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Microstructure and water distribution of commercial pasta studied by microscopy and 3D magnetic resonance imaging

Thomas Steglich [a,b,*], Diana Bernin [b,c], Magnus Röding [d], Magnus Nydén [e], Annelie Moldin [f], Daniel Topgaard [g], Maud Langton [h]

[a] SIK — The Swedish Institute for Food and Biotechnology, Göteborg, Sweden
[b] Department of Chemical and Biological Engineering, Chalmers University of Technology, Göteborg, Sweden
[c] Swedish NMR Centre, Gothenburg University, Göteborg, Sweden
[d] Department of Mathematical Sciences, Chalmers University of Technology, Göteborg, Sweden
[e] Ian Wark Research Institute, University of South Australia, Mawson Lakes SA 5095, Australia
[f] Lantmännen Cerealia, Järna, Sweden
[g] Division of Physical Chemistry, Lund University, Lund, Sweden
[h] Department of Food Science, Swedish University of Agricultural Sciences (SLU), Uppsala, Sweden

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ABSTRACT

Manufacturing pasta is a rather well known process, but it is still challenging to tailor pasta products with new raw materials. In this study, we evaluated the effects of raw materials on the microstructure and water distribution in cooked pasta using ^{1}H magnetic resonance imaging (MRI) as well as bright field and polarized light microscopy. The MRI parameters initial intensity (I_0) and transverse dephasing time (T_2^*) serve as indicators of the local water concentration and water–macromolecule interactions through chemical exchange, respectively. These parameters were mapped throughout the whole pasta volume with a spatial resolution of 78 μm in all three dimensions. MRI was combined with light microscopy to link I_0 and T_2^* to microstructure components such as fiber particles and the extent of starch gelatinization. Four commercial spaghetti samples were analyzed which were made of durum wheat flour, both plain and enriched with wheat fiber, as well as with wholegrain and soft wheat flour. Although all pasta samples showed similar macroscopic water absorption as measured by weight increase, the sample structures differed at the microscopic scale. Compared to durum wheat spaghetti, the presence of fiber particles decreased T_2^*, while spaghetti enriched with soft wheat flour increased T_2^*. In addition, light microscopy showed that large fiber particles partly acted as barriers against water migration and protected starch granules from swelling. Smaller wheat fiber particles did not affect local starch swelling. Thus, the combination of light microscopy and MRI is a powerful tool to study the microstructure and water distribution in pasta.

Introduction

Humans have been cooking pasta for centuries but we still do not fully understand what determines the quality of cooked pasta. Semolina (the coarse flour from durum wheat) has in recent years been enriched, fortified or substituted — to increase the nutritional value, to decrease allergenicity or to use more abundant flours (reviewed by Fuad & Prabhasankar, 2010; Sissons, 2008). One example is to increase the dietary fiber content by using e.g. bran-rich flour. However, this often affects the perceived texture negatively (Aravind, Sissons, Egan, & Fellows, 2012; West, Seetharaman, & Duizer, 2013). It was speculated that the changed texture perception results from a disrupted gluten matrix due to the presence of fiber particles (Manthey & Schorno, 2002) or through increased physical or chemical interaction between fiber and gluten (Noort, van Haaster, Hemery, Schols, & Hamer, 2010).

Cooking transforms the two main components in durum wheat pasta: the starch gelatinizes and the gluten polymerizes. Both components compete for water and the outcome defines the final texture properties (Fuad & Prabhasankar, 2010). Added fiber particles might interfere in this competition for water (Bock, Connelly, & Damodaran, 2013). Thus, a better understanding of water migration in pasta during cooking could reveal how raw materials such as fiber particles influence transformation of starch and gluten.

^{1}H magnetic resonance imaging (MRI) is a non-invasive and non-destructive method to visualize the water distribution in pasta (Lai & Hwang, 2004). Several studies linked the water distribution to texture properties in cooked pasta (Gonzalez, McCarthy, & McCarthy, 2000; Horigane et al., 2006; Irie, Horigane, Naito, Motoi, & Yoshida, 2004; Kojima, Horigane, Nakajima, Yoshida, & Nagasawa, 2004; Maeda, Horigane, Yoshida, & Aikawa, 2009; McCarthy, Gonzalez, & McCarthy,

* Corresponding author at: SIK — The Swedish Institute for Food and Biotechnology, Göteborg, Sweden. Tel.: +46 10 516 67 19.
E-mail address: Thomas.Steglich@sik.se (T. Steglich).

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2002). Bonomi et al. (2012) correlated it to sensory perception and protein network changes. Sekiyama et al. (2012) used MRI to relate the water distribution to differently cooked zones in pasta as determined by fluorescence microscopy.

The aim of this study is to determine the influence of raw materials on the microstructure in cooked pasta. Our MRI acquisition procedure (with a spatial resolution of 78 μm in all three dimensions) yields data with a level of detail that far exceeds the ones from the studies cited above, where the two-dimensional images represent averages over slices with a thickness of 1 mm or more. We monitor ^{1}H MRI parameters and relate them to the microstructure and the local gelatinization degree of starch granules by comparison between MRI and light microscopy images.

Materials and methods

Materials

Four commercial spaghetti products were supplied by Lantmännen Cerealia (Järna, Sweden). The samples were all produced with a diameter of 1.7 mm and were made of 100% fine durum wheat semolina (DS) or of mixtures of durum wheat semolina and wheat fiber (DS + FB), durum wholegrain flour (DS + WG) or soft wheat flour (DS + SW), respectively. The composition of the major components (protein, total carbohydrate, fat, fiber content) of the pasta samples was obtained from Eurofins Scientific (Sweden, Table 1). Distilled water was used for all experiments.

Macroscopic water absorption

Single strands of spaghetti (25 ± 2 mm in length) were placed in glass tubes containing 10 mL of boiling water (100 °C) in accordance with Del Nobile, Baiano, Conte, and Mocci (2005). Samples were removed at intervals (each 30 s for the first 5 min, then each 60 s until 14 min and finally each 120 s until 20 min), cooled down, blotted and weighed. The weight increase was determined as the mass ratio between the cooked and the dry sample (W/W_0), respectively. Tests were performed in triplicate.

Water absorption was evaluated for significance ($P < 0.05$) for every cooking time by analysis of variance (ANOVA) followed by the least significant difference test to compare means ($P < 0.05$) using Microsoft Excel.

Bright field and polarized light microscopy

The four samples had slightly varying recommended cooking times. For better comparison, we chose to cook all samples to the same time being close to the recommended cooking times. Thus, single spaghetti strands were cooked in an excess of boiling water for 10 min and cooled down in ice water.

For polarized light microscopy, samples were immediately frozen to −20 °C and cut to a thickness of 10 μm using a cryostat (Leica, Austria). The slices were analyzed without staining.

For bright field light microscopy, sample pieces of 5 mm length were stored in a fixation solution (2% glutaraldehyde with 0.1% ruthenium) until further preparation to avoid evaporation. Samples were double fixated in 2% glutaraldehyde with 0.1% ruthenium and in 2% osmium oxide in phosphate buffer, respectively. After washing, the samples were dehydrated in ethanol and embedded in epoxy resin (Technovit 7100, Heraeus Kulzer, Germany). Cross sections and longitudinal sections of 1.5 μm thickness were cut using an ultramicrotome (Reichert Jung, Austria) and stained with Light Green for 30 min as well as with iodine in a Lugol's solution (2:1) for 30 s. In the resulting images, proteins are stained in green, starch granules in blue/violet, amylose in blue and amylopectin in brown.

Images were acquired with a Microphot-FXA (Nikon, Japan) equipped with an Altra 20 camera and recorded with cellSens Dimension 1.5 (Olympus, Germany).

Magnetic resonance imaging (MRI)

MRI experiments were run on an 11.7 T Avance II spectrometer (Bruker, Germany) operating at a ^{1}H resonance frequency of 500 MHz. The magnet was fitted with a Bruker MIC-5 microimaging probe giving a maximum gradient strength of 3 T m^{-1} in three orthogonal directions. A 5 mm radiofrequency coil was used for excitation and detection of the ^{1}H signal.

Spaghetti samples were cooked for 10 min and then immediately placed into cold water to avoid further starch gelatinization. A 10 mm lengthwise piece was cut out of the cooked spaghetti and placed immediately in a 5 mm NMR tube filled 20 mm in height with fluorinated oil (HT 230, Lesker, England). The fluorinated oil, which does not contribute to the ^{1}H images, was used to avoid water evaporation from the cooked spaghetti and to reduce image artifacts resulting from differences in magnetic susceptibility between the pasta and the surrounding medium. MRI measurements were started within 15 min after cooking.

A 3D gradient echo pulse sequence (MGE Paravision 4.0, Bruker) was used to acquire images with spatial resolution 78 × 78 × 78 μm^3, image matrix 192 × 64 × 64, and field-of-view 2.0 × 0.5 × 0.5 cm^3. We chose a gradient echo because the small flip angle (30°) of the radio frequency pulse shortens the repetition time and thus the acquisition time. In consequence, this setup allowed acquiring a 4D data set with a spatial resolution of 78 × 78 × 78 μm^3 within less than 2 h. Each excitation gave a sequence of eight echoes at times linearly spaced between 2.14 and 28.53 ms. The signal relaxed back to equilibrium with a repetition time of 900 ms. Two signal accumulations were performed resulting in a total scan time of 92 min. The sample temperature was regulated to 25 ± 0.1 °C.

The acquired complex data was converted to a sequence of 3D images by Fourier transformation and magnitude calculation. The image intensity $y(t)$ as a function of echo time t is given by

$$y(t) = I_0 \exp\left(-\frac{t}{T_2^*}\right),$$

where I_0 is the signal intensity at $t = 0$. For each volume element in the image, the values of I_0 and T_2^* were estimated by regressing the equation above onto the experimental data. Processing and fitting routines were done with Matlab (Mathworks, USA) and the magnitude noise was treated as described in Appendix A. The monoexponential decay in the equation above was not consistent with the signal intensities obtained at the very shortest echo time and these points were consequently

Table 1
Major composition of analyzed spaghetti samples (% dry matter).

Components	DS	DS + FB	DS + WG	DS + SW
	Durum semolina	Durum semolina and wheat fiber	Durum semolina and durum whole grain flour	Durum semolina and soft wheat flour
Carbohydrate	77.5	75.9	74.3	80.5
Protein	15.8	14.8	15.5	14.0
Fat	2.1	1.9	2.2	1.7
Dietary fiber	3.5	6.3	6.6	3.1