

Interactions of Water with Roasted and Ground Coffee in the Wetting Process Investigated by a Combination of Physical Determinations

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Three complementary techniques were used in this study to investigate the physical changes during wetting of roasted and ground coffee. Scanning electron microscopy (SEM) was found to provide indirect evidence of the presence of liquid water in the coffee particles. The effect of wetting on coffee closed porosity was studied by helium pycnometry, and finally, particle sizing was used to determine the swelling kinetics of coffee after wetting. Due to the solubilization of compounds, the presence of liquid water could be detected in the coffee cells by SEM. The technique was then used to investigate different water contents; for example, for roasted and ground coffee containing 1 g of water per gram of coffee on a dry basis, liquid water was present in cells only at the periphery of ~1.0 mm diameter particles. Coffee closed porosity decreased with increasing water content, as evidenced by pycnometry. For roasted and ground coffee containing 1 g of water per gram of coffee, results showed a closed porosity lower than 0.1 cm³/g (~20% of the closed porosity measured in dry particles). The decrease of closed porosity may be attributed to both (1) water filling cells' lumen and (2) plasticization of cell wall polymers, resulting in the matrix relaxation and increase of helium accessibility to the pores. Water binding to the matrix polymers was further investigated by calorimetric measurements. The integration of the endothermic peak of freezing water showed that ~0.15 g of water/g of coffee is nonfreezable water, that is, water bound to the matrix polymers. Finally, the use of particle sizing showed that the average volume of the coffee particles with 1 g of water/g of coffee increased by up to 20–23% at 10–15 min following wetting. Moisture diffusion coefficients in coffee particles [(~2–3) × 10⁻¹¹ m² s⁻¹] were approximated by fitting the swelling curves with a model of diffusion. The observed results may give information about homogeneity and the physical state of water in wetted roasted coffee and thus increase the understanding of the mechanisms of molecular mass transfer during extraction.

KEYWORDS: Roasted coffee; water; porosity; swelling

INTRODUCTION

Extraction is a key operation in the production of instant coffee, which allows soluble and volatile compounds to be recovered. To ensure a "100% coffee" claim for instant coffee, liquid water is the only allowed solvent (1).

Generally, in the investigations of coffee brewing mechanisms, authors directly measure the extraction rates of compounds of interest. Especially the rate of caffeine diffusion has been extensively studied as it is of importance in two fields: decaffeination processes and coffee brewing. The rate of diffusion of caffeine from ground *arabica* beans into distilled water was measured by Spiro and Selwood (2). The authors found three ways of demonstrating that caffeine diffusion within

the bean is the rate-determining step: (1) the low magnitude of the caffeine diffusion coefficient, ~2 × 10⁻¹⁰ m² s⁻¹ at 80 °C versus 2 × 10⁻⁹ m² s⁻¹ for free diffusion of caffeine in water, giving rise to a hindrance factor of 10; (2) its high activation energy, 32 kJ mol⁻¹; and (3) the diffusivity, which varied by a factor of <2 when the particle radius spanned a factor of >40. Analysis of the results suggested that water counterflow, coffee particle swelling, association of caffeine with other solubles, and physical constraints within the bean matrix all contribute to the low diffusion coefficient of caffeine inside the coffee particles. Diffusivity was found to be much higher for water extraction of caffeine from pre-swollen coffee beans (3–5). These studies are of particular interest and show the role of water in the physical behavior of the coffee matrix and in molecular mass transfer.

Variations in total soluble content as a function of time, temperature, grind size, and coffee-to-water ratio were studied

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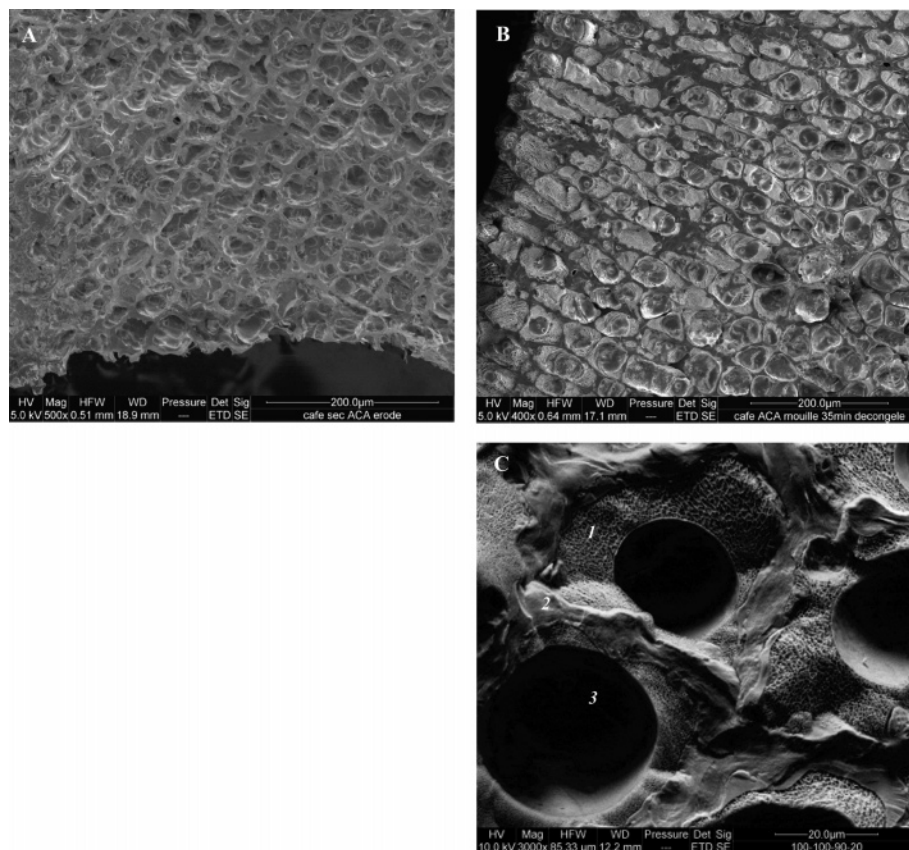


Figure 1. SEM micrographs of a dark-roasted dry coffee particle (A) and a particle immersed in water at ambient temperature for 30 min (B, C): 1, compounds solubilized by water and remaining inside the cells after water sublimation; 2, cell wall; 3, vacuoles due to entrapped gas bubbles.

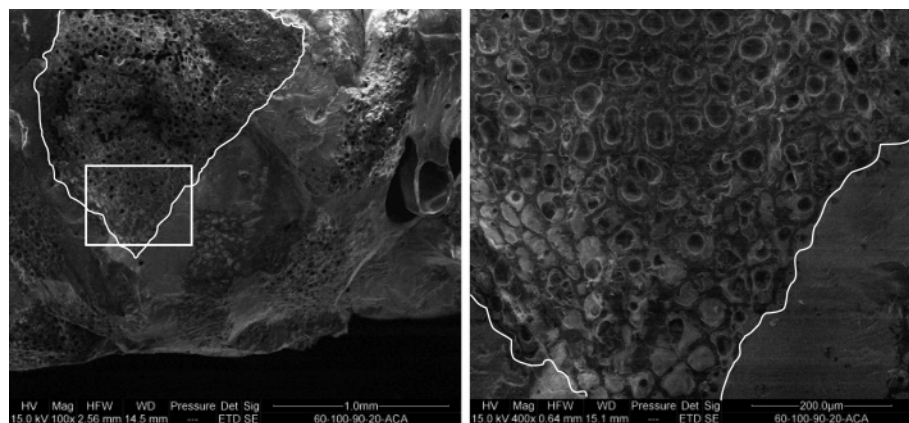


Figure 2. SEM micrographs of dark-roasted coffee particles after wetting with liquid water at 90 °C and stabilized at this temperature for 20 min, water content = 1 g/g of coffee, dry basis. The right micrograph is a higher magnification of the area delimited by the frame in the left micrograph. Whitish coloration of cells reveals the presence of water.

by Voilley and Simatos (6). The authors noticed a rapid increase of solids content during the first minutes of brewing followed by a stabilization after 5–10 min depending on the process conditions.

To conclude, the recovery of soluble compounds has been studied by several authors for brewing applications. However, only a few studies have been devoted to the investigation of water effects on the physical properties of coffee and on the water absorption kinetics of water itself into roasted coffee particles. Yet, a good understanding of water distribution would help to optimize the process of soluble coffee manufacture.

Water mobility in roasted coffee was investigated by some authors using ^1H NMR. Kino and Takagi (8) used NMR to determine free and bound water in coffee during the roasting

process and compared results with moisture distributions observed by MRI. The work by Mateus et al. (9) allowed characterizing the solid and mobile components of the coffee matrix. The authors attributed the observed mobile populations to water in cell wall polymers and to water filling the cell lumen. Furthermore, they observed an increase in the intensity of the mobile phase and a stabilization at ~ 10 min after wetting.

Even though NMR provides useful information about molecular mobility and interactions, it does not always give a clear distribution of water localization in complex matrixes.

Several authors studied the structure and porosity of dry roasted and ground coffee by scanning electron microscopy (SEM) (9, 10) or pycnometry (11, 12). To our knowledge, these techniques have not yet been used to evidence the role of water

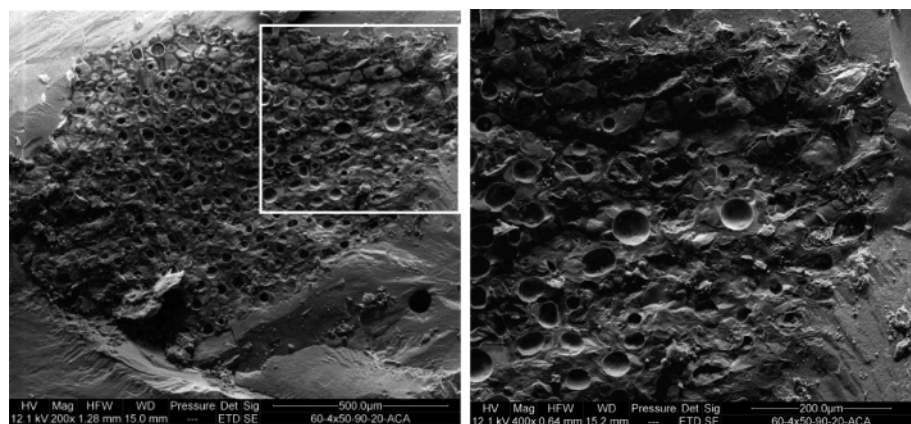


Figure 3. SEM micrographs of dark-roasted coffee particles after wetting with liquid water at 90 °C and stabilized at this temperature for 20 min, water content = 1.5 g/g of coffee, dry basis. The right micrograph is a higher magnification of the area delimited by the frame in the left micrograph.

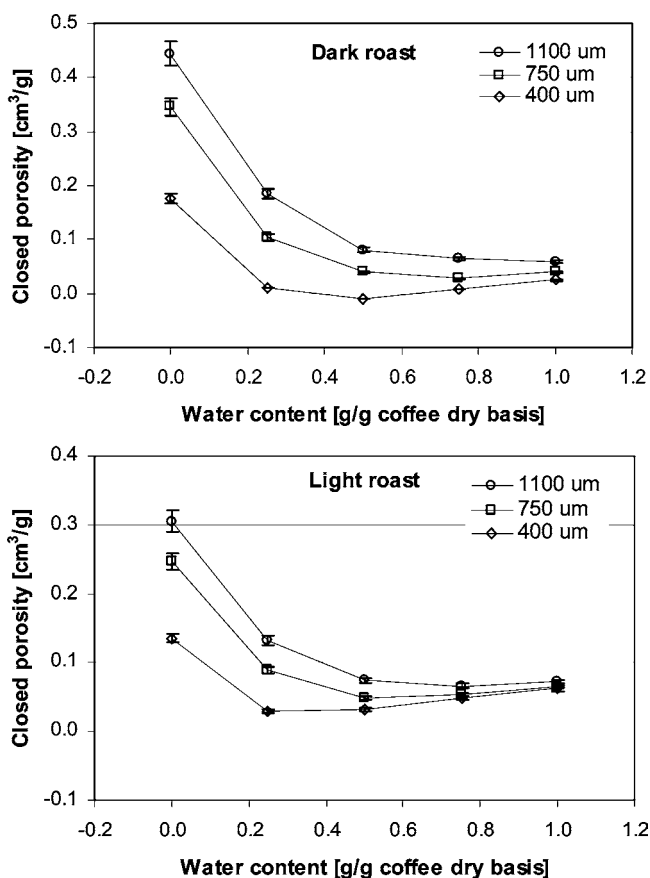


Figure 4. Coffee closed porosity as a function of water content for various particle sizes, as determined from volumetric measurements using helium pycnometry: (top) dark-roast coffee; (bottom) light-roast coffee.

in roasted coffee. Furthermore, studies on release kinetics of soluble compounds from roasted and ground coffee generally indicate that diffusion in the particles is the limiting transfer mechanism, as discussed previously. In the study of other systems during water imbibition, relaxation (mostly due to swelling) was reported to play a significant role in water and soluble mass transfer (13, 14). No data were found in the literature on the effects of coffee swelling during hydration. The objectives of this project were to determine the state of water and its role in the physical structure of the coffee matrix using SEM and helium (He) pycnometry. The effect of water content was assessed to understand interactions between the matrix components and water. Furthermore, the dynamics of water absorption was investigated using particle sizing.

EXPERIMENTAL PROCEDURES

Coffee Samples. Colombian *arabica* coffee was roasted in a fluidized bed hot air roaster (Neuhaus Neotec) to light-, medium-, and dark-roast degrees at 236 °C for 380, 550, and 900 s, respectively. Coffee was roasted in batches of 200 g of green coffee beans. The roasted beans were then ground (Ditting KFA 903 grinder) to 400, 750, and 1200 ± 50 μm particle mean volume diameter $D[4,3]$.

For the wetting process, 20 g of roasted and ground coffee was placed in a 200 mL glass beaker (65 mm diameter) and then wetted according to the following standardized procedure. Distilled water at 90 ± 2 °C was added manually using a graduated glass pipet, and the sample was stirred for 30 s with a stainless steel coffee spoon. The homogeneity of wetting was evaluated by weighing 20–50 mg of particles from six different parts of the samples. The water content of the particles was then determined gravimetrically after drying in an oven following the “methods of test for roasted and ground coffee: determination of moisture content (loss in mass at 103 °C) [routine method ISO 11294-1994 = BS 5752-1995 part 14; International Standard Organization (ISO)]. Experiments for the validation of wetting homogeneity were repeated for several coffee roast degrees and particle sizes. These controls showed that the chosen wetting procedure provided a homogeneous water distribution in the particles, with a relative standard deviation of <5%.

Coffee water content is expressed in grams per gram based on the dry weight of coffee.

Scanning Electron Microscopy (SEM). Coffee particles were mixed with Tissu-tek 45 83-OCT (Sakura Finetek Europe), and a portion of the mixture was deposited on a gold-coated copper cryospecimen holder. Sample freezing was done in nitrogen slush using an ALTO freezing station followed by a transfer under vacuum into a Gatan ALTO2500 cryosystem, with the cold stage at approximately -170 °C. The sample was then freeze-fractured by hitting it with a razor blade to reveal its internal structure and inserted in the microscope chamber, under high vacuum: $(4.0\text{--}5.3) \times 10^{-9}$ bar $[(3\text{--}4) \times 10^{-6}$ Torr]. The fractured specimens were slightly etched on the cryostage in the microscope (heating to -100 °C) and then stabilized at -115 °C. Visualization of samples was done without any coating in a Quanta 200 FEG (FEI Co.) operated at 15 kv in high-vacuum mode.

In preliminary trials, coffee particles were observed dry or after immersion in water at ambient temperature for 30 min. For the investigations of the effect of water content on the coffee matrix, the aforementioned wetting procedure was applied to the coffee samples before observation.

Helium Pycnometry. Closed porosity was determined from volumetric measurements using He pycnometry.

Samples of coffee wetted with water at 90 °C were allowed to stabilize and reach ambient temperature for 1 h before measurement; 20–25 g of wetted coffee was placed in the pycnometer measuring cell. The device used was an AccuPyc 1330 (Micromeritics) with a filling pressure of 1.35 bar (19.6 psi) and a working temperature of <35 °C.

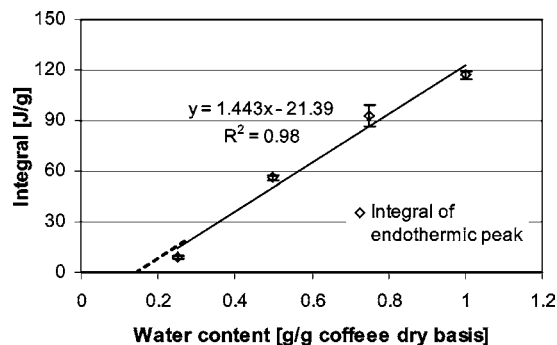


Figure 5. Integral of the endothermic peak of water melting in roasted and ground coffee after wetting at 90 °C as a function of water content and determined by DSC, medium-roast coffee.

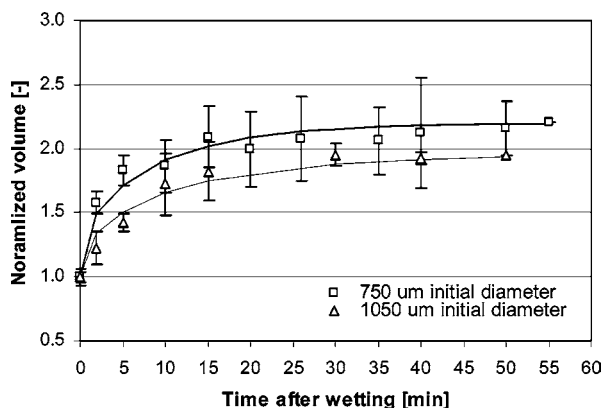


Figure 6. Normalized average volume of coffee particles after wetting at 90 °C with a water content of 1 g/g of coffee, dry basis, as determined using particle size analysis, medium-roast coffee: (□) initial volume of particles $\sim 750 \mu\text{m}$; (Δ) initial volume of particles $\sim 1050 \mu\text{m}$; (line) prediction of the volume using the model of diffusion in a sphere.

The specific volume (cm^3/g) of roasted and ground coffee was calculated knowing the volume (measured by the pycnometer) and the weight of the sample and is defined as

$$v_{\text{He}} = \frac{V_{\text{particles}}}{m_{\text{particles}}} = \frac{V_{\text{solids}} + V_{\text{closed pores}}}{m_{\text{particles}}}$$

where $V_{\text{particles}}$ (cm^3) is the total volume of the roasted and ground particles measured by the He pycnometer, $m_{\text{particles}}$ (g) is the mass of the sample, V_{solids} (cm^3) is the volume of the coffee polymers constituting the matrix, and $V_{\text{closed pores}}$ (cm^3) is the volume not accessible by helium.

V_{solids} was approximated by measuring the volume of $\sim 100 \mu\text{m}$ dry coffee particles, and $V_{\text{closed pores}}$ was assumed to be equal to 0 for $\sim 100 \mu\text{m}$ dry coffee particles. This assumption was made knowing, from microscopy analysis, that coffee porosity is primarily due to the presence of void intact cells in the particles. As the cell diameter ranges between ca. 20 and $50 \mu\text{m}$, it may be concluded that in $100 \mu\text{m}$ coffee particles, the proportion of intact cells is close to zero.

For wetted particles, the specific volume of coffee was calculated after subtraction of water specific volume.

Finally, closed porosity (cm^3/g) was deducted from values of specific volume

$$\epsilon_{\text{closed pores}} = v_{\text{He}} - v_{\text{solids}}$$

where v_{solids} (cm^3/g) is the specific volume of the coffee polymers constituting the matrix, approximated by measuring the specific volume of $\sim 100 \mu\text{m}$ dry coffee particles.

Measurements were performed in triplicates, and the relative standard deviation between the replicates was $<5\%$.

Dynamic Scanning Calorimetry (DSC). The DSC technique was used to quantify the freezing water in wetted roasted and ground coffee.

Samples of 15–20 mg of coffee were manually compressed into 7 mm diameter stainless steel crucibles (medium-pressure ME-29990; Mettler Toledo). The crucibles were tightly sealed with a stainless steel lid, with an O-ring placed between the pan and the lid.

The samples were scanned in a differential scanning calorimeter DSC 821e (Mettler Toledo). DSC mode was used from -40 to $200 \text{ }^\circ\text{C}$. The temperature program consisted in three ramps: (1) heating from -40 to $95 \text{ }^\circ\text{C}$ at $10 \text{ }^\circ\text{C}/\text{min}$; (2) cooling from 95 to $-40 \text{ }^\circ\text{C}$ at $20 \text{ }^\circ\text{C}/\text{min}$; (3) heating from -40 to $200 \text{ }^\circ\text{C}$ at $10 \text{ }^\circ\text{C}/\text{min}$. The endothermic peak of water melting was integrated. Measurements were repeated at least three times, and the relative standard variation between the replicates was found to be $<5\%$.

Particle Sizing. The mean volume diameter $D[4,3]$ of the dry or wetted coffee particles was measured by the use of sizing laser beams (Mastersizer from Malvern Instruments). About 1 g of coffee particles was inserted in the device measuring chamber, where it was dispersed in butanol. Measurements were done at given times after the coffee wetting operation. A new coffee sample was prepared for each measurement performed. Measurements were done in triplicates, and the maximum relative standard deviation between replicates was 7%.

Modeling of Particle Swelling Kinetics. The swelling kinetics of the particles was analyzed using the well-established theory of diffusion. The analytical solution to Fick's diffusion law for a spherical geometry was developed by Crank (16). This model has been used in roasted and ground coffee to approximate release kinetics of solutes (3, 6) or carbon dioxide (13) and in several other biological materials to predict the absorption kinetics of water as reported in ref 14. The model commonly used yields

$$\frac{M_t - M_0}{M_\infty - M_0} = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left[-\frac{D_{\text{app}} n^2 \pi^2 t}{R^2}\right]$$

where M_0 , M_t , and M_∞ are the total amount (g) of diffusing substance present in the sphere at time $t = 0$, t , and after infinite time, respectively; D_{app} is the apparent diffusion coefficient (m^2/s); n is the number of composite spheres; and R is the radius of the sphere (m).

Swelling of roasted and ground coffee was studied by Hsu (17). The author calculated the swelling degree of particles from data on bulk density and found that swelling was proportional to the amount of water used for coffee wetting. Thus, assuming that swelling occurs proportionally to the quantity of water having diffused inside the particles, the aforementioned model was adapted to approximate the moisture diffusivity from the variation in particle volume. Particle volume was calculated using measurements of particle diameter $D[4,3]$:

$$\frac{V_t - V_0}{V_\infty - V_0} = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp\left[-\frac{D_{\text{app}} n^2 \pi^2 t}{R^2}\right]$$

where V_0 , V_t , and V_∞ are the volume of the sphere at time $t = 0$, t , and after infinite time, respectively (m^3); D_{app} is the moisture apparent diffusion coefficient (m^2/s); n is the number of composite spheres (200); and R is the radius of the sphere at $t = 0$ (m).

The aforementioned model does not allow the use of a variable value of particle radius and, thus, we chose to take the initial radius of the particles as constant for the calculations. For this reason, it is clear that this approach allows only a rough approximation of diffusivity values. To get diffusivity values closer to reality, a more complex model would have to be developed, such as the model developed by Rädlerer (18) for the drying kinetics of polymer films. Nevertheless, the development of such a model would be out of the scope of this study.

Values of diffusivity were determined by minimizing the sum of squared error between experimentally measured and predicted values of particle volume. Two coffee samples were analyzed: particles with ~ 750 and $1050 \mu\text{m}$ average diameter before wetting. For each coffee sample, the average diffusivity was calculated using the average values of the volume as a function of time. Due to the relatively high variation in the $D[4,3]$ measured values, diffusion coefficients were also

calculated from each swelling curve using, respectively, the lower and the higher values of $D[4,3]$, that is, the extreme values of the error bars. The variation of the $D[4,3]$ values for a given kinetics yielded a relative standard deviation for D_{app} of $\sim 60\%$. This relatively high variation was attributed to several factors: the inherent variation of the particle swelling kinetics, the use of the broadly size-dispersed particles, and the relatively small amounts of the sample used for the measurements.

RESULTS AND DISCUSSION

Detection of Liquid Water by SEM. Preliminary trials aimed at showing the structural differences between dry and wetted coffee particles. As shown by the micrographs in **Figure 1**, in the dry coffee particle (**A**) cell voids and cell walls are clearly observable. In contrast, a coffee particle that has been immersed in water (**B**) presents morphological changes of the cells. These changes are likely associated with the solubilization of compounds occurring during water imbibition and which remain in the cells after water sublimation (sublimation occurs inside the microscope as measurements are performed at very low temperature and pressure). Morphological changes such as the porous structure of the soluble components could have been accentuated by cryoconcentration, assuming that sublimation of ice crystals occurs during etching. Thus, SEM observation directly showed water penetration into the coffee pores and solubilization of compounds. At higher magnification, it was possible to see the structure of the solubilized materials and some vacuoles remaining in the cells due to the presence of trapped gas (**Figure 1C**).

For coffee samples with 1 g of water per gram of coffee and stabilized at 90 °C for 20 min, liquid water was evidenced only at the periphery of the particles with diameter $D[4,3] \sim 1.0$ mm (**Figure 2**). Samples with higher water content, 1.5 g of water per gram of coffee, were also investigated. **Figure 3** shows a ~ 1.0 mm diameter particle 20 min after wetting and indicates that almost all coffee cells have contained liquid water.

Analysis of coffee with dark and light roasting degrees showed similar results.

To conclude, the SEM technique allowed a better understanding of the distribution of water in coffee particles after wetting. Due to the solubilization of compounds, the presence of liquid water could be detected in the coffee cells. However, this technique did not give any information about the presence of water in cell walls.

Decrease of Coffee Closed Porosity by Wetting. Measurements of dry coffee using He pycnometry showed that closed porosity, that is, pores not accessible to the gas, was higher for larger particles (~ 0.45 and 0.20 cm³/g for 1100 and 400 μ m dry particles, respectively, for dark roast) and for darker roast. These results are in agreement with those of previous studies (12, 13).

For the various coffee samples analyzed (~ 400 , 750, and 1100 μ m diameter particles), the closed porosity decreased with increasing water content (**Figure 4**). For all studied samples (400–1100 μ m particles), above a water content of 0.4–0.5 g/g of coffee, the closed porosity was lower than 0.1 cm³/g. The decrease of closed porosity may be explained by two factors. The first observable factor is that liquid water fills the coffee cell lumen, as demonstrated previously by SEM. The second possible contributing factor is the plasticizing effect of water on the coffee matrix polymers. The plasticizing property of water on biopolymers is due to several characteristics of the molecule (19): (i) it has a good affinity to carbohydrate matrices (in the coffee matrix, polysaccharides are known to be hydrophilic); (ii) it is a small size molecule, which can easily fill and

increase the voids between polymer chains; and (iii) it is able to decrease molecular interactions between biopolymer chains. At the macroscopic level, these properties lead to an increase of the matrix deformability and the mobility of the other constituents. These properties are well-known in matrices such as maltodextrins (20, 21). In coffee, they are involved in the swelling of the coffee matrix and may result in an increase in the diffusivity of other molecules such as He during the pycnometry analysis. An example of the consequences of coffee matrix relaxation (swelling) on the diffusivity of other molecules is given in the studies from Bichsel (3) and Spiro and Chong (5). The authors found that during infusion, caffeine is extracted 2 times more rapidly from pre-swollen coffee than from dry coffee.

To get a quantitative approximation of the water bound to the coffee polymers after wetting, that is, water having a plasticizing effect, samples were analyzed by DSC. The endothermic peak of melting water was determined as a function of the sample's water content. The water freezing ability has often been considered as a measure of the binding state of water (22). Using the calorimetry method, ~ 0.15 g of water/g of coffee on a dry basis was found to be nonfreezable water (**Figure 5**). Above this value, water remains in the "free" state, not bound to the matrix polymers.

We should note that for the lower particle size (400 μ m), the closed porosity tended slightly to re-increase after ~ 0.5 g of water/g of coffee. This phenomenon was not observed for larger particles (1100 μ m) and was probably due to particle aggregation. We supposed that there was creation of air spaces between the particles due to aggregation, decreasing the accessibility of helium.

Pycnometry experiments were possible only at ambient temperature so that kinetics of water migration was not determined with this technique.

Water Absorption Dynamics in Roasted Coffee. The swelling kinetics of coffee particles after wetting was determined from measurements of the particles' diameter $D[4,3]$. Water diffusion coefficients were then calculated from the swelling curves (increase in particles volume) for two coffee samples, ~ 750 and 1050 μ m initial diameter, as reported in **Figure 6**. The volume of coffee particles with a water content of 1 g/g of coffee increased by up to 20–23% at 10–15 min following wetting. This result confirmed the plasticizing effect of water as discussed in the previous section. Besides, we deduced that the parameter "particle diameter" cannot be considered as constant (which is the assumption often made in the literature) but variable as a function of the time. The use of a constant characteristic length to determine diffusion coefficients in swelling biopolymers may lead to discrepancies in the calculated values. Nevertheless, several authors neglect the influence of this parameter, especially in the high water content range, where the effect of swelling on diffusivity is relatively low. In our case, using the initial or the final (after swelling) particle radius would yield a variation of $\sim 30\%$ in the diffusivity values.

The water diffusion coefficients (D_{app}) estimated using the average values of the particle volume as a function of time were 2.3×10^{-11} and 3.4×10^{-11} m²/s for the lower and the larger particle sizes, respectively. Considering the variation in the $D[4,3]$ measured values (RSD $\sim 60\%$), the difference in the average diffusivities calculated for the two particle sizes is not significant, which is in accordance with the diffusion theory.

The time scale of water absorption by coffee particles (~ 10 min) compared well with the time needed for most of the soluble compounds to be released as reported in ref 6. The diffusivity

value for water is about 10 times lower than those found for caffeine [$\sim 2 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ at 80 °C (2)], *trans*-chlorogenic acid [$\sim 2.2 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ at 90 °C (23)], or other soluble compounds [$\sim 1.1 \times 10^{-10} \text{ m}^2 \text{ s}^{-1}$ (6)] during infusion in water continuous medium. This could be due to the fact that we use partially wetted coffee, whereas the values cited from the literature correspond to fully immersed coffee.

Results from this study showed that liquid water was able to reach and fill coffee cell cavities. Furthermore, the occurrence of swelling indicates that water must have diffused through the cell wall matrix. Following these observations, two water transfer mechanisms can be proposed: diffusion through cell wall polymers due to the concentration gradient and capillary transfer due to the porous structure of coffee. These mechanisms are common in porous solids (23).

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