



Evolution of particle structure during water sorption observed on different size fractions of durum wheat semolina



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ABSTRACT

This work examines the effect of the particle size of durum wheat semolina on the water diffusion coefficient and the evolution of the microstructure during hydration. Durum wheat semolina was sieved in 5 size categories. Water–particle interaction of powders (5 fractions and raw semolina) was studied using dynamic vapour sorption. Water sorption isotherms were fitted to G.A.B. and Y&N models. Monolayer values as well as multilayer properties were calculated and compared. Absorption properties were determined by Y&N model. A sorption curve was modelled with the Fickian diffusion equation adapted to water diffusion into solid particles. A dynamic Fickian water diffusion coefficient was calculated for each particle size after conditioning at different RHs. The surface structure of conditioned fractions was observed by ESEM. The microstructure of semolina presented an evolution with the increase of size and RH. Starch grains presented an increased size and protein matrix had a different texture. After correlation of isotherm data and microstructure, the resulting state and properties of the components were a key to understand diffusion on food powder systems.

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1. Introduction

Durum wheat flour (semolina) is a raw material widely used in several processes such as the production of pasta or couscous. It is necessary to understand water transfer in these processes and during the storage of semolina to increase production efficiency and obtain quality products. This transfer can be limited by the surface properties of the particles, which significantly influences a number of functional properties, i.e. hydration, caking, agglomeration. Consequently the study of water transfer on the surface of particles is of relevance.

Water transfer using sorption isotherms of soft flours has been studied [1–3], only a few publications corresponding to semolina are available [4–6]. In addition there are few studies about water diffusion coefficients in flour particles [5,7].

The evolution of the microstructure with increasing water content may produce an increase of particle size as well as changes in the properties of components, thus diffusion can be affected. In this regard, the microstructural changes during the hydration of food powders have a great scientific interest for many practical applications. For example, during milk reconstitution, the water transfer into the milk particles depends on the succession of the different reconstitution steps:

wettability, sinkability, dispersibility and finally solubility of surface molecules. These steps depend on the nature and composition of the powder. In this context, changes on microstructure due to water sorption were studied by Murrieta-Pazos et al. [8]. These authors conditioned milk powder samples at different RH and observed the microstructure by ESEM. Despite the interest of such evolution this approach has not been applied to flour particles. The aim of this work was to study the effect of particle size on the water diffusion coefficient and the microstructural evolution throughout hydration of durum wheat semolina.

2. Materials and methods

2.1. Materials

Industrial semolina (Panzani, Marseille, France) was sieved into different size grades in decreasing mesh size (0, 160, 250, 315, 400, 500 μm) from top to bottom (Retsch, Germany). The operating conditions were chosen after a kinetic study of the sieving process. For this 400 g of powder was placed on the first sieve and sieved for 12 h with amplitude of 40 on the scale of the apparatus. These conditions were optimized to enhance fine particle separation. The powder remaining on the sieves was collected producing 5 fractions with a range of diameters (0–160, 160–250, 250–315, 315–400, 400–500 μm). The original powder (raw powder) was also used in the subsequent experiments.

Three of the size fractions (0–160, 250–315, 400–500 μm) were *pseudo-equilibrated* under different relative humidity at 25 °C for seven days in five hermetic vessels containing saturated salt solutions

Abbreviations: BET, Brunauer–Emmett–Teller model; GAB model, Guggenheim–Anderson–de Boer model; RH, relative humidity; ESEM, environmental scanning electron microscopy; SE, secondary electron; GES, gaseous secondary electron; Y&N model, Young and Nelson model; IUPAC, International Union of Pure and Applied Chemistry.

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(Sigma-Aldrich) of known RH: 0.11 (LiCl); 0.54 Mg(NO₃)₂; 0.75 (NaCl); 0.85 (KCl) and 0.97 (K₂SO₄). In each vessel, one millilitre of toluene (recommended for non-fatty products) was placed in a separated recipient near to the samples in order to avoid the proliferation of microorganisms [9]. This process was performed to determine the change in size and microstructure of the particles under varying RH; however the fitting of the models as well as the calculation of diffusion coefficients was obtained using data from DVS, where humidity is truly equilibrated.

2.2. Bulk composition

Water content was measured by weight loss after drying at 105 °C. Ash content was also measured by weight loss after incineration at 550 °C. Total proteins were determined from the Kjeldahl technique. Extractable lipids were calculated according to the Soxhlet extraction method using a mix of 1:1 (v:v) of diethyl ether–petroleum ether as solvent. The sample mass of semolina was 5 g. The solvent–oil mixture was then evaporated using a rotary vacuum evaporator (Heidolph laborator 4000) for 5 h at 40 °C and 40 rpm to recover pure oil. The boiling flask without solvent was dried in an oven at 100 °C for 1 h and the fat percentage was calculated by difference of weight. Finally, the carbohydrate content was determined by difference of totals. Tests were triplicated.

2.3. Particle size

The particle size distributions were determined using a laser granulometer (Mastersizer 2000 Malvern Instruments, UK) in dry and liquid mode. The Mastersizer 2000 uses high-pressure air to disperse and feed dry powder through the laser ray where the dispersion is controlled by adjusting the air pressure. The instrument is also equipped with a liquid sample dispersion module that is suited for powders in suspension. After dispersion of the powder, the particles diffract the laser, and the diffraction patterns are detected and the particle size is calculated. Five measurements were conducted with each powder sample. Most of the samples were analysed by air dispersion at 3.5 bars. Liquid dispersion was used only for low-quantity samples, where for each measurement approximately 0.25 g of the powder was dispersed in 75 ml of ethanol (to obtain a good obscuration and to avoid particles superposition).

2.4. Microscopy ESEM

The powders were observed with a Field-Emission Environmental Scanning Electron Microscope (ESEM FEG) (XL30, FEI/Philips, Netherlands) operating at 20 kV. High vacuum mode was used for raw and sieved semolina; low vacuum mode (1.4 Torr) was used for powders at different RHs. Powders were spread onto a double-sided adhesive carbon disc fixed on a support. Raw and sieved semolina were coated with platinum by a Polaron sputter coater (SC7640) and observed with a secondary electron (SE) detector. Powders at different RH were directly observed with a gaseous secondary electron (GSE) detector.

2.5. Dynamic vapour sorption

Sorption isotherms of powders were obtained with a Surface Measurement System Automated Dynamic Vapour Sorption (DVS1000) equipped with a controlled atmosphere microbalance. The experiments were carried out at constant temperature (25 °C) with different RH values ranging between 0% and 95%. Approximately 100 mg of powder was loaded onto the quartz sample pan. First, the samples were dehydrated in the DVS chamber (RH = 0%) for 60 min, and then the samples were submitted to a 10 step hydration process. The process was performed with 9 RH increments of 10% and a last step at 95% RH.

The samples were considered to be at equilibrium when the value dm/dt (slope of the changing in mass with time) was set to be <0.005 mg min⁻¹ or equilibration time exceeded 300 min.

G.A.B. (Guggenheim, Anderson and de Boer) and Y&N (Young and Nelson) equations were used to model experimental data. The quality of fit was evaluated by a linear correlation coefficient (R²), where a value above 0.85 indicates an appropriate data model. Monolayer values as well as multilayer properties were calculated by GAB and Y&N models and then compared.

2.5.1. G.A.B.

G.A.B. model (Eq. (1)) is an extension of the Brunauer–Emmett–Teller (B.E.T.) equation. This model is widely used in food studies. It considers that the sorption heat of multilayers is different from the liquefaction heat. The equation was fitted to sorption isotherms from 0.1 to 0.8 a_w. In this equation (Eq. (1)), C is the Guggenheim constant, k is correcting constant involving multilayer properties and bulk liquid properties, X_w is the equilibrium moisture content expressed on dry matter (% dm), and X_m is the monolayer moisture content (% dm) [10].

$$X_w = \frac{X_m \cdot C \cdot k \cdot a_w}{[1 - k \cdot a_w] \cdot [1 + (C - 1) \cdot k \cdot a_w]} \quad (1)$$

2.5.2. Young and Nelson (Y&N)

Y&N is a model derived from the G.A.B. model, by considering that, in addition to surface binding forces, diffusional forces are also present, and these forces could become dominant when multi-molecular layers of water are present. In this case diffusional forces would be harder with the increment of water molecules at the surface. Finally, binding surface forces enable movement of water into the sample, which is the case for a lot of samples in food science. The experimental sorption and desorption data can be fitted to the following equations (Eqs. (2) and (3)):

$$M_S = A(\theta + \alpha) + B\varphi \quad (2)$$

$$M_D = A(\theta + \alpha) + B\theta RH_{\max} \quad (3)$$

M_S and M_D are equilibrium moisture contents for the respective cycle at each relative humidity, and RH_{max} is the maximum exposed relative humidity. A and B are defined as:

$$A = \frac{\rho_w V_{\text{ads}}}{D} \quad (4)$$

$$B = \frac{\rho_w V_{\text{abs}}}{D} \quad (5)$$

ρ_w is the density of water at the experimental temperature; D is the sample dry weight, and V_{ads} and V_{abs} the volumes of adsorbed and absorbed water [11].

The parameters θ, α and φ in Eqs. (2) and (3) are related to an E term through the following expressions:

$$\theta = \frac{RH}{RH + (1 - RH)E} \quad (6)$$

$$\alpha = -\frac{ERH}{E - (E - 1)RH} + \frac{E^2}{(E - 1)} \ln \frac{E - (E - 1)RH}{E} - (E + 1) \ln(1 - RH) \quad (7)$$

$$\varphi = \theta RH \quad (8)$$

$$E = e^{-\frac{q_1 - q_L}{k_B T}} \quad (9)$$

q₁ is the heat of adsorption of water bound to the surface of the sample, q_L is the heat of condensation of water molecules, k_B is Boltzmann's constant and T is the absolute temperature.

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