

Aroma Recovery from Roasted Coffee by Wet Grinding

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Abstract: Aroma recovery as determined by solid phase microextraction–gas chromatography–mass spectrometry (SPME–GC–MS) was compared in coffees resulting from conventional grinding processes, and from wet grinding with cold and hot water. Freshly roasted coffee as well as old, completely degassed coffee was ground in order to estimate the relationship of internal carbon dioxide pressure in freshly roasted coffee with the aroma loss during grinding. The release of volatile aroma substances during grinding was found to be related to the internal carbon dioxide pressure, and wet grinding with cold water was shown to minimize losses of aroma compounds by trapping them in water. Due to the high solubility of roasted coffee in water, the use of wet-grinding equipment is limited to processes where grinding is followed by an extraction step. Combining grinding and extraction by the use of hot water for wet grinding resulted in considerable losses of aroma compounds because of the prolonged heat impact. Therefore, a more promising two-step process involving cold wet grinding and subsequent hot extraction in a closed system was introduced. The yield of aroma compounds in the resulting coffee was substantially higher compared to conventionally ground coffee.

Keywords: aroma stripping, gas chromatography, roasted coffee, solid phase microextraction, wet grinding

Introduction

For coffee brewing, the whole roasted coffee beans need to be ground to increase the surface exposed to extraction. Due to the specific structural properties of coffee beans, grinding is a two-step process. The brittleness of roasted coffee beans requires a crushing phase where the bean is broken down into fragments before these fragments can be ground finely using shear forces (Illy and Viani 1995). During the breakdown of cells, substantial amounts of carbon dioxide and carbon monoxide along with other volatile compounds are released. To minimize loss of volatile substances, condensing or absorbing devices are applied, and the entrapped volatiles are used in later stages of manufacturing and packaging (Clarke and Macrae 1987). The volatiles released during grinding could possibly be trapped in water as proposed by some patents that describe the production of coffee beverages with increased aroma intensity by applying a grinding step with water (Hussmann 1983; Abe and others 2000; Kimura and others 2003). Wet grinding, for example by using a ball mill, would in addition have the advantage that a specific particle size distribution is relatively easy to obtain (Illy and Viani 1995). In addition, the impact of temperature during dry grinding is not negligible and can reach more than 100 °C, probably leading to undesired reactions (Illy and Viani 1995). The cooling effect of water would prevent excessive temperatures during wet grinding. The fact that coffee exhibits a high solubility even in cold water restricts the use of wet-processing equipment to applications wherein an extraction step follows immediately after grinding, as it is the case in the manufacturing of instant coffee.

The aim of this work was to show whether wet grinding methods involving cold or hot water can be established in laboratory and pilot scale and whether these methods can reduce loss of aroma in the grinding process.

Materials and Methods

Roasting process and process characterization

Roasting. Green washed *Coffea arabica* from Colombia was provided by Delica (Birsfelden, Switzerland) and batches of 200 g were roasted on a fluidized-bed hot air laboratory roaster (Buhler-Barth Ltd., Freiberg/Neckar, Germany). The raw coffee was roasted under low temperature—long-time process conditions (Geiger and others 2005). Hot air temperature was 228 °C, hot air velocity was 3.5 m/s, and the roasting time was 12 min. After roasting, an air stream of ambient temperature ensured fast cooling of the beans (4 min). A detailed description of the roaster can be found in Schenker and others (2000). Coffee was roasted to a color of $L^* = 22.4 \pm 0.4$ and to a moisture content of 1.9 g/100 g wb.

For the experiments with completely degassed coffee, a 1:1:1 blend of Sul da Minas (unwashed Arabica, Brazil), Sidamo (washed Arabica, Ethiopia), and Tarrazu (washed Arabica, Costa Rica) provided by Rast Ltd. (Emmen, Switzerland) was used. Roasting was carried out with a G-45 drum roaster (Probat Ltd., Emmerich, Germany) 11 mo before the grinding experiments. Coffee was roasted in a batch of 6 kg to a color of $L^* = 22.4 \pm 0.4$ and to a moisture content of 1.8 g/100 g wb. Coffee beans were stored in a valve bag under nitrogen.

Color. Coffee was ground, gently pressed, and color was measured using a CR 310 photometer (Minolta, Dietikon, Switzerland). Results were expressed in the CIE $L^* a^* b^*$ color space, and L^* values were used as a measure for the degree of roast.

Water content. The moisture of roasted coffee was determined gravimetrically after drying 5 g of ground coffee at 103 °C during 5 h.

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Wet grinding of roasted coffee

For wet grinding of roasted coffee beans, 2 devices were investigated: a Fryma corundum stone colloid mill (MZ-80/R, Fryma-Maschinen Ltd., Rheinfelden, Switzerland), and a Waring laboratory blender (30BL80, Waring Products Inc., New Hartford, Conn., U.S.A.). Furthermore, wet grinding was carried out with cold and hot water. The grinding processes are summarized in Table 2.

Wet grinding with the corundum stone mill. Grinding was carried out alternatively with cold ($20 \pm 1^\circ\text{C}$) or hot ($90 \pm 1^\circ\text{C}$) distilled water. Eighty grams of roasted coffee beans were first ground with 1000 mL of water at a gap width of 0.5 mm. Then the gap width was narrowed to 0.3 mm and the coffee suspension ground again. After removing the suspension, the mill was rinsed with 400 mL of water at a gap width of 1.5 mm. A total of 1400 mL suspension with 57.15 g/L ground coffee resulted.

Wet grinding with blender. Grinding of 30 g of roasted coffee was again carried out with cold ($20 \pm 1^\circ\text{C}$) or hot ($90 \pm 1^\circ\text{C}$) distilled water. Coffee was weighed in the blender, 200 mL of distilled water was added and the coffee was ground during 2 min at lowest speed setting. Afterwards, 280 mL of distilled water were added to obtain a similar concentration of the coffee suspension as in the trials with the corundum stone mill.

Dry grinding of roasted coffee

For dry grinding, 3 devices were investigated: a Bühler-Miag disc mill (model 4000, Bühler-Miag Ltd., Milano, Italy), a Ditting disc mill (KFA 1403, Ditting Machines Inc., Bachenbuelach, Switzerland), and the Waring laboratory blender described earlier.

Table 1—Analytes and standards used in GC-MS analyses.

Analyte (A)	Selected ion (m/z) of A	Internal standard (IS)	Selected ion (m/z) of IS
Dimethyl sulfide (1)	47	[$^2\text{H}_6$]-1	50
Hexanal (2)	56	[$^2\text{H}_2$]-2	58
2-ethyl-3,5-dimethylpyrazine (3)	135	[$^2\text{H}_6$]-3	141
2-ethyl-3,6-dimethylpyrazine (4)	135	[$^2\text{H}_6$]-3	141
2-methylbutanal (5)	86 ^a	[$^2\text{H}_2$]-6	88 ^a
3-methylbutanal (6)	71	[$^2\text{H}_2$]-6	73
2,3-butanedione (7)	43	[$^{13}\text{C}_4$]-7	45
2,3-pentanedione (8)	100	[$^{13}\text{C}_2$]-8	102
Pyridine (9)	79	[$^2\text{H}_5$]-9	84
Dimethyl trisulfide (10)	126	[$^2\text{H}_6$]-10	132
4-vinylguaiaicol (11)	150	[$^2\text{H}_5$]-11	153

^aA response factor of 0.42 was applied to quantify 2-methylbutanal via [$^2\text{H}_2$]-3-methylbutanal.

The grinding processes are summarized in Table 2.

Dry grinding with disc mills. Coffee was ground at level 3 (Bühler-Miag) and level 1 (Ditting). The 1st 30 g of ground coffee was discarded in each run.

Dry grinding with blender. Batches of 30 g of coffee were ground for 2 min at the lowest speed setting.

Determination of particle size distribution of ground roasted coffee

The particle size distribution of dry ground coffee was determined by a sieve analysis on a vibrating sieve cascade (Magnetic Inc., Liestal, Switzerland) with five sieves of 1114, 630, 400, 200, and 125 μm mesh width, respectively.

To determine the particle size distribution of the wet ground coffees, the coffee powder had been recovered from the aqueous coffee suspension and dried. First, the coffee suspension was sieved through a 63- μm mesh sieve. The sieved off coffee particles were washed with water until the washing water was completely colorless, dried for 15 h at 103°C and sieved through the cascade as described earlier.

Sampling of ground coffee for aroma analysis

Each grinding run described above was carried out in triplicate and the resulting coffee suspensions were subjected to a sampling procedure and aroma analysis. Statistical analyses were performed using Student's *t*-test with $P < 0.05$.

Wet ground coffee. For retrieving samples from the suspensions of wet ground coffee, the suspensions were stirred for 2 min and then samples of 20 mL and 100 mL, respectively, were removed with 20 mL and 100 mL syringes with a wide aperture of around 7 mm. The 20 mL samples were transferred to flasks, diluted to 100 mL, and used for the analysis of the aroma compounds that were present in higher concentrations (Compounds 5 to 11, cf. Table 1), while the 100 mL samples were transferred to flasks and used directly for the analysis of aroma compounds present in lower concentrations (Compounds 1 to 4, cf. Table 1). During sampling, temperatures of cold and hot ground suspensions were kept at $20 \pm 1^\circ\text{C}$ and $90 \pm 1^\circ\text{C}$ for the whole sampling procedure. The amounts of dry mass in the samples are summarized in Table 2.

Dry ground coffee. To maximize the comparability of the grinding methods, the sampling procedure for dry coffee was kept similar to the procedure for coffees resulting from wet grinding. The dry ground coffee powders were transferred to stainless steel buckets, suspended in cold ($20 \pm 1^\circ\text{C}$) or hot ($90 \pm 1^\circ\text{C}$) water and stirred for 2 min. Then samples were retrieved with syringes

Table 2—Summary of wet and dry grinding runs and of sampling of ground coffee for aroma analysis.

Grinding method	Coffee used per run [g]	Water used for grinding per run [mL]	Water added after grinding per run [mL]	Dry mass in final suspension sampled for aroma analysis:	
				Compounds 1 to 4 ^c (5 g samples) [g]	Compounds 5 to 11 ^c (1 g samples) [g]
<i>Wet Grinding</i>					
W1 Corundum stone mill	80	1400	0	5.53 \pm 0.03	1.06 \pm 0.01
W2 Laboratory blender	30	200	280	5.5 \pm 0.3	0.99 \pm 0.07
<i>Dry Grinding</i>					
D1 Ditting ^a	80	0	1400	5.45 \pm 0.08	1.02 \pm 0.04
D1 Ditting ^b	80	0	0 ^d	4.905 \pm 0.005	0.981 \pm 0.005
D2 Laboratory blender ^a	30	0	480	5.1 \pm 0.3	1.03 \pm 0.04
D3 Bühler-Miag 4000 ^b	80	0	0 ^d	4.905 \pm 0.005	0.981 \pm 0.005

^aTests for comparison of aroma recovery with that obtained from wet grinding.

^bTests with microwave heating (hot extraction of ground coffee).

^cAccording to Table 1.

^dFor extraction and aroma analysis, samples were directly prepared with 100 mL water and 5 g and 1 g ground coffee, respectively.

as described earlier. The amounts of dry mass in the samples are summarized in Table 2.

Hot extraction of cold wet and dry ground coffee. Hundred milliliters of suspensions of cold wet ground coffee, prepared as described earlier, were microwave heated in closed flasks at 780 W for 65 s. Then the suspensions were stirred for 10 min, cooled to room temperature under running tap water for 20 min and used for analysis.

Five grams and 1 g samples of dry ground coffee were weighed into 100-mL flasks, suspended in 100 mL of cold water, microwave heated, and further treated as just described. The 5 g and 1 g samples were used for analysis of lower and higher concentrated aroma compounds, respectively.

Determination of the coffee mass in the suspension samples. In order to determine the amount of coffee taken up from the suspension by the syringe and to evaluate the repeatability of the sampling procedure, 5 samples of each grinding method were analyzed for dry matter by drying at 103 °C until weight remained constant. The respective values are summarized in Table 2.

Headspace solid phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) analysis of coffee aroma

Quantitative analysis of aroma compounds was carried out using the method described in Baggenstoss and others (2007). Three aldehydes (2-methylbutanal, 3-methylbutanal, hexanal), 2 diketones (2,3-butanedione, 2,3-pentanedione), 2 sulfides (dimethyl sulfide, dimethyl trisulfide), 3 heterocyclic compounds (pyridine, 2-ethyl-3,5-dimethylpyrazine, 2-ethyl-3,6-dimethylpyrazine), and 1 phenolic compound (4-vinylguaiacol) were analyzed (Table 1). Due to concentration differences and to avoid interferences between degradation products of analytes and standards (for example, dimethyl sulfide and dimethyl trisulfide), aroma compounds were split into 2 groups for analysis: aroma compounds 1 to 4 (approximately 5 g/100 g dry mass in 100 mL water) and aroma compounds 5 to 11 (approximately 1 g/100 g dry mass in 100 mL water). The coffee solutions obtained from the sampling procedures were spiked with definite amounts of stable isotope labeled internal standards (Table 1), stirred during 10 min, and 7 mL were transferred to a 20-mL headspace vial.

Coffee aroma compounds were sampled with solid phase microextraction at 40 °C during 10 min using a Supelco 50/30 μm StableFlex DVB/CAR/PDMS fiber (Supelco, Buchs, Switzerland). The GC-MS analysis of aroma compounds was carried out on a Fisons 8000 Series gas chromatograph (Thermo Electron, Allschwil, Switzerland) coupled to a quadrupole mass spectrometer SSQ710 (Finnigan MAT, San Jose, Calif., U.S.A.). The SPME fiber was desorbed in the split/splitless injector at 240 °C with a splitless time of 240 s. The aroma compounds were then separated on a 60 m \times 0.25 mm \times 0.25 μm medium polar ZB-1701 column (Phenomenex, Aschaffenburg, Germany) with the following temperature programs: 40 °C (6 min), 4 °C/min, 135 °C (0 min), 40 °C/min, 240 °C (5 min) (compounds 1 to 4, see Table 1), and 40 °C (6 min), 4 °C/min, 140 °C (0 min), 40 °C/min, 240 °C (5 min) (compounds 5 to 11 see Table 1). Helium was used as a carrier gas at a constant column head pressure of 135 kPa. The mass spectrometer was operated in the single ion monitoring mode with an ionization potential of 70 eV. All SPME-GC-MS measurements were run in triplicate.

Results and Discussion

Particle size distribution of ground coffee

Figure 1 shows the particle size distributions of ground coffees. It is clearly visible that wet ground coffee contained a high fraction of very fine particles ($<100 \mu\text{m}$) that was nearly absent in dry ground coffee. The coffee ground with the Ditting coffee mill exhibited a relatively small peak at average particle size of around 300 μm . Grinding with the Bühler-Miag mill resulted in particles of average size of around 900 μm . Among dry grinding methods, the laboratory blender produced the largest particles and the broadest distribution, because of the rather inadequate comminution technique using a rotating knife only. Wet grinding methods exhibited a high fraction of very small particles of around 100 μm , which was the major part in ground coffees from the corundum stone mill. The blender produced very small particles as well, but the main fraction was relatively coarse (around 1000 μm) as seen with dry grinding. Compared to dry grinding, wet grinding with the blender resulted in a more uniform particle distribution due to better mixing and the increased contact of coffee with the rotating knife. Wet grinding using the corundum stone mill yielded more very fine particles when using cold water compared to grinding with hot water.

Aroma concentration in ground coffee

To assess the impact of wet grinding methods on the aroma retention in coffee, the following aroma compounds were chosen: dimethyl sulfide (cabbage-like), hexanal (grassy), 2-ethyl-3,5-dimethylpyrazine and 2-ethyl-3,6-dimethylpyrazine (earthy, roasty), 2-methylbutanal (cocoa), 3-methylbutanal (malty), 2,3-butanedione (buttery), 2,3-pentanedione (buttery), pyridine (pungent, pyridine-like), dimethyl trisulfide (sulfury, cabbage-like), and 4-vinylguaiacol (spicy, clove-like). Most of these compounds are of high importance for the coffee aroma (Blank and others 1992). The others are used as specific marker compounds: dimethyl sulfide for freshness (Poisson and others 2006), hexanal for lipid oxidation (Baggenstoss and others 2007), and pyridine as a relatively stable process marker (Baggenstoss and others 2007).

Table 3 and 4 summarize concentrations of aroma compounds obtained from the various grinding methods. Comparisons between wet and dry grinding methods are illustrated in Figure 2. The wet grinding methods were compared to the dry grinding methods with as similar particle size distributions as possible. The coffees obtained from wet grinding with the corundum stone mill were compared to dry ground coffees from the Ditting mill. The wet ground coffees obtained from the laboratory blender were compared to the dry ground coffees from the same laboratory blender or, for investigations on the hot extraction of cold wet and dry ground coffee, from the Bühler-Miag mill. However, as seen earlier, the very fine fraction obtained from wet grinding could not be reproduced in dry grinding of coffee.

Cold grinding with the blender and the corundum stone mill both resulted in significantly higher amounts of dimethyl trisulfide, 2-methylbutanal, 3-methylbutanal, 2,3-butanedione, and 2,3-pentanedione when using wet grinding methods. In contrast, for dimethyl sulfide, hexanal and the less-volatile compounds 4-vinylguaiacol, pyridine, 2-ethyl-3,5-dimethylpyrazine, and 2-ethyl-3,6-dimethylpyrazine, differences between dry and wet grinding methods were small and not significant.

It is known that during conventional grinding, substantial amounts of volatile substances are released (Akiyama and others 2005). Water may help to retain the volatile compounds that

otherwise would be lost during grinding. It was supposed that release of volatile aroma compounds during grinding was closely connected to release of carbon dioxide (Nicoli and others 1993; Schenker 2000). To test this hypothesis, completely degassed coffee was ground conventionally and with water using the corundum stone mill. It was expected that, after complete degassing, the major part of the remaining aroma compounds was bound within the coffee bean and only a very small part remained in gas phase within the cells. Therefore, only minor differences would be observed comparing the resulting concentrations of aroma compounds from wet and dry grinding. Since complete degassing of coffee beans can take several months (Radtke 1975), the coffee subjected to the experiment was stored for 11 mo before grinding. To minimize aroma loss by oxidative degradation, storage took place in a valve bag under nitrogen atmosphere. Figure 2 shows that differences between the concentrations of aroma compounds from wet and dry grinding were small. Only hexanal was found in significant higher amounts in the wet ground coffee, whereas dimethyl sulfide was found in significantly lower amounts after wet grinding. All other analyzed compounds did not exhibit significant differences. This is an indication that higher aroma recovery from wet grinding of freshly roasted coffee is indeed caused by the trapping of volatiles and not by a better extraction because of generation of smaller particles.

The recovery of ground coffee from water used in grinding is not practicable because of the high solubility of coffee in water. Therefore, reasonable applications of wet grinding would involve the use of the resulting coffee suspension for a subsequent extraction process. To combine grinding and extraction in one operation, wet grinding with the corundum stone mill was carried out with hot water and compared to dry grinding with the Ditting mill. The advantages of the wet grinding method with regard to the retention of aroma compounds were relinquished when hot water was applied in the grinding process (Figure 2). In addition, compared to the cold grinding processes, absolute concentrations of several highly volatile aroma compounds (dimethyl sulfide, 2-methylbutanal, 3-methylbutanal, 2,3-butanedione, and 2,3-pentanedione) were substantially diminished in both hot wet and dry grinding (Table 4). It is assumed that because of the long contact time of hot water with ground coffee—the process took more than 5 min until grinding and sampling was complete—considerable losses of highly volatile compounds took place in the open grinding and sampling system.

As grinding with hot water led to losses of aroma compounds, an approach where coffee was ground with cold water and then extracted with hot water in a closed system seemed to be more promising. Therefore, a two-step process involving cold grinding with subsequent hot extraction by heating the coffee suspension

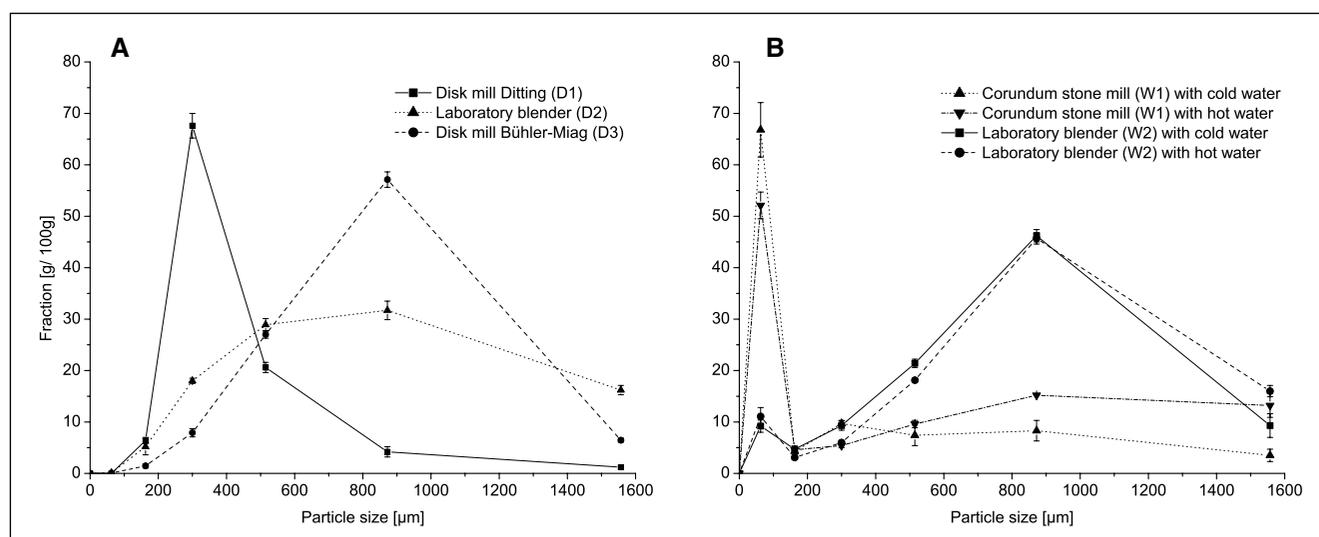


Figure 1—Particle size distribution of dry ground (A) and wet ground (B) coffee. The grinding methods are summarized in Table 2.

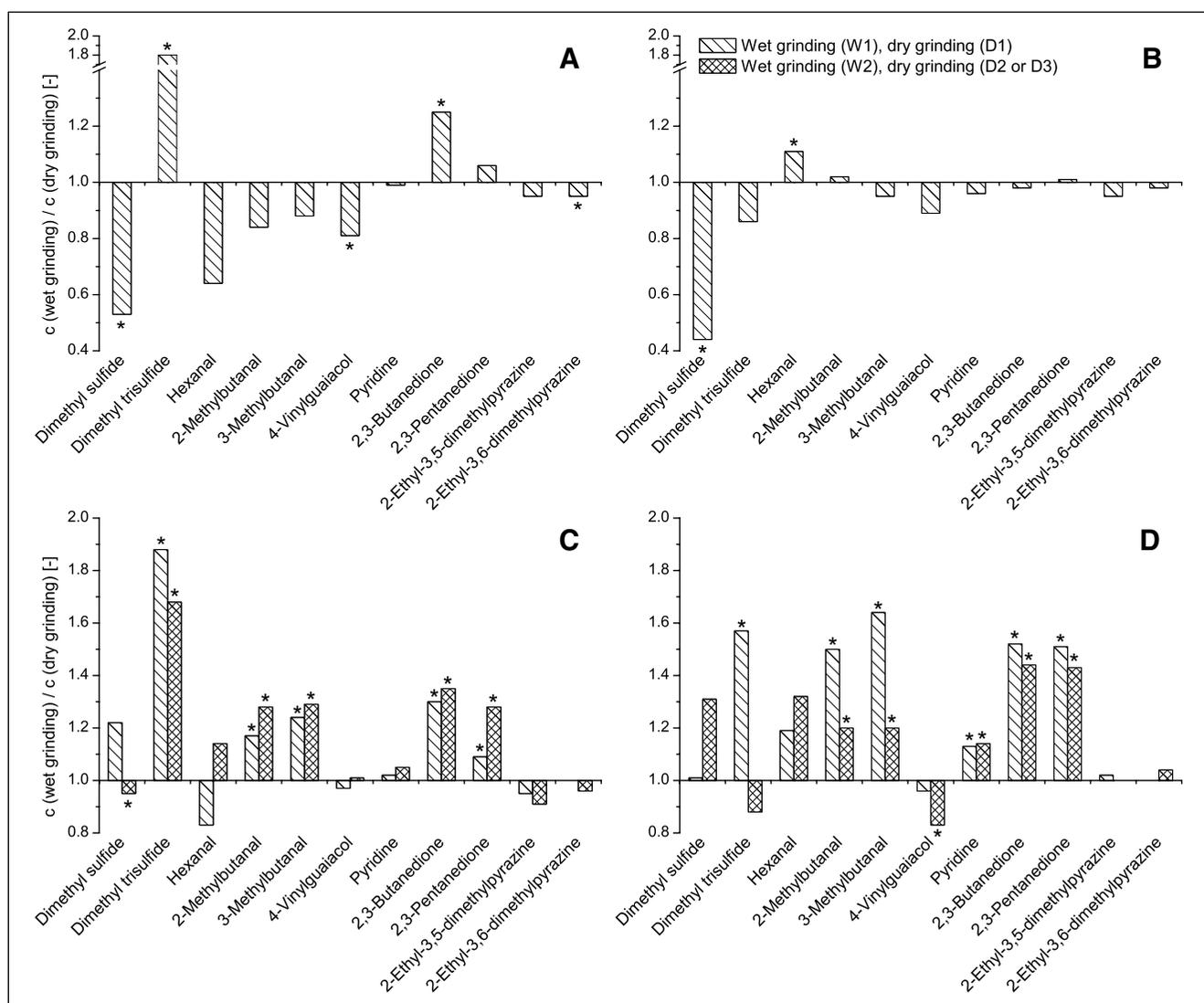
Table 3—Absolute concentrations of selected aroma compounds in dry and wet ground coffee (wet-grinding experiments with cold water).

Compound	Aroma concentration for grinding method [mg/kg dm] ^a					
	Cold grinding of freshly roasted coffee		Cold grinding of freshly roasted coffee		Cold grinding of degassed coffee	
	W1	D1	W2	D2	W1	D1
Dimethyl sulfide [mg/kg dm]	1.4 ± 0.1	1.18 ± 0.05	1.14 ± 0.04	1.2 ± 0.1	0.13 ± 0.01	0.22 ± 0.03
Dimethyl trisulfide [mg/kg dm]	0.23 ± 0.02	0.12 ± 0.01	0.21 ± 0.03	0.12 ± 0.02	0.26 ± 0.02	0.30 ± 0.01
Hexanal [mg/kg dm]	0.97 ± 0.03	1.2 ± 0.1	0.9 ± 0.2	0.78 ± 0.04	0.37 ± 0.01	0.33 ± 0.01
2-methylbutanal [mg/kg dm]	24.7 ± 0.5	21.0 ± 0.5	26 ± 1	20.7 ± 0.4	16.7 ± 0.7	16 ± 1
3-methylbutanal [mg/kg dm]	15 ± 1	12.1 ± 0.3	15.4 ± 0.6	11.9 ± 0.5	7.54 ± 0.07	8.0 ± 0.4
4-vinylguaiacol [mg/kg dm]	27 ± 1	28 ± 2	23.3 ± 0.5	23.0 ± 0.6	21.7 ± 0.6	24 ± 3
Pyridine [mg/kg dm]	144 ± 1	142 ± 5	152 ± 13	146 ± 9	169 ± 3	176 ± 5
2,3-butanedione [mg/kg dm]	25.0 ± 0.5	19.2 ± 0.6	27 ± 1	19.6 ± 0.6	16.2 ± 0.4	16.6 ± 0.3
2,3-pentanedione [mg/kg dm]	15.8 ± 0.5	14.5 ± 0.4	18.5 ± 0.9	14 ± 1	8.2 ± 0.3	8.2 ± 0.1
2-ethyl-3,5-dimethylpyrazine [mg/kg dm]	3.2 ± 0.2	3.32 ± 0.07	3.6 ± 0.3	3.9 ± 0.2	4.4 ± 0.1	4.6 ± 0.1
2-ethyl-3,6-dimethylpyrazine [mg/kg dm]	1.08 ± 0.05	1.08 ± 0.01	1.35 ± 0.08	1.4 ± 0.1	1.27 ± 0.04	1.30 ± 0.04

^aGrinding methods according to Table 2.

Table 4—Absolute concentrations of selected aroma compounds in dry and wet ground coffee (wet grinding experiments with hot water, and wet-grinding experiments with cold water and subsequent hot extraction).

Aroma compound	Aroma concentration for grinding method [mg/kg dm] ^a					
	Hot grinding		Cold grinding and hot extraction		Cold grinding and hot extraction	
	W1	D1	W1	D1	W2	D3
Dimethyl sulfide [mg/kg dm]	0.5 ± 0.1	1.0 ± 0.2	0.8 ± 0.1	0.81 ± 0.03	1.3 ± 0.1	1.2 ± 0.6
Dimethyl trisulfide [mg/kg dm]	0.11 ± 0.01	0.06 ± 0.01	0.20 ± 0.03	0.130 ± 0.004	0.10 ± 0.01	0.11 ± 0.01
Hexanal [mg/kg dm]	0.52 ± 0.03	0.8 ± 0.2	1.2 ± 0.2	1.01 ± 0.06	1.0 ± 0.2	0.8 ± 0.1
2-methylbutanal [mg/kg dm]	8 ± 1	10 ± 1	23 ± 2	15 ± 1	23 ± 1	18.9 ± 0.4
3-methylbutanal [mg/kg dm]	5.2 ± 0.7	5.9 ± 0.7	15 ± 1	9.0 ± 0.9	14.2 ± 0.7	11.8 ± 0.5
4-vinylguaiacol [mg/kg dm]	22 ± 2	27 ± 2	21.8 ± 0.7	22.9 ± 0.7	20 ± 2	23.7 ± 0.3
Pyridine [mg/kg dm]	124 ± 1	125 ± 3	136 ± 3	120 ± 3	131 ± 5	115 ± 3
2,3-butanedione [mg/kg dm]	18 ± 1	14 ± 1	23.9 ± 0.7	15.7 ± 0.5	24.6 ± 0.9	17 ± 2
2,3-pentanedione [mg/kg dm]	9.2 ± 0.7	8.7 ± 0.8	13.1 ± 0.4	8.7 ± 0.4	13.8 ± 0.8	9.6 ± 0.3
2-ethyl-3,5-dimethylpyrazine [mg/kg dm]	3.1 ± 0.1	3.26 ± 0.09	3.36 ± 0.02	3.3 ± 0.1	3.2 ± 0.3	3.2 ± 0.5
2-ethyl-3,6-dimethylpyrazine [mg/kg dm]	1.08 ± 0.01	1.13 ± 0.02	1.14 ± 0.03	1.14 ± 0.04	1.08 ± 0.09	1.0 ± 0.2

^aGrinding methods according to Table 2.**Figure 2—Relative comparison between wet and dry grinding methods. (A) Hot grinding (W1 compared to D1). (B) Cold grinding of degassed coffee (W1 compared to D1). (C) Cold grinding (W1 compared to D1, and W2 compared to D2). (D) Cold grinding with subsequent hot extraction (W1 compared to D1, and W2 compared to D3). Significant differences between wet and dry ground coffees are marked with an asterisk. The grinding methods are summarized in Table 2.**

with microwaves was developed as a step toward a possible industrial application of wet grinding. The grinding trials were carried out with both the blender and the corundum stone mill. For comparison with dry grinding methods, the coffee was ground with the disk mills (Ditting and Buhler-Miag), suspended in water, and directly extracted by microwave heating in closed flasks to minimize losses of odorants. Figure 2 shows that, after wet grinding with blender and corundum stone mill, concentrations of 2-methylbutanal, 3-methylbutanal, pyridine, 2,3-butanedione, and 2,3-pentanedione were significantly higher compared to those from the respective dry grinding method. The concentration of dimethyl trisulfide was significantly higher in wet ground coffee with the corundum stone mill. The amount of 4-vinylguaiacol was significantly lower after wet grinding with the blender, while for concentrations of dimethyl sulfide, hexanal, 2-ethyl-3,5-dimethylpyrazine, and 2-ethyl-3,6-dimethylpyrazine, no significant differences were found.

Conclusions

The results of the present investigation show that it is possible to minimize the loss of aroma compounds released during grinding by using wet grinding processes. The close relationship of internal carbon dioxide pressure with aroma stripping during grinding was demonstrated by wet and dry grinding of stored, completely degassed coffee. Due to the high solubility of roasted coffee even in cold water, the applications of wet grinding are restricted to processes with an extraction step after grinding. Coffee grinding with hot water in an open system, intended to combine grinding and extraction, resulted in high loss of aroma compounds. Therefore, a two-step process involving a grinding step with cold water and an extraction step with hot water in a closed system was proposed. Applying this approach, a higher recovery of aroma compounds was obtained.

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