

# Assessment of water diffusivity in gelatine gel from moisture profiles. I—Non-destructive measurement of 1D moisture profiles during drying from 2D nuclear magnetic resonance images

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## Abstract

A method to assess the relationship between water diffusivity and water content was developed. This method combines the non-destructive measurement of water distribution in sample by NMR imaging during isothermal drying process and data treatment for shrinking/swelling materials and which needs no a priori on the relationship  $D=f(X)$ .

Gelatine gel samples (60 bloom) were used to test the method. Their initial moisture contents were between 1.8 and 5 kg water/kg dry basis, pH between 4 and 8 and temperature in the range of 10 and 24 °C.

This first part describes the experiments. A specific assembly adapted to the small microimaging probehead was built to promote isothermal and unidirectional drying of the samples. Successive steps were developed to calculate local moisture contents from independent calibration and sources of error were evaluated. It was shown that 2D NMR signal amplitude images must be recorded to derive the true 1D moisture content profiles although the time acquisition is longer. The time variation of 1D moisture content profiles with a spatial resolution of 49 µm and shrinkage was analysed.

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## 1. Introduction

Food characteristics are greatly influenced by moisture content. Product stability is of special concern because many chemical and enzymatic reaction rates (i.e., oxidation, browning) are water activity dependent.

Most of the processes used by the industry for food processing induce water transfer often coupled with heat transfer (i.e., chilling, freezing, cooking, drying, aging, salting, smoking). Such processes must be optimized to

reduce operating costs and maximize product quality. For instance, the energy needed to vaporize water in dryers is very high (Bimbenet, 1978) while in carcass chilling the water loss decreases product yield. Thus, the common 2% loss of initial carcass weight represents 60% of the operating costs (Daudin & Swain, 1990; Mallikarjunan & Mittal, 1994).

As regards safety, microbial growth on the surface of meat or cheese during the aging process depends on superficial water activity, which in turn is the result of a balance between water evaporation on the surface and internal water migration. Thus, accurate determination of the water diffusivity coefficient ( $D$ ) is important.

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## Nomenclature

$a_w$	water activity (ND)	$v$	air velocity (m/s)
$B_1$	radiofrequency field (T)	$X$	moisture content (kg water/kg dry basis)
$B_0$	static magnetic field (T)	$x$	abscissa in $Ox$ direction
$C$	volumetric moisture content (kg water/m <sup>3</sup> )	<i>Greek symbols</i>	
$D$	apparent water diffusivity (m <sup>2</sup> /s)	$\varepsilon$	gel shrinking coefficient (ND)
$k_v$	constant (number of nuclei/kg water)	$\rho_w$	water density (kg water/m <sup>3</sup> )
$P_a$	partial water vapor pressure in air (Pa)	$\rho_s^0$	solid phase density (kg dry matter/m <sup>3</sup> )
$P_w$	moisture content (kg water/kg wet basis)	$\rho_T$	gel density (kg total/m <sup>3</sup> )
$P_s$	water vapor pressure at the sample surface (Pa)	$\rho_v$	hydrogen nuclei density (number of nuclei/m <sup>3</sup> )
$S_v$	NMR signal intensity (arbitrary units)	$\phi_w$	drying rate (kg water/m <sup>2</sup> s)
$T_a$	air temperature (°C)	$\phi_r$	relative drying rate (ND)
$T_{bot}$	sample bottom temperature (°C)	<i>Subscripts</i>	
$T_E$	echo time (ms)	$o$	at time $t=0$
$T_h$	air wet bulb temperature (°C)	$t$	at time
$T_R$	repetition time (s)	$x$	at abscissa
$T_d$	air dew point temperature (°C)	$v$	in a voxel
$T_s$	sample surface temperature (°C)		
$T_1$	longitudinal, or spin–lattice, relaxation time (s)		
$T_2$	transverse, or spin–spin, relaxation time spin–spin (ms)		

For biological products,  $D$  is known to be very low and to vary with moisture content ( $X$ ). However, the relationship between these two variables:  $D=f(X)$  is still not well defined. Zogzas, Maroulis, and Marinos-Kouris (1996) reported wide variations in published data. Differences in product composition and structure must be considered the primary sources of variation but experimental techniques and data analysis are additional sources of variation.

Three main methods have been used to assess water diffusivity: drying kinetics, sorption or desorption kinetics, and moisture profile analysis (Roques, 1987).

In most studies the water flux exchanged between air and sample is determined from air drying weight loss kinetics (Alzamora & Chirife, 1980; Gou, Mulet, Comaposada, Benedito, & Arnau, 1996) and parameter estimates of  $D=f(X)$  in a mathematical model are obtained by fitting the model to the data. Published results vary widely probably because of differences in the fitted models or use of over simplified models limited to describe the physical phenomena involved. The sorption or desorption kinetics method consists in recording the weight changes of a sample placed in a cell where temperature and water vapour pressure are controlled until equilibrium is reached (Crank, 1975). It is similar to the drying kinetics method except that the sample temperature is constant and the water flux exchanged is much lower.

$D=f(X)$  can be advantageously derived from the time course of the moisture profile which is more sensitive to

this function than the weight loss. However, the measurement of moisture profiles inside biological materials is difficult because moisture gradients are steep and because the product shrinks with water removal. Litchfield and Okos (1992) and Andrieu and Stamatopoulos (1988) measured the water content in slices of pastas by oven drying but the accuracy of this destructive technique is limited. The main drawbacks are: (1) it is difficult to cut slices orthogonal to the direction of water migration, (2) the spatial resolution is limited and (3) the time variation of the moisture profile is assessed from measurements on different samples. Thus, non-destructive techniques are preferred. Gamma ray absorption can be used to measure moisture distribution along one direction; for instance Chiang and Petersen (1987) measured 1D moisture profiles in parallelepipedic samples of apples with a spatial resolution of 2 mm. However, the most promising technique is NMR imaging which allows measurement of water proton density in tiny parallelepipedic volumes, or voxels, and therefore moisture distribution assessment in three dimensional space.

Gummerson et al. (1979) analyzed water movement inside building materials. Later, Maneval, McCarthy, and Whitaker (1990) and Hills, Wright, Wright, Carpenter, and Hall (1994) studied water transfer during drying of sands with different particle size distributions. NMR images have also been used to visualize water distribution in one, two or three directions during the drying or rehydration of various foodstuffs: apple (Perez, Kauten, & McCarthy, 1989; Verstreken, Van Hecke,

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