstructured line scales labelled from “low” (0) to “high” (9)

The results on table 20 had shown that only color attribute had significant differences between the yogurt foam samples processed by freeze-drying or by dehydration methods.

In particular, freeze-dried foams had scored 1.90 and dried foams had scored 4.91; which were in accord with CIE L*a*b* color values, since freeze-dried foams were the most light samples and dried foams the most dark samples.

Although Tukey test wasn’t enough to detect significant differences in both hardness and crispy (sound) attributes, since $0.05 < p < 0.1$ this had showed a trend for significant differences, and indeed the panellists, whom had evaluated at the same time for each of the six sessions both two samples had scored higher hardness and crispness values on dried foams, than on freeze-dried foams.

So, for hardness, dried foams had scored 6.01 and freeze-dried foams had a lower score of 1.72, which was in agreement with textural hardness on table 2, with the highest maximum force value (43.64 N).

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Related to crispness (sound), dried foams had scored 6.75 and freeze-dried foams had a lower score of 2.11, thus these values were as well in accord with crispness textural values, which had presented on freeze-dried foams a lower value (71.77) than dried foams (156.56). In addition, these results indicated that a certain degree of sensory hardness is necessary for crispness perception, as those values had a similar behaviour.

Since friable and pasty attributes are connected, it was notable that panellists perfectly had understood these attribute, since those values were proportionally inverted. In fact, panellists had given a higher score to dried foams on friability (5.96), thus these samples had an outstanding vitreous state, and freeze-dried foams, with score of 2.61 on friable character, hadn't revealed such a glassy state during the sensory sessions.

In the mean time, pasty attribute had scored the highest value on freeze-dried foams with 4.81, while dried foams had scored 2.46, therefore these last samples didn't entirely lost its glassy state, in view of the fact that its pulp character was minor when comparable with freeze-dried foams predominant mash character during mastication phase.

In relation to sweat, salty and acid taste, besides the fact that these attributes hadn't showed significant differences, it was perceptible that dried foams had higher values in all three attributes, which was in conformity with the fact that this foams had lower moisture content than freeze-dried foams. As a consequence, even having the same formulation, the dried foams differentiate more these attributes, since this sweat, salty and acid characteristics were more concentrated.

In this attributes, acid taste was the one that presented the highest scores in both foams, which fact was expectable due to the yoghurt acid source and also to the hydrocolloid selection. In fact, the choice of the proper type of hydrocolloid used is one of the most important factors in the manufacture of fermented dairy products. In applications such as yoghurt, it is important that the hydrocolloid do not mask the natural flavour of the product and that they are effective at the typical product pH range (4.0 – 4.6), and thus, suitable hydrocolloids often include xanthan gum (Williams *et al.*, 2003; Gallardo-Escamilla *et al.*, 2007).

Flavour character to toasted had a higher score on dried foams (4.03) than on freeze-dried foams (2.11) given the fact that the dried foams were at 57°C for 24h while

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freeze-dried foams were always at lower temperatures. Further, and for the same reasons pointed above, the moisture content was lower on dried foams which enhanced flavour attribute.

Finally, on samples overall acceptability, besides the fact that there weren't significant differences, the panellists had given a higher score to dried foams with 5.60 than to freeze-dried foams with 3.53. Additionally, even without significant differences, it was important to achieve this result, since both samples were well accepted by the panellists, but their aim was to distinguish a crispy product, and therefore this panel was well succeeded.

4. CONCLUSIONS

Freeze-dried foams or dried foams prepared with hydrocolloids, such as metilcellulose or xanthan gum, will permit to achieve the production of crispy snacks with unique taste, colour and flavour.

Dehydration and freeze-drying will promote different characteristics on these foams microstructure, and thus lead to a distinguishably instrumental texture and sensory characteristics.

IV – RESULTS AND DISCUSSION

(IV - RESULTADOS Y DISCUSIÓN)

Texture of crispy snacks produced by an intense dehydration (by frying, microwaving, dehydrating or freeze-drying) of either a protein or a carbohydrate base, strongly depends on the extent of dehydration, and thus the final water content.

This latter factor is directly related to the development of a puffed structure in the case of puffed snacks (such as microwaved pork rinds) in which the water steam serves as the force enabling the structure to expand.

Moreover, is also related to the glassy state of the compounds forming the structure of the snacks, in such a way that if the water content is too high, compounds will remain in an elastic rubbery state, which in turn would lead to a chewy and less crispy texture. On the other hand, if the water content is low enough, a glassy structure will be achieved, leading to a brittle structure which results in a crispy and crunchy texture.

In battered fried foods, during frying processing the product undergoes expansion in the thickness due to gas bubble inside the product, and, thus puffed structure occurred at about time the crust was forming. So, at the same time, the formation of the crust greatly reduces the rate of moisture transfer and causes an increase in pressure inside the frying product, which buildup leads to an expansion of the pores, resulting in a crispy final product.

Edible coatings and incorporation of active ingredients not so common, such as CO₂ and ethanol, can improve batter functionality. For instance, CO₂, led to a batter with more bubbles, and therefore, a coater less thickener and crispier, and alcohol during frying, evaporates more rapidly than water, and consequently, after frying, the coater dries quickly and becomes crispy. So, that these products are tender and moist on the inside with a porous crunchy and crispy crust, which will determine the final crispness after reheating these coatings (for instance on oven).

Other factors related to the structure and microstructure, such as pore size distribution, solid density of walls surrounding pores or gluten development, will also strongly affect the crispy and crunchy texture of these types of products. As a general

Results and Discussion

rule, the higher the number of pores, the crispier will be the product. Very compact microstructure will lead to a harder texture when chewing, but not necessarily to a crispier texture.

METHODS: different analytical methods will inform about different texture features of this type of snacks. Sensory methods are very useful since provide direct information about crispiness or crunchiness. However, it shows also drawbacks: they are time consuming, they are highly influenced by the persons included in the panel and their training, and some times, and they could be not sensitive to exactly describe the changes or differences produced as a consequence of modifications in the production methodology.

Instrumental texture measurement methods can be very useful, since they allow the analysis of a high number of samples in short time. These methods can very accurately measure features related to the crispiness of the product, such as number of fracture events. However, they strongly depend on the type of probe used and the design of the procedure. Thus, sometimes even methods described for this type of crispy snacks, are not sensitive enough to detect differences that are evidences using other methods.

Puncture tests can be tested individually or as bulk when contained in a cell. A spherical probe penetration into the food causes irreversible crushing, with the purpose to stimulate the incisors impact at biting, but provide the instrumentally texture assessment, in particular, the area below the force curve, number of force peaks (drop in a force threshold), and gradient (slope of the curve up to the first major peak). The Kramer cell compression–extrusion test applies a force by descending the blades to a food until it flows through an outlet in the bottom of the cell. The food is compressed until the structure of the food is disrupted and it extrudes through these outlets, in order to measure breaking point; specific shear force and toughness (work or area). The curves force-deformation in crisp materials is usually irregular and compressive force alone was insufficient to describe the texture of crispy foods.

Results and Discussion

The measurement of sound produced during the fracture events that take place during a penetration test using a spherical probe seems to be a very valuable and sensitive method for measuring crispness. While this method needs a somehow sophisticated equipment and methodology, it offers a very real measurement of what the consumer feels when chew a crispy snack. From the sound curves, it is given information about the number of sound peaks (drop in a sound pressure level threshold) and also provides the sound pressure level (average of the ten higher peaks, SPL_{max10}).

In fact, crispier samples produce more total noise, as a result of higher sound amplitude or a greater density of sound occurrence. In other words, the loudness of the sounds produced the number of sounds produced in a given biting distance, seemed to cause a change in perceived crispness.

In food structures, cell wall fracturing releases the internal pressure which can be detected as the characteristic crispy sound. On this basis, recording sounds and force patterns produced during the force application in instrumental assessments of texture could be an experimented method to obtain quantitative information regarding crispy sounds and to predict the sensory appreciation of crispness.

Indirect methods provide very interesting pieces of information, such as the moisture content, number of pores, pore size distribution, arrangement of solids forming the microstructure, or architecture of such microstructure. While some of these methods are very useful from a practical point of view (for example, water content or moisture loss), since they are easy and cheap and provide information that could be used as a quality marker (they are related to the final quality of the product), others (SEM or image analysis) are not useful from a production line point of view, but provide important information about the structure of the product (useful when developing the product to understand the events that take place during production).

V – CONCLUSIONS

(V – CONCLUSIONES)

1. Las cortezas de cerdos expandidas en microondas tienen características similares a las de las tradicionales (fritas) pero con un contenido mucho menor de grasa.
2. La deshidratación de las cortezas de cerdo desestructuradas es mucho más rápida que la de las cortezas íntegras. Sin embargo éstas últimas se expanden mejor cuando son calentadas en el microondas
3. El grado de desecación de las cortezas de cerdo, previo a la expansión en microondas, tiene una gran repercusión sobre las características estructurales y texturales del producto, ya que determina tanto el grado de expansión durante el calentamiento, como el estado vítreo o plástico, que alcanza la malla proteica expandida.
4. La medida de algunas de las características de textura instrumental y de sonido durante la compresión con una sonda esférica de las cortezas de cerdo expandidas en el microondas, proveen de una información que se relaciona adecuadamente con la obtenida mediante análisis sensorial y con las características del producto. Sin embargo, los parámetros de textura instrumental obtenidos en una celda Kramer no reflejaron adecuadamente las diferencias entre productos ni las puntuaciones de los catadores.
5. La sustitución del agua por etanol en la elaboración de la masas para rebozado permite obtener un rebozado más crujiente y en el que se mitiga la disminución de esta característica durante su almacenamiento y posterior recalentamiento en el horno. La incubación de dicha masa con CO₂ tiene consecuencias no tan claras, aunque en general parece aumentar el carácter crujiente del rebozado.
6. La elaboración de espumas desecadas desecadas o liofilizadas con mezclas de hidrocoloides tales como la metilcelulosa o la goma xantana, permite la elaboración de snacks crujientes con características personalizadas de sabor, aroma y color.

Conclusions

7. La desecación y la liofilización conducen a características muy diferentes en la microestructura de estas espumas deshidratadas, que se reflejan tanto en la textura instrumental como en las características sensoriales de las mismas.

**VI – REFERENCES
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