



Understanding microstructural changes of starch during atmospheric and vacuum heating in water and oil through online in situ vacuum hot-stage microscopy

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ABSTRACT

Starch gelatinization is a critical micro-scale phenomenon occurring during frying, which gets particular relevance during vacuum frying, where minimum conditions need to be fulfilled to get the required structure. The objective of this research was to understand the effect of water accessibility, temperature and vacuum level, in starch microstructural changes during heating in water and oil, in situ and in real time, using vacuum hot-stage microscopy. Potato starch was heated in water or in a pre-gelatinized corn starch solution, at different pressures (atmospheric, 6.5 kPa, 30 kPa). Also, a gluten–starch matrix immersed in oil was followed under the microscope. An increase in the heating rate as well as water inaccessibility increased the on-set gelatinization temperature. Under high vacuum no changes were observed, whereas under low vacuum, partial gelatinization occurred. Overall, this microstructural approach may help in explaining the high oil absorption of vacuum-fried formulated starchy products reported in previous studies.

Industrial relevance: Vacuum frying is a novel and promising technique that allows obtaining fried products with a low amount of oil, making them healthier than atmospheric fried ones. So far most of the studies have focused on agricultural products, such as potatoes, which are already structured by nature, without focusing on fabricated/restructured products. This imposes a challenge since minimum conditions must be ensured to form structure during processing in this product category. In fact, vacuum fried restructured products have shown to increase their oil content compared to their atmospheric counterpart, probably due to lack of structure formation during processing. From this perspective, the understanding of the main microstructural changes of starch in formulated products during processing under atmospheric or vacuum conditions is of interest. Since, most of the structural changes occur at the micro-scale, process miniaturization using vacuum hot-stage microscopy seems to be a good approach.

In accordance, the objective of this research was to understand the effect of water availability, temperature and vacuum level, in starch microstructural changes during heating in water and oil, in situ and in real time. Potato starch was heated in excess of water or in a pre-gelatinized corn starch solution, at different pressures. Also, a gluten–starch matrix immersed in oil was followed under the microscope.

We found that an increase in the heating rate as well as water unavailability increased the on-set gelatinization temperature, which is a key process to reduce oil absorption. Further, under high vacuum no changes were observed, whereas under low vacuum, partial gelatinization occurred. The lower degree of gelatinization determined in vacuum-fried matrices compared to atmospheric fried ones, may explain the high oil absorption of formulated starchy products reported in previous studies, as a consequence of deficient structure development. The understanding of these properties could help the food industry to produce healthier low-fat formulated snacks.

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1. Introduction

In foods, most of the critical processes and transformations, such as transport properties, textural and rheological behavior occur in a scale range which is below 70 μm (Wayne, 2008) and, therefore, cannot be

seen with the naked eye (Aguilera, 2005). Different microscopy techniques have allowed researchers to understand the microstructure of several food materials. This, in conjunction with fundamental scientific knowledge, allows improving food product design, which may well be achieved through product formulation.

A widely used operation in the snack industry to develop ready to eat products is deep-fat frying. In this process the main changes and properties, such as oil absorption and microstructure development, occur in the micrometric length scale. Consequently, studying the microstructural changes in formulated products at the micro-scale could

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be an adequate technique to understand the main changes associated to the process. Advantages of using specific food building blocks to create controlled formulated matrices include their reproducibility, uniformity and lack of defects. To be able to design these new snacks it is important to understand the properties of starch, since starchy foods represent an important product category. This includes the understanding of factors that may affect its gelatinization temperature as well as its microstructural changes during processing and storage, to better understand the relationship between macro (finished-product) and micro-properties.

Starch granules are insoluble in water at room temperature. In the presence of adequate temperature and water its crystalline structure is disrupted, allowing the amorphous regions to become more accessible to absorb water and swell, a process known as starch gelatinization. If the amount of water is insufficient, the process cannot occur. Fukuoka, Ohta, and Watanabe (2002) studied the effect of moisture content on the gelatinization temperature of wheat starch using differential scanning calorimetry (DSC), concluding that a decrease in moisture content led to a higher gelatinization temperature. According to this, the degree of gelatinization of a starchy product should depend on the temperature and water availability in the system (Primo-Martin, van Nieuwenhuijzen, Hamer, & van Vliet, 2007).

During frying the product is immersed in edible oil heated at a high temperature (generally above 150 °C). This complex unit operation includes simultaneous heat and mass fluxes and results in significant microstructural changes (Singh, 1995). The main changes that a starchy product may suffer are associated to fast dehydration, starch gelatinization, softening of cell walls and crust formation, among others (Bouchon & Aguilera, 2001; Llorca et al., 2007). An important issue related to frying is the amount of oil absorbed by the product. This process is not yet fully understood, but it is believed that the escape of water would act as a barrier to oil absorption during frying and most of the oil would penetrate upon cooling, after removing the product from the oil bath (Bouchon, Aguilera, & Pyle, 2003). Overall, there is wide evidence that relates the microstructure of the crust with oil absorption (Bouchon & Aguilera, 2001; Pinthus, Weinberg, & Saguy, 1995) and the degree of gelatinization of the granules may certainly act as a hurdle to oil uptake (Rajkumar, Moreira, & Barrufet, 2003).

Vacuum frying, on the other hand, is a novel technique that allows the removal of moisture in a low temperature and oxygen environment due to water boiling point depression (Garayo & Moreira, 2002). As Dueik and Bouchon (2011a) proposed, the temperature processing should not be too low, since the desired microstructural changes may be inhibited. Due to the characteristics of the process, in some food matrices, oil reduction may be achieved. However, the mechanism of oil uptake during vacuum frying is less understood than in atmospheric frying. Mariscal and Bouchon (2008), when studying vacuum frying of apple slices, observed that atmospheric apple slices absorbed 21% more oil than vacuum fried ones. Also, Dueik and Bouchon (2011b) reported that vacuum fried potato and carrot chips absorbed 50% less oil than the atmospheric fried ones. However, when vacuum frying formulated matrices made from starch, gluten and water, an increase in oil absorption was observed (Kawas & Moreira, 2001; Sobukola, Dueik, & Bouchon, *in press*). This has been attributed to a decrease in the degree of starch gelatinization, which does not allow the formation of a barrier to preclude oil absorption. Sobukola et al. (*in press*) studied the oil absorption of formulated products using different vacuum levels. Their results showed that a higher water boiling point (lower vacuum) favored the capacity of the matrix to form structure, reducing the oil content of the fried products. In addition, since the processing temperature is substantially lowered, beneficial compounds and color may be greatly preserved (Da Silva & Moreira, 2008; Dueik & Bouchon, 2011b).

A way to understand the dynamics of starch gelatinization and the development of the fried structure in real time is through process miniaturization. This term was coined by Aguilera and Lilford (1996) and refers to the following-up of a tiny process in a particular stage (e.g. hot-stage) *in situ* and in real time under the lens of a microscope. A

major advantage of this technique is that it is non-invasive, since sample preparation requires minimal processing. Another advantage is that the operation conditions (e.g. temperature, pressure, relative humidity, among others) may be carefully controlled; thus the microstructural changes can be associated to specific conditions. This technique has been used to study diverse processes and therefore could be a valuable tool to get a better understanding of some microstructural changes during atmospheric and vacuum frying of formulated products. Bouchon and Aguilera (2001) studied atmospheric frying of potato tissue. Kaushik and Roos (2007) examined the encapsulation of limonene during freeze-drying and Heuer, Cox, Singleton, Barigou, and van Ginkel (2007) studied the changes in corn syrup foam microstructure under pressure. Arellano, Aguilera, and Bouchon (2004) studied lactose crystallization kinetics in water whereas James and Smith (2009) followed fat crystal growth in chocolates. Also, Baier-Schenk et al. (2005) followed the microstructural changes of wheat dough during freezing and Saragoni, Aguilera, and Bouchon (2007) examined powder caking of coffee particles during storage.

In accordance, the aim of this study is to understand the effect of water accessibility and processing conditions (temperature and vacuum level) in the microstructural changes that occur in potato starch granules heated in water and oil *in situ* and in real time, through process miniaturization, to improve understanding of starchy food behavior during atmospheric and vacuum frying.

2. Materials and methods

2.1. Materials

Potato starch granules and pre-gelatinized corn starch were obtained from Blumos S.A. (Santiago, Chile) and wheat gluten was acquired from Asitec S.A. (Santiago, Chile).

2.2. Sample preparation

Different systems were prepared to understand the effect of water accessibility in potato starch behavior.

2.2.1. Isolated granules behavior in water

Ten droplets of water were added to the sample holder (microcapsule to be mounted in the microscope). Starch granules were immersed and scattered over it to improve isolation and identification.

2.2.2. Isolated granules behavior in solutions of pre-gelatinized corn starch

First, the water content of the pre-gelatinized corn starch was determined drying in a forced air oven (LDO-080F, Labtech, Korea) at 105 °C for 24 h (to constant mass). Then, 3 and 5% pre-gelatinized corn starch solutions were prepared by carefully determining the amount of water and pre-gelatinized starch to be added. Then, 10 droplets of the solution were added to the sample holder and few potato starch granules were immersed and scattered over it.

2.2.3. Starch granules behavior in starch–gluten–water matrices

Matrices of potato starch, wheat gluten and water were prepared in order to understand the role of gluten and water in the gelatinization process. Experimental procedures were based on a formulated matrix made of a reconstituted blend of gluten (12% d.b.) and starch wheat (88% d.b.) instead of wheat flour, in order to accurately control ingredient proportions. The dough was prepared according to the methodology proposed by Moreno, Brown, and Bouchon (2010), ensuring a water content of 35% (w.b.) in the dough. This amount is lower than the one in standard dough, which is around 40% (Moreno et al., 2010). It was used to be able to observe and distinguish the granules from each other in the matrix during the miniaturization process.

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