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Article *in* Journal of Food Science · April 2000

DOI: 10.1111/j.1365-2621.2000.tb16026.x

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Pore Structure of Coffee Beans Affected by Roasting Conditions

S. SCHENKER, S. HANDSCHIN, B. FREY, R. PERREN, AND F. ESCHER

ABSTRACT: Hot-air roasting of coffee beans not only forms color and flavor compounds but also leads to a complete alteration of the bean microstructure. The resulting pore structure controls mass transfer phenomena during roasting and storage. The principal objective of the present project was to investigate the influence of different roasting conditions on volume increase and pore-structure development. Coffee beans were roasted in 2 different, well-defined roasting processes to equal degree of roast. Volumetry, mercury porosimetry, and electron microscopy were employed to study structural product properties. The roasting conditions were found to have a major impact on microstructure. High-temperature roasted coffees had greater bean volume, pore volume and larger micropores in the cell walls as compared to low-temperature roasted beans.

Key Words: coffee beans, roasting, porosity, mercury porosimetry, scanning, electron microscopy

Introduction

HOT-AIR ROASTING OF COFFEE BEANS IS A TRADITIONAL THERmal process, which in spite of its great importance in practice, is still designed and operated on an empirical basis. During coffee roasting at temperatures above 200 °C, drying takes place, water is redistributed, and complex chemical reactions such as Maillard reaction and pyrolysis are induced (Clarke and Macrae 1985; Illy and Viani 1995). Loss of water and organic mass leads to large amounts of gases and causes high internal pressure. The formation of color and flavor compounds is accompanied by a large increase in bean volume and changes in ultrastructure of both the cell wall and the cytoplasm of the green bean. In particular, it is assumed that the roasting process alters the porosity of the cell wall (Saleeb 1975; Puhlmann 1986; Massini and others 1990; Gutiérrez and others 1993; Illy and Viani 1995; Wilson 1997).

Roasting-induced changes in pore structure have a major impact on the final product quality. The pore structure controls mass transfer phenomena during storage and determines the high gas adsorption capacity and the degassing properties of the roasted bean (Radtke 1975; Saleeb 1975; Massini and others 1990). Fine micropores allow the mobilized coffee oil to migrate to the bean surface (Puhlmann 1986; Illy and Viani 1995; Wilson 1997). The loss of flavor compounds and the subsequent change in flavor profile during storage (staling) are probably related to the extent of exposure of inner surface and to the oxygen accessibility (Holscher and Steinhart 1992; Gutiérrez and others 1993). Both volume increase of the bean and development of pores during roasting are known to be highly dependent on the roasting conditions (Ortolá 1998). Therefore, the selection of appropriate roasting conditions to optimize microstructure presents a major goal in roasting technology.

The macropore system of roasted coffee beans mainly consists of excavated cells and has been described extensively using light microscopy, scanning electron microscopy (SEM), and image analysis procedures (Bürgin 1969; Dentan 1977; Dentan and Illy 1985; Puhlmann 1986; Massini and others1990; Gutiérrez 1993; Illy and Viani 1995; Wilson 1997). Radtke (1975) reported bean porosity values ranging from 0.38 to 0.49 depending on the origin and the pretreatment of coffee. Kazi and Clifford (1985) found different average cell sizes for "high yield" (34 to 40 μm) and "regular" coffees (21 to 23 μm), respectively. Massini (1990) described the development of pores in the course of roasting using SEM and reported the entire bean surface to be cracked after 10 min of roasting. The most comprehensive investigation on coffee bean porosity has been presented by Gutiérrez and others (1993). Various physical methods as well as SEM and image analysis were used to determine the porosity of coffee roasted at different temperatures to the same degree of roast. Again, high-temperature roasted coffee was found to have a statistically significantly higher macropore area than low-temperature roasted products.

So far, very little is known on the formation of micropores in the cell wall as affected by roasting conditions. According to Dentan (1977) cell walls of green coffee beans are crossed by numerous plasmodesmata. Saleeb (1975) concluded from gas adsorption measurements that the macropores of roasted beans are accessible through very narrow micropores of molecular magnitude (2.8 nm), which form a so-called ink-bottle structure. In contrast, Wilson (1997) found no evidence of pre-existing channels within the cell walls of the green beans, but 2 different types of micropores of an average diameter of 100 nm and 10 nm, respectively, were present in roasted beans.

A direct microscopic visualization of micropores in the cell wall is difficult. On the other hand, mercury porosimetry allows the determination of pore volume and pore size distribution (Adamson 1990; Perren and Escher 1997). Our objective was to assess mercury porosimetry on coffee and to investigate the influence of different roasting conditions on the micropore system of coffee beans. The application of mercury porosimetry to coffee requires additional information for correct interpretation. In particular, the localization of mercury in intruded beans was studied in frozen bean fractures by cryo-scanning electron microscopy and energy-dispersive X-ray microanalysis (Echlin 1992).

Results and Discussion

THE DEVELOPMENT OF BEAN VOLUME DURING HIGH- AND low-temperature roasting is presented in Fig. 1. High-temperature conditions resulted in much higher expansion rates as compared to low-temperature conditions. Figure 2 compares the relative bean volume as a function of roast loss and clearly shows the large difference between the 2 roasting processes at a medium degree of roast. Also, volume increase was steady in both processes as no instantaneous expansion is observed that would lead to a discontinuity in the curve. Table 1 summarizes the volumetric and porosimetric measurements on coffee samples from high-temperature short-time roasting pro-

cess (HTST) and low-temperature long-time process (LTLT) roasting. Depending on the time temperature program, a high- or low-density coffee as expressed by bean density and bean porosity may be obtained. Finally, the large expansion of cell volume is shown in the SEM micrographs in Fig. 3 and Fig. 4 of a green bean sample and a LTLT roasted bean sample, respectively. The unusually thick cell wall in both the green and roasted coffee bean should be noted in particular.

The pore size distribution in roast coffee was dominated by small micropores in a very narrow dia range of 20 to 50 nm (Fig. 5). At equal degree of roast curves of cumulated pore volume were influenced by the roasting conditions. High-temperature roasted samples showed the major increase in cumulated pore volume (r_{main}) to occur at slightly greater equivalent pore radius than low-temperature roasted samples (13.45 nm versus 11.22 nm). A significant difference between the 2 roasting processes was also found for the final value of cumulated pore volume (Table 1).

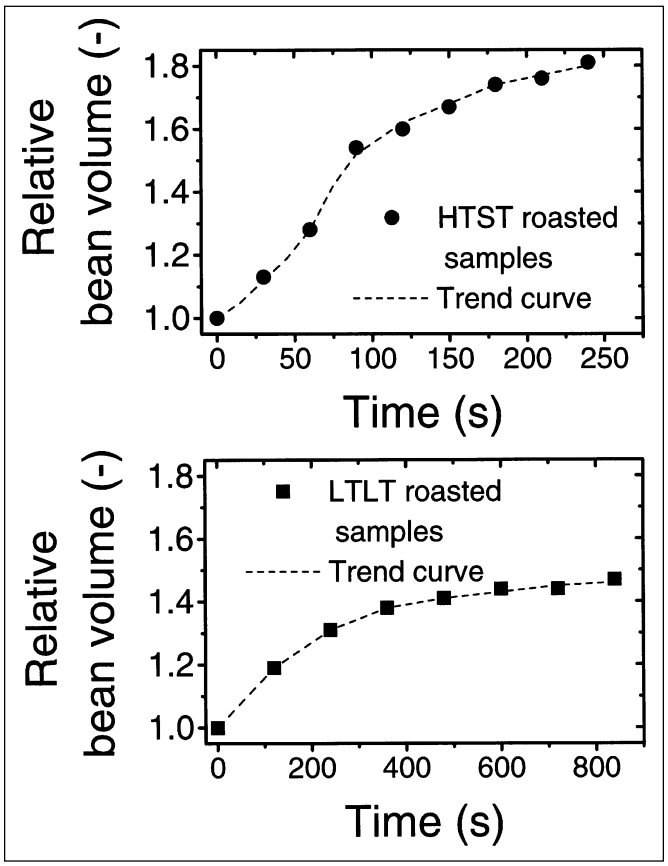

Fig. 1—Development of relative bean volume increase during high- and low-temperature roasting.

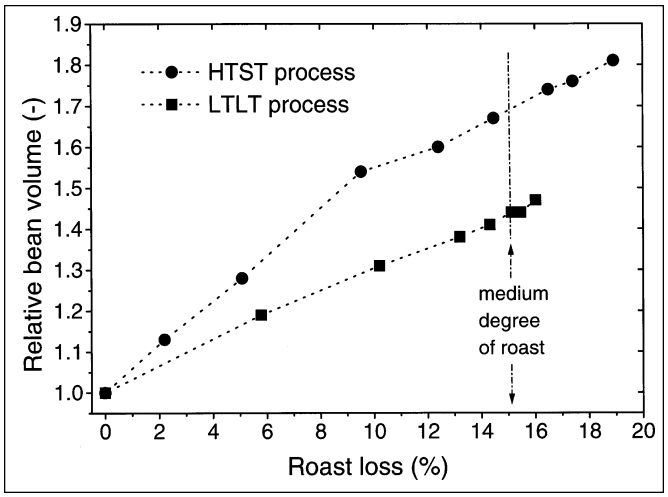

Fig. 2—Development of bean volume increase as a function of roast loss during high- and low-temperature roasting.

Table 1—Influence of roasting conditions to equal roast loss on volume and pore characteristics of roasted beans

	HTST roasting	LTLT roasting
Roast loss (%)	14.95	15.01
Bean density (kg m^{-3})	622	747
Bean volume V_B (mm^3 g^{-1})	1609	1350
Hg-intruded volume V_{Hg} (mm^3 g^{-1})	850	640
Bean porosity $\epsilon = V_{Hg}/V_B$ (-)	0.528	0.474

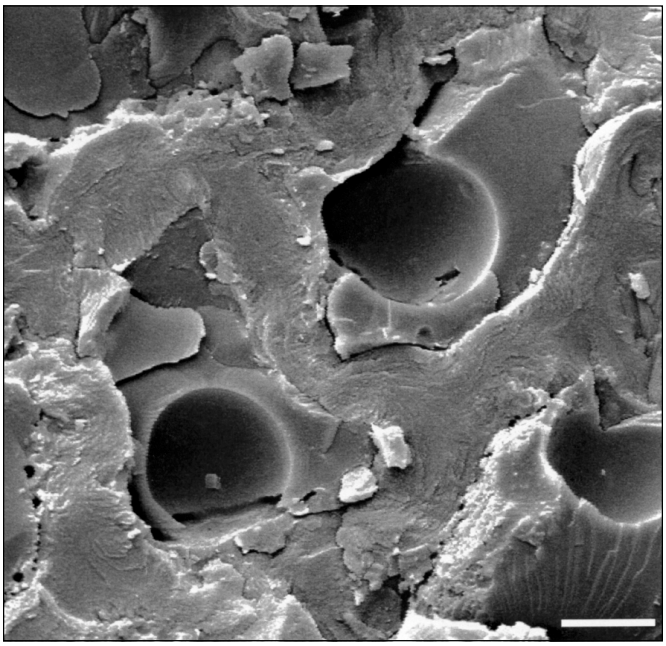

Fig. 3—Cryo-SEM micrograph of cells in green coffee beans. Scale bar = 10 μm.

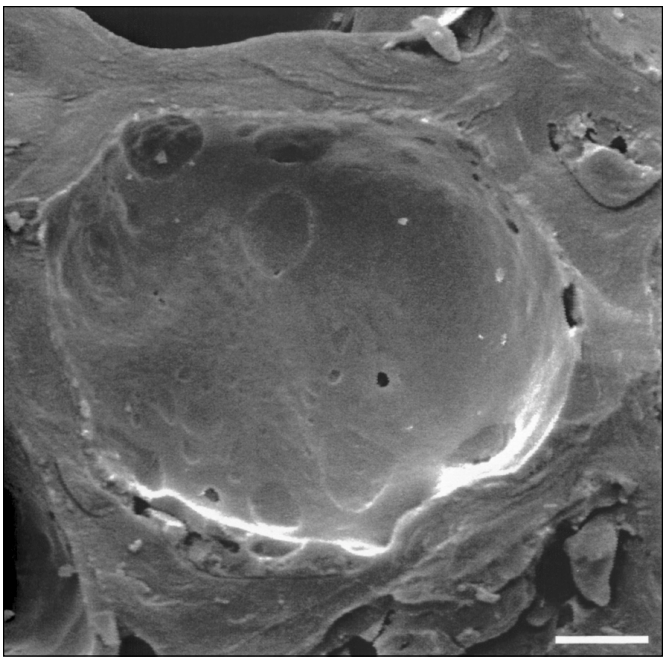

Fig. 4—Cryo-SEM micrograph of a cell in roasted coffee beans. Scale bar = 10 μm.

The volume increase of coffee beans during roasting is affected by a driving force and, opposed to it, by structure resistance. Development of internal gas and water vapor imposes a driving force for expansion, whereas the glass transition phenomena may play an important role in structure resistance. Because the glass transition temperature T_g is greatly influenced by the product dehydration, the bean is assumed to develop from a more glassy state into a more rubbery state and, finally, to fall back into a more glassy state due to the low water content. The shift in balance between force and resistance because of changes in temperature and water content causes the steady and continuous increase of bean volume. Greater driving force and a more rubbery structure probably led to greater volume increase at higher roasting temperatures. In parallel to a greater bean volume cell lumina, represented by the cumulated mercury-penetrated pore volume, were also larger in high-temperature as compared to low-temperature roasted beans. Therefore, while porosity values presented here are in the same order as found by Radtke (1975), our conclusions contrast those of Gutiérrez and others (1993) who did not find a significant influence of roasting conditions on bean porosity.

SEM analysis of mercury-intruded roast coffee material revealed a picture of still intact cell wall structure and mercury-filled cell lumina (Fig. 6). No artifacts , such as structure collapse due to high pressure during porosimetry, were observed. The cell lumina were filled with spheres. The elemental mapping of mercury in a freeze fracture across the cells is shown in Fig. 7, while Fig. 8a and 8b show single-spot spectra of a sphere

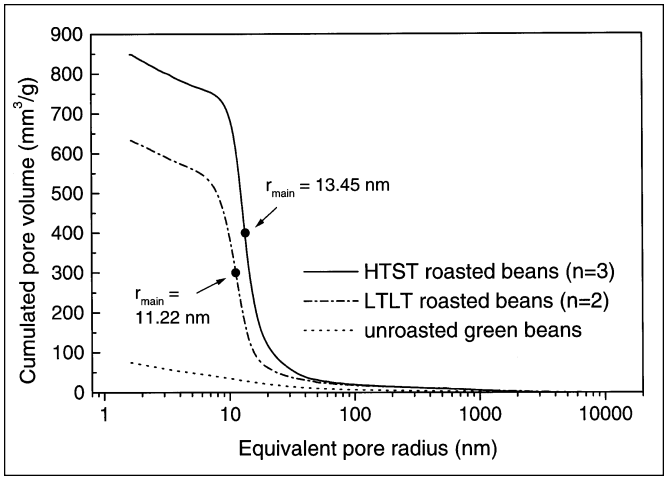

Fig. 5—Influence of roasting process on porosimetric curves. High-temperature roasted beans exhibit greater cumulated pore volume as compared to low-temperature roasted beans. Different sizes of cell wall micropores are indicated by substantial differences in r_{main} values.

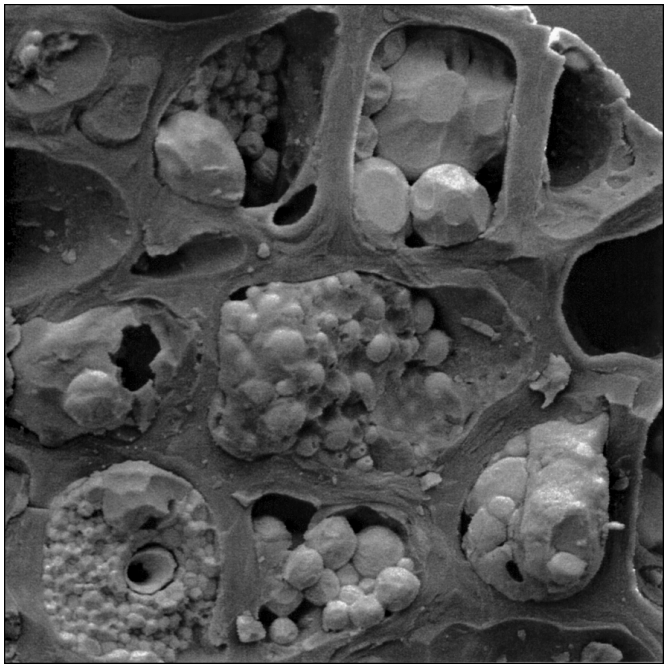

Fig. 6—Cryo-SEM micrograph of cells in a roasted bean intruded with mercury during porosimetric analysis. Integer cell wall structure with mercury-filled cell lumina. Image width = 137 μm.

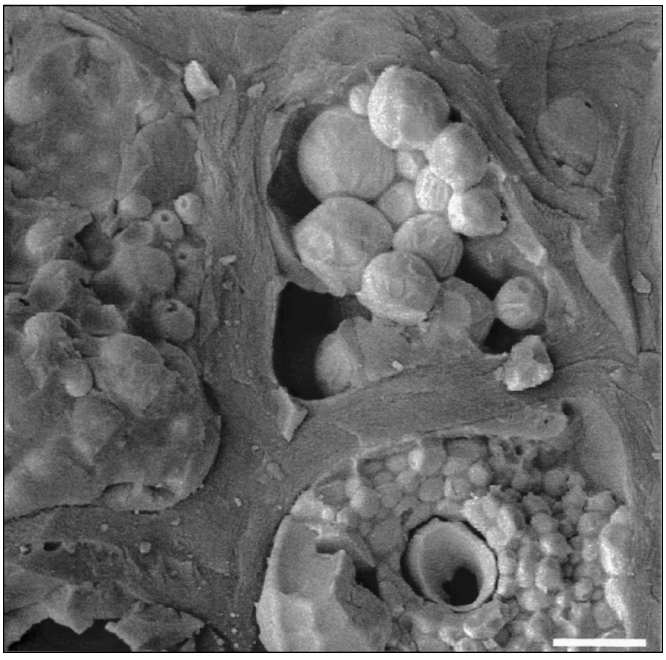

Fig. 7a—Cryo-SEM micrograph of a roasted coffee bean intruded with mercury during porosimetric analysis. Scale bar = 10 μm.

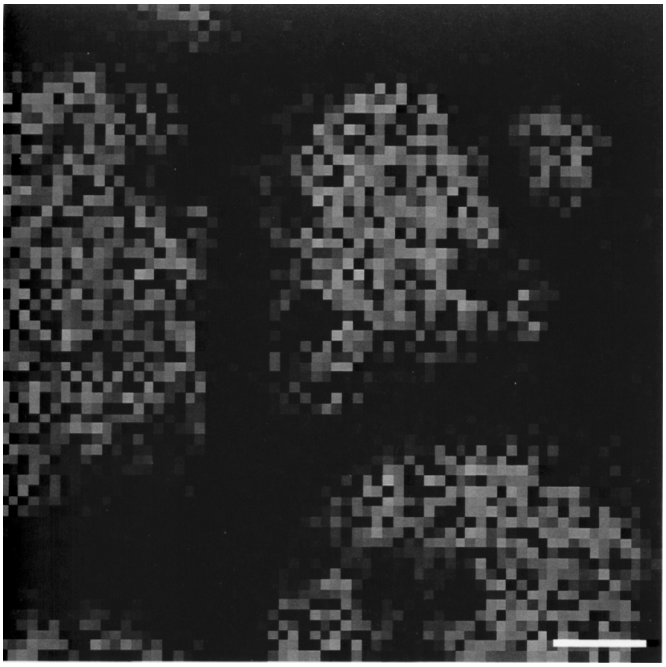

Fig. 7b—Mercury mapping obtained by X-ray microanalysis of a roasted coffee bean intruded with mercury (identical bean sample and sector as in 7a). Scale bar = 10 μm.

in the cell lumen and a cell wall cross-fracture, respectively. The mapping clearly confirms that mercury does enter pores to the full extent during porosimetry. A weak signal was even detected in the cell walls.

Access for mercury to the cell lumina is provided by small micropores in the cell walls, forming a so-called ink-bottle pore system. Therefore, only a high pressure corresponding to the small size of the entrance pores allows for mercury penetration of the cell lumina. Consequently, high values of apparent pore volume for the micropores of the cell wall were obtained, while this corresponded to the filling of the cell lumina. Hence, the pore size at the maximum of the distribution function (r_{main}) represents the size of cell wall micropores. Values for r_{main} fall between the 2 values reported by Wilson (1997) and are considerably higher than the value proposed by Saleeb (1975). Also the equivalent pore radius of these micropores was found to be significantly larger in high-temperature roasted beans (Fig. 5). High-temperature roasted beans may therefore exhibit increased permeability for gas and oil and promote easier access for oxygen.

Applying high pressures to sensitive foodstuffs can be a limitation of mercury porosimetry. It requires thorough evaluation of the application, checking for artifacts and careful interpretation of the results. The stability of coffee bean tissue exposed to mercury porosimetry is mainly due to the very thick cell walls. The withdrawal of a minor part of intruded mercury during the pressure release procedure could not be prevented. Nevertheless, the main part of mercury was trapped and remained in the cell lumen. The formation of mercury spheres was because of contamination of mercury with cell components. Residual mercury within the micropores of the cell walls may have caused the weak mercury-signal in the elemental spectra. The slight but continuous increase in cumulated pore volume observed in green beans (Fig. 5) may be caused by micropores to some extent and/or represent an artifact due to compression of coffee oil at high pressure. The concept of mercury porosimetry is based on a series of idealizing assumptions, such as a cylindrical shape of the intruded pores.

The question of origin and structure of the micropore system within the cell wall is not yet answered satisfactorily. It is still unclear whether this micropore system consists of countable discrete microchannels such as plasmodesmata rather than of a complex 3-dimensional permeable wad-like network of polysaccharide. The existence of modified plasmodesmata channels in roast coffee cell walls was confirmed by transmis-

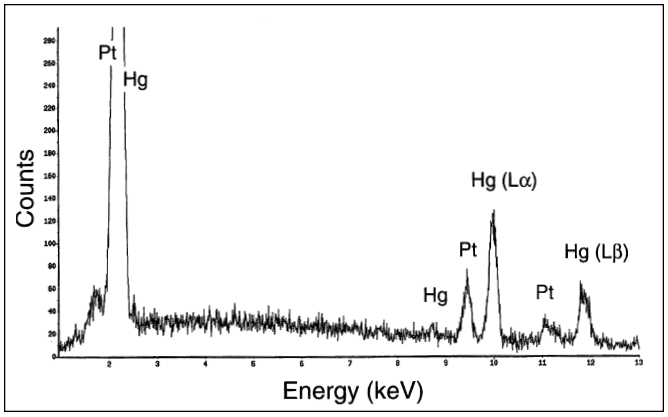

Fig. 8a—Net counts of mercury from X-ray microanalysis in a cell of a roasted coffee bean intruded with mercury during porosimetric analysis.

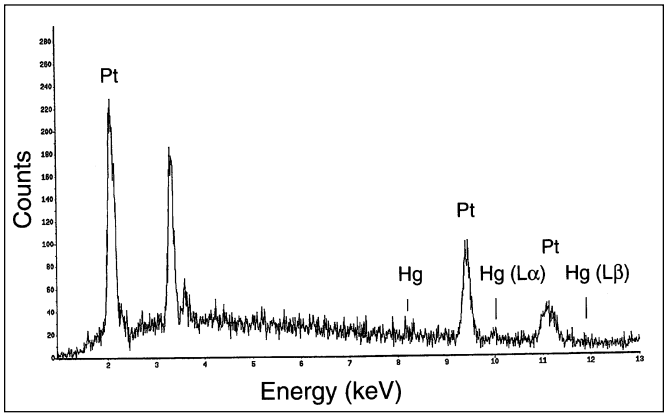

Fig. 8b—Net counts of mercury from X-ray microanalysis in the cell wall of a roasted coffee bean intruded with mercury.

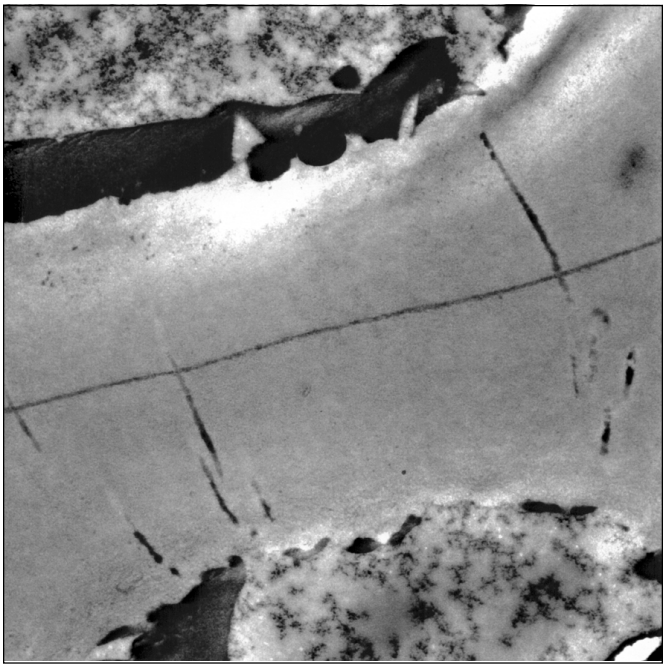

Fig. 9—TEM micrograph of a cell wall in a partially roasted bean. The continuous dark line is formed by the middle lamella, which lays between the thick cell walls and remainings of cytoplasm of 2 adjacent cells. Dark lines perpendicular to the middle lamella are parts of modified plasmodesmata channels through the wall. Image width = 9.06 μm.

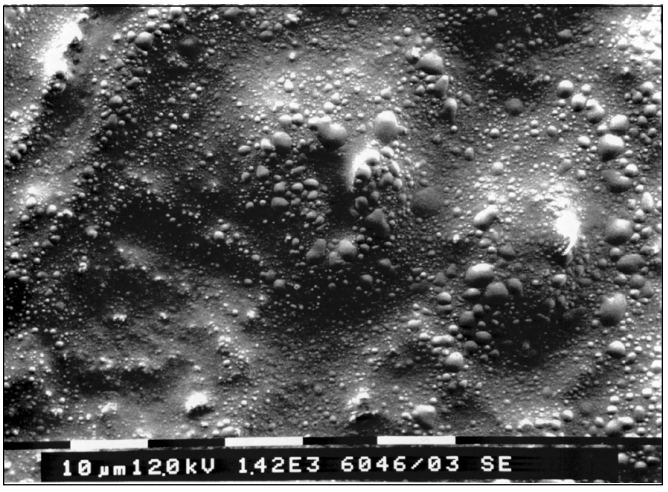

Fig. 10—Cryo-SEM micrograph of the surface of a high-temperature roasted bean after 1 d of storage. Numerous very small oil droplets covering the surface show the beginning oil migration process and indicate the presence of an extensive network of micropores.

sion electron microscopy (TEM) analysis (Fig. 9). However, these channels are cell-to-cell connections and do not provide access to the bean surface, which then would allow for degassing and oil migration. SEM-analysis (Fig. 10) of the bean surface in the state of the beginning oil migration process shows numerous very small oil droplets covering more or less the entire surface. These findings may support the model of a 3-dimensional permeable wad-like network of polysaccharides, where the latter have been partially degraded and removed during roasting. In this case, increased polysaccharide degradation at higher temperatures could be the cause for the wider cell wall micropores found in high-temperature roasted samples.

Conclusions

MERCURY POROSIMETRY IS A VALID AND USEFUL METHOD FOR investigating pore structure of roast coffee. Micropores in the range of 20 to 50 nm dia in the cell walls allow for mercury penetration of the cell lumina. At the same degree of roast, roasting conditions have a major impact on the structural product properties of coffee beans. High-temperature roasted beans exhibit greater bean volume, cumulative pore volume, and larger micropores in the cell walls compared to low-temperature roasted coffees. Larger micropores may promote faster degassing and oil migration as well as enhanced oxygen accessibility and accelerated loss of flavor compounds. The origin and structure of the micropore network in the cell wall remains unclear.

Materials and Methods

Raw material and roasting

Two wet-processed *Coffea arabica* Linn. varieties from Colombia and Costa Rica were obtained from a Swiss import company. The green bean water content was 10.8%and 11.1% (wb), respectively. Roasting experiments were carried out with a fluidized-bed hot-air laboratory roaster in batches of 100 g green beans. For the present experiments, samples were roasted in a high-temperature short-time roasting process (HTST) and a low-temperature long-time process (LTLT) defined in Table 2. Roasting was monitored and controlled by online temperature measurements, and, to describe roasting dynamics, samples were removed at regular intervals during roasting and analyzed. In order to be able to compare the 2 processes, roasting was targeted to the same degree of roast, based on overall weight loss and final product color. Typical product properties are also presented in Table 2. All samples that were used for porosimetric analysis had a roast loss of 15%, which in practice corresponds to a "medium degree of roast."

Color measurement

Color was measured with a tristimulus colorimeter Chroma Meter CR-310 (Minolta, Japan) with a measured reflection area of 19.6 cm^2. Ground coffee samples were placed in a petri dish and gently pressed by hand with equal force to form an even surface. Results were depicted in the CIE *L*a*b** color space.

Volumetry

A displacement method based on a system described by Mohsenin (1986) was used to determine bean volume and bean density. A small container was filled with peanut oil (density 910 kg m^{-3} at 25 °C) and placed on a balance. Thirty g of roasted beans were weighed into a wire basket that was suspended on a support beside the balance. The basket was immerged into the oil and moved up and down for 15 s in order to release air bubbles trapped between the beans. The oil surface tension and the limited immersion time assured a negligible amount of oil penetrating the pores of the sample. Immersion was carried out likewise with the empty basket. The weight difference of the immersed basket with and without coffee beans corresponds to the weight of oil displaced by the beans, and bean volume was calculated using oil density. Bean density was computed as ratio of bean weight in air and bean volume. Relative bean volume at progressing roasting was based on the volume of green beans, taking the weight loss during roasting into account.

Table 2—Roasting parameters for HTST and LTLT process and typical properties of roasted products

	HTST roasting	LTLT roasting
Process parameters:		
Hot-air flux (m^3s^{-1})	0.01885 ± 0.00079	
Hot-air temperature (°C)	260 ± 0.5	220 ± 0.5
Hot-air humidity	dry (no air recirculation; no water added)	
Roasting time(s)	155 ... 180	540 ... 720
Cooling	cold air; bean temperature < 40 °C reached within 60 s	
Product properties (typical values):		
Color (L*/a*/b*)	24.06 / 9.26 / 11.33	24.02 / 9.27 / 11.17
Roast loss (%)	15.33	15.81
Water content (g H_2O/100g coffee)	2.68	2.15

Mercury-porosimetry

Porosimetry was carried out using a mercury-porosimeter Carlo Erba 2030 (Carlo Erba Strumentazione, I-Rodano) comprising a macro- and a micropore unit. About 0.4 g roast coffee (4 to 5 bean halves) were placed in a dilatometer and evacuated in the macropore unit for some 15 min. The dilatometer was carefully filled with mercury and then transferred to the micropore unit. The pressure was gradually increased to 400 MPa (4000 bar) during 45 min, and the volume of intruded mercury recorded. For samples used in SEM analysis, a high-pressure release rate of 16 bar s^{-1} was applied, in order to partially prevent mercury from withdrawing out of the pores. Pressure values were converted into values of "equivalent pore radius" r, based on the Washburn equation (Adamson 1990), which describes a linear relationship between the size of an intrudable circular pore and the applied mercury pressure. The pore radius corresponding to the maximum in the distribution function is defined r_{main} = maximum $dV/d \log (r/r_0)$, whereby V = cumulated pore volume and r_0 = 1 m (normalization, dimensionless exponent). Porosity ϵ was defined as the ratio of absolute volume per g bean and absolute volume of intruded mercury per g bean.

Cryo-Scanning electron microscopy (Cryo-SEM)

The bean-pore structure was investigated by cryo-SEM, with a Philips 515 microscope (Philips, The Netherlands) equipped with a SEM cryo unit (SCU 020, Bal-Tec, Balzers, Principality of Liechtenstein). Pieces of beans were frozen in liquid nitrogen, fractured by a scalpel, and transferred to the cold stage of the preparation chamber. The samples were exposed to −80 °C for 10 min under $p < 2 \cdot 10^{-4}$ Pa and cryo-sputtercoated with 15 nm platinum. The specimens were examined at a temperature below −130 °C at an accelerating voltage of 12 kV.

Energy-dispersive X-ray microanalysis

Elemental analysis was performed in the Philips SEM, equipped with a Tracor® Northern energy-dispersive X-ray analysis system and as described in detail by Frey and others (1996). The microscope was operated at an accelerating voltage of 25 kV and a working distance of 12 mm. Elemental mapping was carried out by energy window mapping. The spatial resolution was 64 × 64 pixels with a dwell time of 0.1 s. All spectra in the spot mode were acquired at a magnification of 10 000 for 60 s (live time) and a dead time of 24 s (40%) in the energy range of 0 to 13 keV.

Transmission electron microscopy (TEM)

Sample preparation for TEM analysis was carried out with a modified procedure according to Angermüller and Fahimi (1982). Small bean pieces were fixed in half-strength Karnovsky fixative at room temperature for 1 h. After washing in 0.1 M phosphate buffer, postfixation was done for 1 h in 1% osmium tetroxide in 0.1 M imidazole buffer. Samples were then rinsed in distilled water and dehydrated in a graded series of ethanol with stepwise embedding in Epon/Araldite resin. Ultrathin sections were stained with uranyl acetate and lead citrate before examining in a Hitachi H-600 transmission electron microscope at 100 kV. Images were digitally recorded using a Gatan slow-scan CCD camera.

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MS 19990446 received 4/26/99, revised 12/27/99, accepted 3/30/00.

We gratefully acknowledge the funding of this work by G.W. BARTH GmbH & Co., Germany; support of Keme Food Engineering AG, Haco AG and Migros Betriebe Birsfelden AG, Switzerland; and the Institute of Geotechnical Engineering, ETH Zurich, for putting the porosimeter at our disposal.

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