Effect of processing conditions on water mobility and cooking quality of gluten-free pasta. A Magnetic Resonance Imaging study

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Abstract

A new approach for producing gluten-free pasta from hydrated (50 °C, 20 min) rice kernels, skipping the grinding step, was explored. Magnetic Resonance Imaging (MRI) was used to study the hydration kinetics of rice, by monitoring the time evolution of both proton density and water transverse-relaxation rate during water diffusion. Results showed that the optimal water diffusion was reached after 180 min, allowing the extrusion of hydrated rice kernels into pasta. MRI analysis also highlighted in cooked pasta gradients of water distribution and mobility, in agreement with the high shear force that was measured using the Kramer cell (1066.5 vs 896.4 N). The high hydration in the external layers of pasta did not negatively affect the cooking quality (cooking loss, compression energy, firmness) of the product. MRI analysis provided an experimental evidence for the optimization of early steps in the technological process of grains for the production of gluten-free pasta.

Keywords: Magnetic resonance imaging; gluten-free pasta; rice kernel hydration; parboiling; cooking quality

1. Introduction

Gluten-free (GF) dried pasta can be mainly prepared using two approaches (Marti & Pagani 2013). The first one focuses on the use of pregelatinised GF raw materials (starches and/or flours), in which starch is already mostly gelatinized. Here, the pre-treated raw materials can be formed into pasta by the same extrusion press and conditions commonly used in durum wheat pasta plant. This way is surely characterised by low investment costs related to the technology but relatively high costs for the raw materials (Marti & Pagani 2013). In the second approach – namely cooking-extrusion process - native flour is treated with steam and extruded at high temperatures (more than 100 °C) for promoting a relevant gelatinization of starch granules directly inside the cooker-extruder. Cooking-extrusion of native rice did not seem to be efficacious in creating a continuous and smoothed starchy matrix, which instead resulted in the disruption of the surface structure during cooking and thus in low firmness (Marti, Caramanico, Bottega & Pagani, 2013). On the contrary, the extrusion of a pre-treated flour (i.e. parboiled rice) in the cooking-extruder induced a new starch organization, assuring a good texture in the cooked product and avoiding additives (Marti, Seetharaman & Pagani, 2010). In addition to the technological aspects, parboiled rice resulted in improved nutritional properties compared to milled rice. Indeed, during the process part of vitamins and minerals migrate towards the endosperm (Bhattacharya, 2004), and resistant starch is formed (Casiraghi, Brighenti, Pellegrini, Leopardi & Testolin, 1993).

The role of formulation and processing conditions on structural and cooking quality (Mariotti, Iametti, Cappa, Rasmussen & Lucisano, 2011; Lucisano, Cappa, Fongaro & Mariotti, 2012; Marti, Barbiroli, Marengo, Fongaro, Iametti & Pagani, 2014) and nutritional properties (Marti, Abbasi Parizad, Marengo, Erba, Pagani &

Casiraghi, 2017) have been recently addressed. In particular, differences in starch digestibility might be driven by differences in biopolymer interactions promoted during processing. At this regard, the effects of pasta-making processing on starch-starch (Marti et al., 2010; Marti, Pagani & Seetharaman, 2011a; Marti, Pagani & Seetharaman, 2011b) and starch-protein (Barbiroli, Bonomi, Casiraghi, Iametti, Pagani & Marti, 2013; Cabrera-Chavez et al., 2012) interactions in GF pasta has been investigated in previous studies. On the other hand, to the best of our knowledge no information on starch-water interaction is available in a non-gluten system. Thus, this study aims at assessing water mobility in GF pasta using Magnetic Resonance Imaging (MRI), that has succesfully been used in the past for semolina pasta (Horigane, Kawabuchi, Uchijima & Yoshida, 2009; Bonomi et al., 2012).

In addition, in the view of process simplification, this study proposes a new approach for producing GF pasta directly from parboiled rice kernels, after their hydration, skipping the grinding step. In fact, although the grinding of rice is a simple and easy step, it is inevitably associated with the formation of dust that can favor the infestation of pests and increase the risk of explosions (Posner & Hibbs, 2005). MRI was used to assess kernel hydration properties useful to choose the processing conditions of pasta production. Finally, the effect of processing conditions on cooking behavior, starch properties and water mobility of cooked pasta was also investigated.

2. Materials and Methods

2.1 Raw materials

Indica rice (*Oryza sativa* L.; starch: 85%; amylose: 25 g/100 g total starch) of commercial origin was used in this study. Parboiled milled rice kernels - henceforth RK – and the related flour - henceforth RF - were kindly provided by Riso Viazzo s.r.l., (Crova, Italy). Starch (80.9 g in 100 g dry matter), protein (10.7 g in 100 g dry matter), lipids (0.4 g in 100 g dry matter), and ash (0.9 g in 100 g dry matter) content of RK were determined using the standard AACC methods (AACCI, 2001).

2.2 Pasta samples

Pasta (20 kg) from RF - henceforth PaRF - was prepared as reported by Marti et al. (2010), with little modification (Fig. S1). The mixture of flour and water (with a final water content of 40%) was extruded in a Progel® extruder (single screw; Braibanti, Milano, Italy) fed with steam at 150 kg/h and 130 °C. After the first extrusion step, the pellets from heat-treated dough were formed in a macaroni shape in the continuous extruder (45 °C) used for the conventional process (Braibanti, Milano, Italy).

RK were soaked in tap water for 20 min at 50 °C (kernel:water ratio = 1:1.5). After removing the excess of water, the kernels were tempered for 3 hours at 22 °C keeping the kernels in sealed containers to avoid moisture changes. Rice kernels were then formed into pasta - henceforth PaRK - as described above. Both pasta samples (PaRF and PaRK) were dried in an experimental drying cell (Braibanti, Milan, Italy) using a low-temperature drying cycle (50 °C for 14 hours) and stored at room temperature until analyzed. Before the analysis, rice pasta samples were ground obtaining a product with less than 500 μm particle size.

2.3 Magnetic Resonance Imaging (MRI)

MRI experiments were carried out on both rice kernels after their hydration and cooked pasta.

Five grams of rice grains were soaked at 50 °C in distilled water (kernel:water ratio = 1:1.5). After 20 min of soaking, grains were removed, drained for 1 min and blotted with filter paper to remove any excess water attached to the surface. The blotted grains were inserted into NMR tubes (diameter: 10 mm) for MRI experiments that were performed up to 265 min of tempering time.

MRI studies of rice kernels and cooked pasta were carried out using a standard bore Bruker Avance AV600 spectrometer (Bruker Biospin GmbH, Rheinstetten, Germany) equipped with a 10 mm ^{1}H micro-imaging probe and a variable temperature control unit. The magnetic field strength was 14 T, corresponding to a ^{1}H resonance frequency of 600.1 MHz. A series of MRI slices of rice/pasta samples was taken at room temperature with the following acquisition parameters: MSME (Multi Slice Multi Echo) acquisition; number of slices from image: 8; thickness: 0.3 mm; no slice separation; repetition time: 800 ms; echo time 3.2 ms; number of echoes: 8; number of scans: 8; total acquisition time: 15min. Field of view was set to 9×9 mm^2, with a matrix size 128×128 units, corresponding to an in-plane resolution of 70×70 μm^2. For each slice, T_2 values were extracted by a multi-parametric non-linear fitting ($y=A+B*e^{-t/T2}$) of the intensity decays and used for reconstruction of the corresponding parametric images. Acquisition, data processing and image analysis were performed with ParaVision v.4.0 (Bruker BioSpin MRI GmbH, Ettlingen, Germany). Non-linear least squares fitting analysis of MRI data was performed with OriginPron 2016 (OriginLab Corporation, Northampton, MA, USA).

2.4 Pasta quality indices

A colorimeter (CR 210, Minolta Co., Osaka, Japan) was used to measure the lightness (L*) and saturation of the color intensity value (a*, redness-greenness; b*, yellowness-blueness) of flours, using the CIE l*a*b* color scale. The measurement was replicated five times and the average value was used.

Cooking behavior was evaluated by determining the grams of solid lost into cooking water for 100 g of dry pasta, at a pasta:water ratio = 1:10 with no salt addition. After cooking, pasta was drained, water was brought back to the initial volume, and an aliquot was dried to constant weight at 105°C. Weight increase of pasta due to water absorption during cooking was evaluated gravimetrically.

Texture measurements were carried out using a Texture Z005 (Zwick Roell, Ulm, Germany), equipped with a Kramer Shear Cell. An aliquot of pasta (25 g) was cooked in 250 ml distilled water till the optimal cooking time (OCT). After cooking, pasta was drained for 60 s, cooled under water for 30 s and drained one more time for 60 s. Pasta samples were analyzed after 10 min of rest at room temperature in a sealed container. Firmness (N, expressed as the maximum force necessary to pack the sample), compression energy (N*mm, corresponding to the area of the curve until the maximum force), and shear force (N, expressed as the force necessary so that blades pass through the sample) were automatically calculated by the software provided with the instrument. Average data of 10 replicates for each sample were reported.

2.5 Micro-Visco-AmyloGraph (MVAG)

Pasting properties were measured in duplicate in a Micro-Visco-AmyloGraph device (Brabender, Duisburg, Germany), on samples ground to particles smaller than 0.5 mm. An aliquot of 15 g of the sample was dispersed in 100 mL of distilled water, scaling both flour and water weight on a 14% flour moisture basis. The pasting properties were evaluated under constant conditions (speed: 250 rpm by using the following temperature profile: heating from 50 °C up to 95 °C; holding at 95 °C for 30 min; cooling from 95 °C to 50 °C; holding at 50 °C for 30 min, and cooling from 50 °C to 30 °C). The heating and cooling phases were carried out with a temperature gradient of 3 C/min. Data were elaborated as reported by Mariotti et al. (2011).

2.6 Statistical analysis

T-test was used for comparing the average of pasta samples for color and cooking bahavior. Analysis of variance (ANOVA) was performed for MVAG indices, utilizing Statgraphics XV version 15.1.02 (StatPoint Inc., Warrenton, VA, USA). Samples were used as a factor. When the factor effect was found to be significant ($p \leq 0.05$), significant differences among the respective means were determined using Fisher's Least Significant Difference (LSD) test.

3. Results and Discussion

3.1 Water distribution in rice kernels before and after hydration by MRI

MRI was used to determine the optimal tempering time to allow homogeneous water distribution in RK before pasta processing. Indeed, this technique directly detects the protons of the water molecules and generates images with good spatial resolution. The relative intensities of the pixels can be rendered quantitatively reliable as long as appropriate protocols are used to acquire and process the data (Takeuchi, Maeda, Gomi, Fukuoka, & Watanabe, 1997).

To date, several MRI investigations have been applied to rice and other grains to monitor various changes in the physical structure and moisture distribution during or after cooking (Gruwel, Chatson, Yin & Abrams, 2001; Hong et al., 2009). Moreover, MRI technique has successfully been used to discriminate between free and bound water, and to assess meanwhile the total amount of water present in foods, including pasta (Takeuchi et al., 1997; Horigane et al., 2009; Bonomi et al. 2012).

Fig. 1 shows the water distribution in parboiled rice, measured in 0.3 mm slices from the image, as function of the resting time of soaked kernels in water for 20 min. In these images, each MRI pixel intensity is proportional to the water content of the corresponding rice voxel. The graduation in the grayscale intensities seen within the rice grains reveals that water is not evenly distributed. Initially, it is mainly present in an outer layer of the rice kernel. Then, water slowly diffuses into the core, reaching an equilibrium state after at least 3 hours, where it appeared to be homogeneously distributed and the distinction between outer and inner layers is no more apparent.

As the MRI images were taken from a "multi-slice-multi-echo" experiment, it was possible to derive the distribution of the water transverse relaxation rate (T_2) within each slice, as depicted by the parametric image of Fig. 2. As it has been shown elsewhere (Takeuchi et al., 1997), T_2 values are directly related to water mobility at a molecular level (Carini, Curti, Littardi, Luzzini, & Vittadini, 2013). In our rice samples, values span in the interval between 10ms (blue pixels) and 50ms (red pixels). Such values are typical for water molecules hydrogen-bonded to macromolecular matrix, whereas values greater than 100 ms are in general more related to freely diffusible water.

A comparison between the two MRI images in Fig. 1 and 2 proves that there is an initial external layer of tightly bound water, arising from the macromolecular network, mainly composed by amylose and amylopectin, hydrated during the parboiling process (blue regions). Such regions do not change their state during the diffusion process. By contrast, inner regions initially define a not-yet-hydrated core with a low proton density. At the interface with the external layer, some residual water is evident with a mobility even higher than the external layer (pixels in green to red color, $T2>30ms$), corresponding to mobile water not entrapped within the starch crystallites. As water diffuses from the outer regions into the rice kernel core, the low-hydration regions reduce in size and are gradually replaced by such higher mobility water regions.

MRI images thus provide an experimental basis for a quantitative determination of the kinetics of hydration pathways. The MRI proton density images were quantified by measuring the integrated pixel intensity of the inner region in two rice kernels, chosen according to their orientation within a single slice (long or short axis; see Fig. 1), while the T_2 parametric images were analyzed by pixel count of the blue channel in the RGB image ($T_2 < 10$ ms).

As shown in Fig. 3A, hydration, measured from the MRI proton density images, might be modelled by a 1st order kinetics, according to the following mono-exponential equation:

$$I = I_0 + I_1(1 - e^{-kt})$$

Where I is the integrated intensity of the MRI area under examination, I_0 and I_1 are the initial and final integrated intensities, respectively, and k is the 1st order kinetics time constant of water diffusion. The parameters derived by a non-linear least-squares fit analysis depend on the section of the rice kernel taken into consideration: (direction 1, long axis) I_0= 13.1±0.8%; I_1= 24.6±0.8%; k= 0.010±0.001 min^{-1}; (direction 2, short axis) I_0= 3.2±0.5%; I_1= 9.3±0.5%; k= 0.015±0.002. The time constants are the most interesting parameters, as they can directly be related to the water diffusion process. On the other hand, water mobility (see Fig. 3B) follows a different time course, as it proceeds with a sigmoidal-shape plot, reminiscent of a cooperative behavior. The time course might be modelled according to the following equation:

$$A = A_0 + (A_0 - A_1)/(1 + 10^{(c-t)^\wedge p})$$

The following parameters were thus derived: A_0 (bottom asymptote) = 63.6±0.9%; A_1 (top asymptote) = 96.2±0.9%; c (center)= 157.7±2.2 min; p (Hill slope) = 0.073± 0.02. Actually, an abrupt change in water mobility occurs between 148 min and 170 min. At 180 min water mobility can be considered completely stabilized in a tightly bound status up to a 96% level. For this reason, the optimal resting time for rice kernel soaking was considered to be at least 180 min.

3.2 Pasta Quality

Pictures of pasta samples are reported in Fig. S1. From a qualitative standpoint, both samples appeared omogeneous and appealing in terms of color and structure. From a quantitative standpoint, pasta-making process did not affect the products luminosity (Table 1), whose values were similar to pasta from durum wheat semolina (Marti et al., 2013). PaRF and PaRK significantly differed for both $a*$ and $b*$ values, with PaRK exhibiting higher redness and lower yellowness than PaRF. Differences in color might be due to the soaking step. Loss of pigment into the soaking water should be taken into consideration, as shown in the study of Lamberts, Brijs, Mohamed, Verhelst & Delcour (2006). Both pasta samples presented lower luminosity and higher yellowness than commercial 100% rice pasta (Marti et al., 2013). This result is due to the use of parboiled rice as raw material. As well-known, the darker and more yellow color after parboiling is a consequence of the migration of pigments from the husk and/or bran to the endosperm (Bhattacharya & Ali, 1985) and non-enzymatic browning (Dendy, 2000). The impact of different hydration kinetics in flour vs kernels might also play a role in color differences.

No differences in cooking time were detected between the samples (Table 1). This is valid also for leaching into cooking water and water absorption, which values were not significantly different between PaRF and PaRK (Table 1). Because of the lack of a gluten network in all gluten free pasta, starch polymers were less efficaciously entrapped in the matrix in comparison with semolina pasta, resulting in a product with a high cooking loss, even three-four times more than that of the semolina sample (Marti et al., 2013). A decrease in starch leaching during cooking can be promoted by the adding texturing proteins (Marti et al., 2014) or emulsifiers (Lai, 2002).

As regards textural properties, no significant differences ($p<0.05$) were measured in terms of firmness and compression energy. On the other hand, PaRK exhibited a significant ($p<0.05$) higher shear force than PaRF. Processing conditions (e.g. extrusion temperature) adopted during the extrusion-cooking step should be optimized in order to decrease the extreme firmness of the product. Indeed, experimental pasta samples exhibited firmness values even two-fold higher than those of commercial semolina pasta (data not shown).

3.3 Pasting Properties

The MVAG test is widely used for evaluating the changes in viscosity of cereal flours or starches during heating and cooling. This approach has also been successfully used on conventional pasta to assess the contribution of macromolecules interactions and of temperature-dependent structural changes on finely ground pasta (Bonomi et al., 2012; Marti et al., 2014). It has been also used on non-conventional pasta, providing information on molecular changes promoted by pasta-making process (Marti et al., 2010; Marti et al., 2013) and/or ingredients (Marti, Pagani & Seetharaman, 2011c; Cabrera-Chavez et al., 2012). The pasting properties of rice flour and pasta samples are shown in Fig. S2 and the related indices in Table 1. Pasting temperature was lower in PaRK then PaRF, likely indicating that the extrusion step was more effective in starch gelatinization in hydrated rice kernels compared to flour, probably due to the high water avalaibility in soaked kernels. Indeed, the higher the degree of gelatinization, the lower the pasting temperature (Marti et al., 2013). Moreover, PaRK showed a higher maximum viscosity then PaRF (Table 1) suggesting greater starch swelling ability, likely related to faster hydration and water absorption during cooking (data not shown). Finally, during cooling at 30°C, PaRK exhibited higher setback values suggesting greater retrogradation tendency, likely explaining the high shear force of the cooked product (Table 1). Our results confirmed previous studies that stated that the setback value was positively related with the firmness of rice noodles (Bhattacharya, Zee & Corke, 1999) and rice pasta (Marti et al., 2010).

3.2 Water distribution in rice pasta by MRI

The same kind of MRI measurements used for monitoring grain hydration kinetics during soaking were then performed on the two kinds of experimental rice pasta PaRF and PaRK, in order to ascertain whether differences in the processing of rice kernels might affect the quality of the obtained pasta.

Fig. 4 shows both the water distribution and mobility in the two pasta samples after cooking at optimal time (15 min). Pasta samples exhibited some differences, especially at the surface layer, where the PaRK sample seems to be more hydrated then ParRF. Differences in water mobility were confirmed by measuring the spin–spin relaxation time (Fig. 5). Both pasta types present a non-uniform dispersion of T_2-values, indicating the presence of at least three distinct regions with different water mobilities: (a) low water mobility (T_2 = *ca.* 15 ms); (b) intermediate water mobility (T_2 = 25 ms); (c) high-mobility water (T_2 = 30 ms). In particular, group (a) is equally present in both PaRF and PaRK samples, whereas in the PaRK sample there is an evident shift in population from group "b" to group "c" (high-mobility), consistent with the increase in hydration observed in the external layer of this kind of pasta.

Gradients of water distribution and mobility in conventional pasta from durum wheat semolina have been positively related to the ''al dente'' feeling in cooked pasta (Horigane et al., 2009; Bonomi et al., 2012). This result is in agreement with the higher shear force measured in PaRK compared to PaRF (Table 1).

4. Conclusions

In this paper we have shown that MRI is able to provide an experimental basis for the quantitative analysis of the kinetics of hydration pathways, by monitoring the time evolution of both proton density and water transverse-relaxation rate during moisture diffusion in non-spherically shaped samples, such as the parboiled rice kernels. The MRI analysis thus provided an experimental evidence for the optimization of important early steps in the technological process of parboiled rice for the production of rice flour-based pasta.

Results on the overall pasta quality suggest that it is possible to prepare rice pasta directly from the extrusion of parboiled rice kernels previously hydrated and maintained moistened for at least 3 hours before processing, as suggested by the water status of soaked kernels during the resting phase. The cooking behavior was not negatively affected by the processing, suggesting that this new process might be applied to other raw materials, and represents a simplification of the pasta-making process from raw materials different from wheat flour and/or semolina. The consequent economical benefits could be of great interest especially in developing Countries. Further studies will focus on the optimization of parboiling processing with the aim of decreasing the time required for kernels hydration (3 h), to make the process more suitable for industrial scale-up.

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Conflict of interest

There is no conflict of interest.

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Captions

Fig. 1. MRI images (proton density) of parboiled rice at increasing time after initial soaking in water at 50°C. Labels 1 and 2 (in the image at time = 20 min) indicate rice kernels considered for further numerical analysis.

Fig. 2. Parametric MRI images (T_2) of parboiled rice measured at increasing times after initial soaking in water. T_2 values are color-coded from blue (T_2= 5ms) to red (T_2=53ms). Hydration times (min) are shown on top. The rice kernel is the same as that one labelled "1" in Fig. 1

Fig. 3. A: Time course of MRI signal along the two principal directions, 1 (long axis) a0nd 2 (short axis), in selected slices of individual rice kernels (labelled 1 and 2 in Fig. 1. B: Time course of the area containing tightly bound water (T_2<15ms) in a selected rice kernel. Data were collected from the slice depicted in Fig. 2. Continuous lines are drawn from a non-linear least squares fitting analysis.

Fig. 4. MRI images on selected slices (0.3 mm thickness) of PaRF (left) and ParK (right) pasta samples after 15 min cooking. Top: proton density image, color-coded from blue (low signal) to red (high signal); bottom: T_2 distribution (parametric image). T_2 values are color-coded from blue (T_2= 5ms) to red (T_2=53ms).

Fig. 5. T_2 distribution (pixel count) of the MRI T_2 parametric images shown in Fig.6 for PaRF (left) and PaRK (right) cooked pasta samples. Letters a, b and c denote the three main regions with different water mobility.

Supplementary material

Fig. S1. Processing conditions for experimental rice pasta-making from rice kernels and rice flour.

Fig. S2. Pasting properties of PaRF (full line) and PaRK (black dashed line). Temperature profile in grey full line.

PaRF, gluten-free pasta prepared from parboiled rice flour

PaRK, gluten-free pasta prepared directly from from parboiled rice kernels

Table 1. Pasta color, cooking behavior, and pasting properties

		PaRF	PaRK
Color	Luminosity (*L**)	80.9 ± 0.44	81.0 ± 0.32
	Redness (*a**)	0.30 ± 0.09+	0.48 ± 0.11
	Yellowness (*b**)	15.1 ± 0.70+	13.6 ± 0.26
Optimal Cooking Time (min)		15	15
Water Absorption (g/100g)		95.4 ± 5.4	91.9 ± 4.2
Cooking losses (g/100g)		12.0 ± 3.6	11.1 ± 2.8
Texture	Firmness (N)	1337.8 ± 144.6	1431.7 ± 134.9
	Compression Energy (N*mm)	5256.0 ± 521.4	5403.7 ± 599.8
	Shear Force (N)	896.4 ± 31.0+	1066.5 ± 36.5
Pasting properties	Pasting Temperature (°C)	62.4 ± 0.30+	55.6 ± 0.01
	Maximum Viscosity (BU)	176.5 ± 5.5+	258.5 ± 6.5
	Partial Setback (BU)	184.5 ± 0.71+	286.0 ± 11.3
	Final Setback (BU)	246.0 ± 8.5+	364.0 ± 4.2

+ significantly differences (t-test, p<0.001)

Pasting temperature, temperature at which an initial increase in viscosity occurs.

Maximum viscosity, viscosity achieved at 95 °C.

Partial Setback, difference between the viscosity at 50 °C and the viscosity reached after the holding period at 95 °C).

Final Setback viscosity, difference between the final viscosity at 30 °C and the viscosity reached at 50 °C).

PaRF, gluten-free pasta prepared from rice flour; PaRK, gluten-free pasta prepared directly from from rice kernels

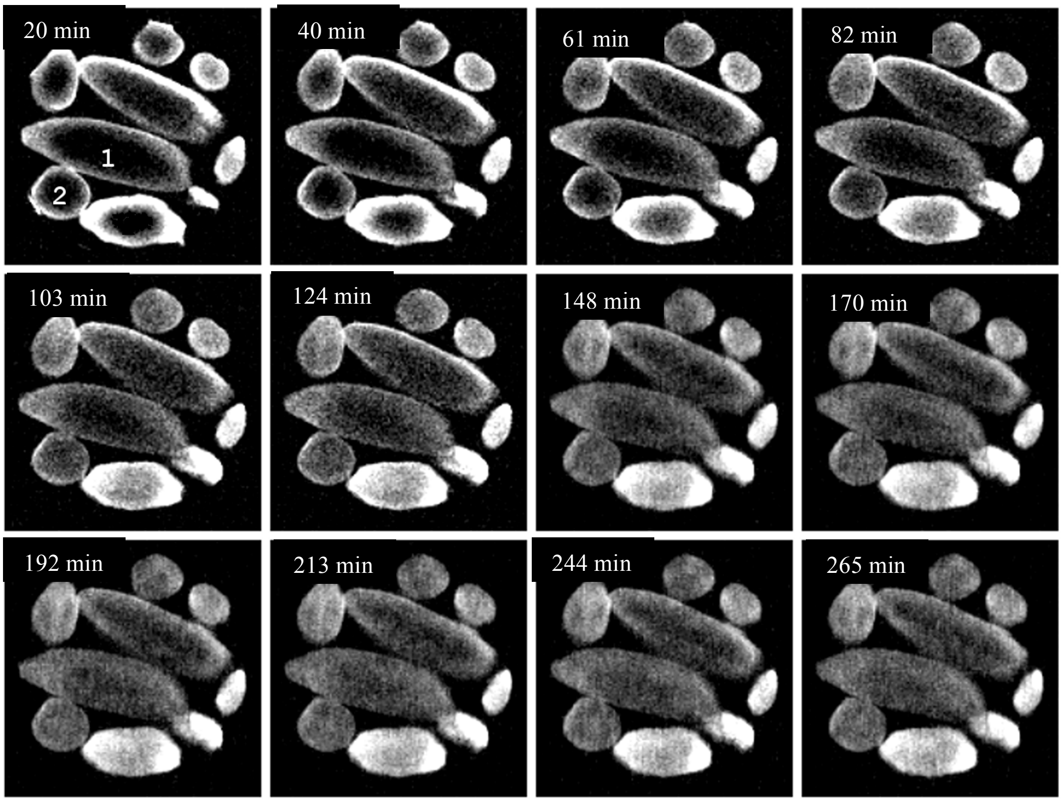
20 min
1
2
40 min
61 min
82 min
103 min
124 min
148 min
170 min
192 min
213 min
244 min
265 min

Fig. 1.

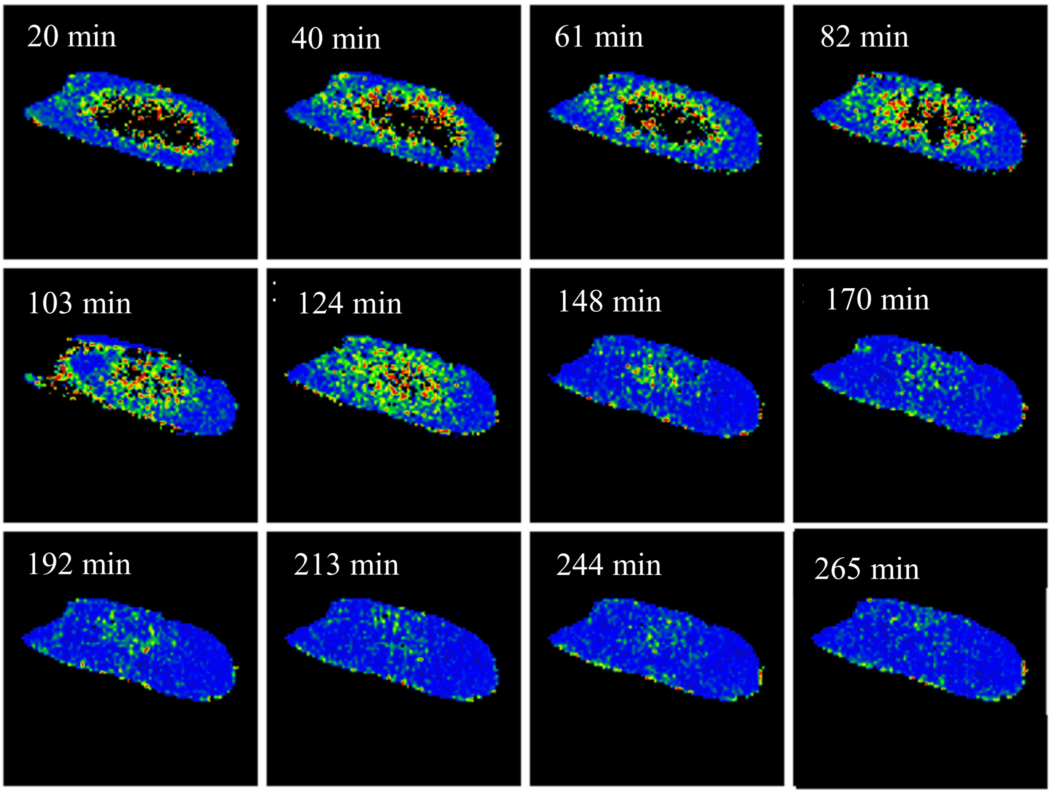
20 min
40 min
61 min
82 min
103 min
124 min
148 min
170 min
192 min
213 min
244 min
265 min

Fig. 2.

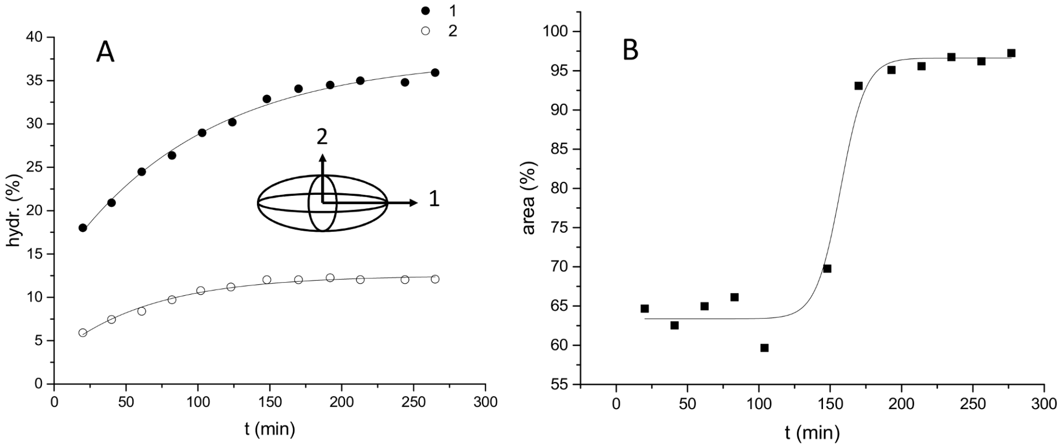

Fig. 3.

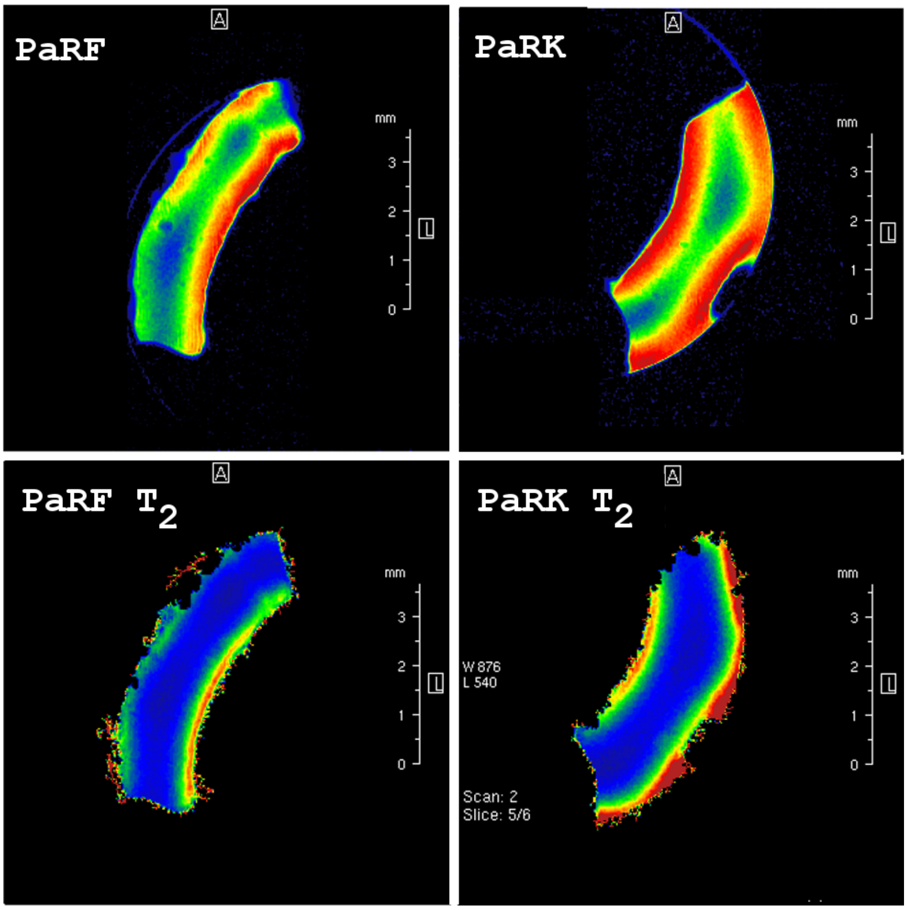

Fig. 4.

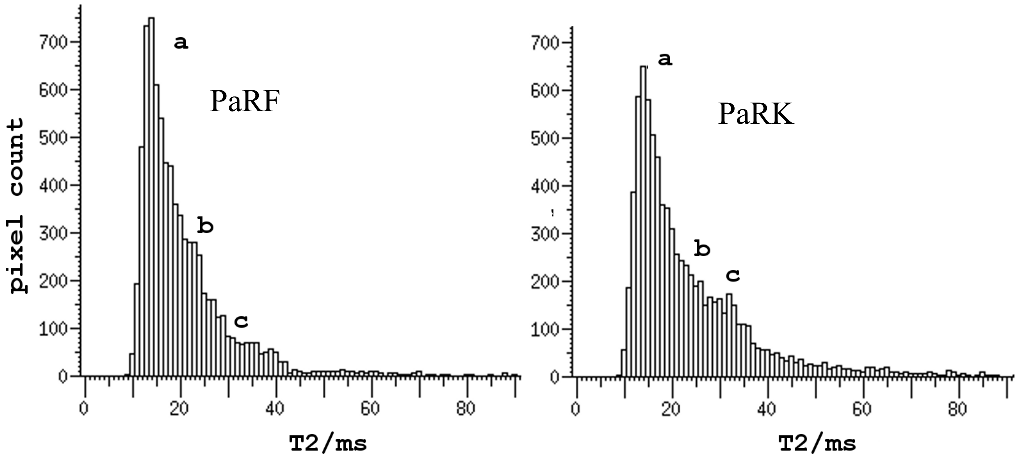
PaRF
PaRK
a
b
c
pixel count
T2/ms
0
20
40
60
80
100
200
300
400
500
600
700

Fig. 5.