

Changes in Volatile Compounds and Overall Aroma Profile during Storage of Coffee Brews at 4 and 25 °C

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In this work, the chemical changes occurring in the volatile fraction of Arabica coffee brews during storage at 4 and 25 °C for 30 days have been characterized for the first time by means of HS-GC-MS. A total of 47 compounds were identified and quantified: 2 sulfur compounds, 7 aldehydes, 3 esters, 15 furans, 5 ketones, 1 alcohol, 2 thiophenes, 4 pyrroles, 1 pyridine, 5 pyrazines, 1 alkene, and 1 acid. No new volatile compounds were detected at the end of the storage time. The changes observed are, in general, slower and less pronounced at refrigeration temperature. Storage also affects the sensory characteristics of the stored coffee brews, which lose part of their aroma intensity and freshness, acquiring some nondesirable notes such as rancid aroma, mainly during storage at 25 °C. Furthermore, seven aroma indices have been proposed as indicators of coffee brew staling, which show a good correlation with some sensory attributes, not only for aroma but also overall sensory quality. Consequently, they could be considered useful to monitor both the “age” and the sensory quality of stored coffee brews.

KEYWORDS: Coffee; coffee brew; aroma; headspace analysis; GC-MS; storage; temperature; aroma indices; sensory analysis

INTRODUCTION

Sensory quality is the ultimate criterion for the acceptance of food products. Moreover, one of the most contributory factors for the high acceptability of coffee by consumers is its aroma, which involves more than 800 volatile compounds. However, it is well-known that the aroma of coffee brews changes very quickly after its preparation, resulting in a loss of quality. This matter becomes especially important when trying to obtain a ready-to-drink coffee brew, destined to be stored for a period of time before consumption, without loss of its sensory characteristics. Although the causes of the changes that lead to the deterioration of coffee brews are not completely understood, Holscher and Steinhart (1) suggested two mechanisms for staling: loss of low-boiling potent aroma components, particularly sulfur-containing key odorants responsible for the fresh aroma, partly explained by interactions with nonvolatile compounds, such as melanoidins (2–5); and oxidative reactions, the latter being responsible for off-flavor formation.

The changes in the volatile compounds of roasted coffee during storage and the influence of the temperature and other conditions have been widely studied (1, 6–11), but the same is not true for coffee brews. Thus, to the best of our knowledge,

only Steinhart et al. (12) have investigated the changes occurring in the volatile fraction of a coffee brew kept at a high temperature (80 °C) for a short period of time (4.5 h). However, the results of this study cannot be extrapolated to coffee brews stored at lower temperatures for a longer period, because previous investigations have revealed that some changes occurring at high temperatures are not observed at lower ones (13). Moreover, the work of Steinhart et al. (12) was focused on only 14 compounds, whereas there are other volatile compounds that contribute to the coffee aroma (14–16).

On the other hand, the study of the chemical changes of a stored coffee brew should be completed with the knowledge of how these changes affect the sensory properties of the brews. In fact, in the coffee industry, sensory profiling is still the most widespread technique employed to evaluate the final quality of both raw material and finished products. Several research groups have tried to associate coffee staling with chemical changes in roasted coffee, obtaining the ratios between certain pairs of volatile compounds, called aroma indices, which have been used as indicators of coffee storage time (6, 17–19). Although most of the studies on coffee staling deal with whole coffee beans or ground coffee, Steinhart et al. (12) found that some indices which are valid for the roasted coffee could be transferable to monitor the aging of the coffee brew at high temperature (80 °C).

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Table 1. Evolution of the Area ($\times 10^{-3}$) of the Volatile Compounds Identified in the Headspace of Coffee Brews throughout Storage at 4 and 25 °C^a

QI ^b	KI ^c	storage time								
		day 0	day 1	day 3	day 7	day 10	day 15	day 20	day 30	
Sulfur Compounds										
62	671	dimethyl sulfide ^{Ad}								
	25 °C	92 ± 26 b	nd a	nd a	nd a	nd a	nd a	nd a	nd a	nd a
	4 °C	92 ± 26 b	111 ± 7 c	nd a	nd a	nd a	nd a	nd a	nd a	nd a
	SL		***							
94	1077	dimethyl disulfide ^A								
	25 °C	333 ± 35 d	281 ± 6 c	279 ± 6 c	261 ± 4 c	267 ± 6 bc	267 ± 14 bc	240 ± 6 b	149 ± 2 a	
	4 °C	333 ± 35 ab	387 ± 17 b	335 ± 23 ab	299 ± 12 a	292 ± 4 a	328 ± 14 a	285 ± 4 a	328 ± 41 a	
	SL		***	*	**	**	**	***	***	
Aldehydes										
43	645	acetaldehyde ^A								
	25 °C	670 ± 41 a	694 ± 11 a	646 ± 32 a	823 ± 53 b	855 ± 42 b	763 ± 10 ab	990 ± 75 c	1015 ± 81 c	
	4 °C	670 ± 41 a	703 ± 75 a	785 ± 55 ab	688 ± 87 a	793 ± 57 ab	779 ± 117 a	952 ± 121 b	1009 ± 141 b	
	SL		ns	*	*	ns	ns	ns	ns	
58	712	propanal ^A								
	25 °C	743 ± 73 bc	686 ± 4 ab	673 ± 3 a	771 ± 7 c	849 ± 12 d	979 ± 21 f	950 ± 15 ef	889 ± 7 de	
	4 °C	743 ± 73 ab	704 ± 64 ab	675 ± 42 ab	567 ± 57 b	695 ± 58 ab	889 ± 168 b	877 ± 80 b	881 ± 89 b	
	SL		ns	ns	*	*	ns	ns	ns	
41	747	2-methylpropanal ^A								
	25 °C	2711 ± 198 bcd	2273 ± 8 a	2301 ± 25 a	2562 ± 3 b	2778 ± 26 cde	2950 ± 42 e	2867 ± 37 de	2651 ± 33 bc	
	4 °C	2711 ± 198 cd	2615 ± 88 bc	2434 ± 87 b	1967 ± 59 a	2351 ± 83 b	2831 ± 141 cd	2885 ± 77 cd	2907 ± 75 d	
	SL		*	ns	**	**	ns	ns	**	
39	880	2-methylbutanal ^A								
	25 °C	3339 ± 253 c	2436 ± 49 a	2784 ± 35 b	2949 ± 24 b	3282 ± 18 c	3274 ± 32 c	3225 ± 13 c	2968 ± 24 b	
	4 °C	3339 ± 253 d	3194 ± 162 cd	3002 ± 99 bc	2405 ± 93 a	2849 ± 80 b	3343 ± 130 d	3316 ± 67 d	3493 ± 30 d	
	SL		***	*	**	**	ns	ns	***	
44	884	3-methylbutanal ^A								
	25 °C	4782 ± 324 d	3741 ± 0 a	3889 ± 64 ab	4172 ± 20 abc	4755 ± 271 d	4318 ± 288 bc	4542 ± 87 cd	4130 ± 87 abc	
	4 °C	4782 ± 324 cd	4347 ± 16 bc	4266 ± 234 b	3419 ± 87 a	4157 ± 236 b	4814 ± 233 cd	4767 ± 23 cd	4988 ± 191 d	
	SL		***	ns	***	*	ns	*	**	
56	1084	hexanal ^A								
	25 °C	50 ± 4 ab	45 ± 0 a	55 ± 4 abc	55 ± 4 abc	94 ± 9 d	71 ± 16 c	65 ± 4 bc	55 ± 9 abc	
	4 °C	50 ± 4 bc	48 ± 5 bc	37 ± 7 b	11 ± 5 a	67 ± 0 c	52 ± 13 bc	67 ± 15 c	65 ± 4 c	
	SL		ns	*	***	*	ns	ns	ns	
84	1102	2-methyl-2-butenal ^A								
	25 °C	22 ± 3 c	14 ± 0 b	traces a	traces a	25 ± 3 cd	28 ± 0 d	26 ± 3 d	traces a	
	4 °C	22 ± 3 de	19 ± 0 bc	14 ± 0 ab	nd a	14 ± 0 ab	23 ± 5 e	28 ± 5 e	25 ± 3 e	
	SL		ns	ns		*	ns	ns	**	
Esters										
60	682	formic acid, methyl ester ^B								
	25 °C	1172 ± 97 e	894 ± 29 cd	870 ± 6 cd	922 ± 16 d	953 ± 20 d	806 ± 22 bc	742 ± 16 b	527 ± 6 a	
	4 °C	1172 ± 97 d	1092 ± 59 cd	1016 ± 33 bc	745 ± 33 a	943 ± 33 b	1102 ± 85 cd	1137 ± 42 cd	1082 ± 45 bcd	
	SL		**	**	**	ns	**	***	***	
43	782	acetic acid, methyl ester ^B								
	25 °C	596 ± 47 b	528 ± 38 ab	483 ± 8 a	536 ± 5 ab	533 ± 6 ab	555 ± 24 ab	564 ± 34 ab	497 ± 39 a	
	4 °C	596 ± 47 c	596 ± 74 c	510 ± 11 bc	417 ± 19 a	500 ± 25 b	586 ± 51 c	591 ± 22 c	603 ± 20 c	
	SL		ns	*	***	ns	ns	ns	*	
43	1484	1-hydroxy-2-propanone acetate ^B								
	25 °C	nd a	nd a	nd a	nd a	131 ± 10 b	158 ± 22 c	nd a	nd a	
	4 °C	nd a	nd a	nd a	nd a	nd a	nd a	124 ± 26 b	nd a	
	SL					**	***	***		
Furans										
68	716	furan ^A								
	25 °C	2127 ± 244 a	1809 ± 129 a	1763 ± 226 a	1924 ± 24 a	1922 ± 258 a	1932 ± 56 a	1902 ± 121 a	1731 ± 233 a	
	4 °C	2127 ± 244 ab	2250 ± 330 ab	1958 ± 305 ab	1645 ± 208 a	1710 ± 245 a	2092 ± 419 ab	1866 ± 223 ab	2459 ± 109 b	
	SL		*	ns	ns	ns	ns	ns	**	
82	832	2-methylfuran ^A								
	25 °C	9961 ± 1208 d	7039 ± 770 bc	7467 ± 1231 c	6839 ± 90 bc	6936 ± 1010 bc	5543 ± 194 bc	5182 ± 432 b	3405 ± 518 a	
	4 °C	9961 ± 1208 a	9946 ± 1540 a	9040 ± 1682 a	6907 ± 1036 a	7202 ± 1190 a	8063 ± 1875 a	6576 ± 969 a	8124 ± 378 a	
	SL		*	ns	ns	ns	ns	ns	***	
82	858	3-methylfuran ^A								
	25 °C	572 ± 76 c	397 ± 47 b	434 ± 69 b	429 ± 6 b	442 ± 68 b	349 ± 12 b	343 ± 31 b	229 ± 37 a	
	4 °C	572 ± 76 a	553 ± 79 a	531 ± 107 a	397 ± 63 a	429 ± 76 a	483 ± 114 a	412 ± 57 a	518 ± 23 a	
	SL		*	ns	ns	ns	ns	ns	***	
96	930	2,5-dimethylfuran ^A								
	25 °C	650 ± 110 b	415 ± 61 a	485 ± 96 ab	505 ± 16 ab	561 ± 77 ab	461 ± 25 a	514 ± 0 ab	392 ± 59 a	
	4 °C	650 ± 110 a	611 ± 101 a	612 ± 121 a	468 ± 81 a	483 ± 87 a	531 ± 134 a	480 ± 81 a	636 ± 33 a	
	SL		*	ns	ns	ns	ns	ns	***	
94	1075	2-vinylfuran ^B								
	25 °C	198 ± 12 d	132 ± 6 c	139 ± 16 c	124 ± 2 c	127 ± 9 c	93 ± 3 b	91 ± 3 b	52 ± 1 a	
	4 °C	198 ± 12 b	195 ± 17 b	183 ± 23 b	138 ± 11 a	139 ± 12 a	139 ± 18 a	124 ± 9 a	150 ± 3 a	
	SL		**	ns	ns	ns	*	**	***	

Table 1. (Continued)

QI ^b	KI ^c		storage time								
			day 0	day 1	day 3	day 7	day 10	day 15	day 20	day 30	
108	1160	2-vinyl-5-methylfuran ^B									
		25 °C	286 ± 30 e	174 ± 5 d	118 ± 15 c	56 ± 2 b	56 ± 6 b	16 ± 3 a	nd a	nd a	
		4 °C	286 ± 30 f	245 ± 8 d	188 ± 25 e	100 ± 15 c	69 ± 6 bc	37 ± 9 ab	23 ± 4 a	19 ± 2 a	
		SL		***	*	**	*	*	*	*	
81	1252	2-furfuryl methyl ether ^B									
		25 °C	35 ± 2 abc	31 ± 7 ab	28 ± 2 a	31 ± 2 ab	36 ± 0 abc	38 ± 7 bc	41 ± 7 c	37 ± 4 abc	
		4 °C	35 ± 2 a	38 ± 7 a	30 ± 3 a	28 ± 7 a	32 ± 4 a	35 ± 2 a	36 ± 3 a	38 ± 2 a	
		SL		ns	ns	ns	ns	ns	ns	ns	
43	1284	2-methyltetrahydrofuran-3-one ^A									
		25 °C	90 ± 14 ab	78 ± 2 a	80 ± 5 a	90 ± 1 ab	104 ± 7 bc	111 ± 10 c	108 ± 5 c	98 ± 4 bc	
		4 °C	90 ± 14 abc	79 ± 7 ab	84 ± 5 abc	63 ± 6 a	95 ± 10 bc	129 ± 14 d	113 ± 20 cd	101 ± 11 bcd	
		SL		ns	ns	**	ns	ns	ns	ns	
96	1490	furfural (2-furancarboxaldehyde) ^A									
		25 °C	1139 ± 40 bc	976 ± 0 a	948 ± 45 a	1104 ± 21 b	1158 ± 66 bc	1163 ± 26 bc	1217 ± 48 c	1012 ± 16 a	
		4 °C	1139 ± 40 b	978 ± 96 b	973 ± 65 b	794 ± 58 a	1085 ± 55 b	1141 ± 52 b	1315 ± 107 c	1087 ± 88 b	
		SL		ns	ns	**	ns	ns	ns	ns	
81	1516	2-furfuryl methyl sulfide ^A									
		25 °C	42 ± 4 c	34 ± 4 c	21 ± 3 b	nd a	nd a	nd a	nd a	nd a	
		4 °C	42 ± 4 c	41 ± 0 c	28 ± 4 b	nd a	nd a	nd a	nd a	nd a	
		SL		ns	*						
95	1536	2-acetylfuran ^A									
		25 °C	95 ± 13 ab	87 ± 3 a	86 ± 6 a	97 ± 4 ab	104 ± 8 abc	110 ± 3 bc	116 ± 7 c	95 ± 4 ab	
		4 °C	95 ± 13 bc	87 ± 10 abc	84 ± 5 ab	71 ± 7 a	101 ± 7 bcd	107 ± 9 cd	122 ± 12 d	97 ± 8 bc	
		SL		ns	ns	**	ns	ns	ns	ns	
81	1559	2-furfuryl acetate ^A									
		25 °C	358 ± 65 f	327 ± 0 f	269 ± 11 e	225 ± 8 de	186 ± 11 cd	136 ± 8 bc	102 ± 3 ab	49 ± 0 a	
		4 °C	358 ± 65 c	352 ± 42 bc	269 ± 22 abc	247 ± 42 ab	233 ± 41 a	223 ± 15 a	289 ± 0 abc	242 ± 65 a	
		SL		ns	ns	ns	***	***	*	*	
110	1605	5-methylfurfural ^A									
		25 °C	456 ± 24 abc	424 ± 0 ab	408 ± 21 a	461 ± 12 abc	494 ± 52 bcd	522 ± 24 cd	550 ± 41 d	442 ± 21 ab	
		4 °C	456 ± 24 abc	419 ± 67 ab	396 ± 34 ab	346 ± 35 a	486 ± 45 bc	507 ± 48 bc	588 ± 81 c	432 ± 43 ab	
		SL		ns	ns	**	ns	ns	ns	ns	
91	1636	2-furfurylfuran ^B									
		25 °C	77 ± 10 d	68 ± 0 d	50 ± 4 c	45 ± 4 bc	45 ± 4 bc	34 ± 0 b	43 ± 8 bc	7 ± 0 a	
		4 °C	77 ± 10 c	58 ± 5 b	47 ± 0 b	47 ± 0 b	50 ± 4 b	50 ± 4 b	52 ± 4 b	34 ± 0 a	
		SL		**	ns	ns	ns	*	ns	**	
98	1686	furfuryl alcohol ^A									
		25 °C	nd a	709 ± 27 bc	nd a	663 ± 24 b	838 ± 128 cd	878 ± 11 de	988 ± 69 e	758 ± 72 bcd	
		4 °C	nd a	686 ± 154 bc	648 ± 11 bc	498 ± 95 b	934 ± 48 d	798 ± 169 cd	nd a	585 ± 123 bc	
		SL		ns	***	*	ns	ns	**	ns	
Ketones											
58	753	2-propanone ^A									
		25 °C	1169 ± 95 b	1042 ± 3 a	1032 ± 25 a	1171 ± 12 b	1290 ± 7 c	1436 ± 23 e	1400 ± 19 d	1327 ± 12 cd	
		4 °C	1169 ± 95 bcd	1129 ± 81 bc	1020 ± 42 b	817 ± 45 a	1093 ± 51 b	1368 ± 141 d	1359 ± 70 d	1301 ± 67 cd	
		SL		ns	ns	***	*	ns	ns	ns	
43	866	2-butanone ^A									
		25 °C	396 ± 28 bc	369 ± 1 ab	358 ± 5 a	399 ± 7 cd	426 ± 3 d	463 ± 14 e	484 ± 6 e	480 ± 11 e	
		4 °C	396 ± 28 bc	366 ± 34 b	373 ± 35 b	301 ± 26 a	361 ± 8 b	424 ± 23 bc	441 ± 13 c	421 ± 17 bc	
		SL		ns	ns	**	***	ns	**	**	
43	962	2,3-butanedione ^A									
		25 °C	458 ± 42 e	381 ± 6 d	376 ± 12 cd	337 ± 8 c	356 ± 11 cd	228 ± 15 b	245 ± 4 b	141 ± 0 a	
		4 °C	458 ± 42 c	413 ± 53 c	400 ± 27 c	364 ± 32 abc	404 ± 41 c	364 ± 25 abc	377 ± 40 bc	274 ± 48 a	
		SL		ns	ns	ns	ns	**	**	**	
43	1058	2,3-pentanedione ^A									
		25 °C	740 ± 63 e	588 ± 23 d	603 ± 40 d	453 ± 7 c	454 ± 11 c	271 ± 2 b	235 ± 24 b	113 ± 0 a	
		4 °C	740 ± 63 f	714 ± 61 ef	668 ± 23 def	485 ± 42 bc	599 ± 11 cde	497 ± 54 bc	558 ± 70 bcd	363 ± 38 a	
		SL		**	ns	ns	ns	**	**	***	
43	1323	1-hydroxy-2-propanone ^B									
		25 °C	145 ± 21 c	107 ± 7 ab	105 ± 7 ab	95 ± 10 a	137 ± 10 ab	129 ± 23 c	138 ± 9 c	132 ± 0 bc	
		4 °C	145 ± 21 c	110 ± 8 abc	131 ± 11 bc	77 ± 13 a	140 ± 17 bc	136 ± 34 bc	139 ± 17 bc	96 ± 18 ab	
		SL		ns	*	ns	ns	ns	ns	ns	
Alcohols											
57	1509	2-ethyl-1-hexanol ^A									
		25 °C	153 ± 36 c	87 ± 8 b	66 ± 3 b	nd a	72 ± 3 b	125 ± 29 c	nd a	74 ± 7 b	
		4 °C	153 ± 36 c	90 ± 12 b	nd a	nd a	nd a	132 ± 7 c	73 ± 4 b	73 ± 4 b	
		SL		ns	**	**	ns	***	ns	ns	
Thiophenes											
84	1021	thiophene ^A									
		25 °C	202 ± 11 c	155 ± 5 ab	174 ± 10 b	163 ± 0 ab	171 ± 11 b	172 ± 8 b	164 ± 4 ab	149 ± 12 a	
		4 °C	202 ± 11 cd	216 ± 13 d	176 ± 18 abc	154 ± 9 a	150 ± 9 a	187 ± 21 abc	160 ± 11 ab	200 ± 4 cd	
		SL		***	ns	ns	ns	ns	**	**	

Table 1. (Continued)

QI ^b	KI ^c	storage time								
		day 0	day 1	day 3	day 7	day 10	day 15	day 20	day 30	
97	1097	2-methylthiophene ^A								
		25 °C	102 ± 7 c	77 ± 4 b	81 ± 11 b	77 ± 3 b	86 ± 5 b	73 ± 2 b	73 ± 5 b	55 ± 6 a
		4 °C	102 ± 7 b	101 ± 8 b	87 ± 14 ab	75 ± 9 a	73 ± 7 a	81 ± 11 ab	75 ± 9 a	95 ± 3 ab
	SL		**	ns	ns	ns	ns	ns	**	
Pyrroles										
81	1149	1-methylpyrrole ^A								
		25 °C	429 ± 28 d	305 ± 18 c	219 ± 2 b	28 ± 5 a	traces a	nd a	nd a	nd a
		4 °C	429 ± 28 f	437 ± 9 f	311 ± 12 e	169 ± 2 d	121 ± 0 c	60 ± 5 b	traces a	nd a
	SL		***	***	***	**	*			
80	1194	1-ethyl-1H-pyrrole ^B								
		25 °C	34 ± 4 c	28 ± 3 b	traces a	nd a	nd a	nd a	nd a	nd a
		4 °C	34 ± 4 c	40 ± 3 d	21 ± 4 b	nd a	nd a	nd a	nd a	nd a
	SL		**	**						
67	1542	1H-pyrrole ^A								
		25 °C	57 ± 12 b	49 ± 6 b	traces a	nd a	nd a	nd a	nd a	nd a
		4 °C	57 ± 12 c	75 ± 9 d	28 ± 4 b	nd a	nd a	nd a	nd a	nd a
	SL		**	**						
81	1833	N-furfurylpyrrole ^B								
		25 °C	43 ± 0 e	36 ± 2 d	nd a	14 ± 3 c	7 ± 2 b	nd a	nd a	nd a
		4 °C	43 ± 0 e	37 ± 8 e	27 ± 3 d	25 ± 3 d	23 ± 3 cd	17 ± 3 c	5 ± 2 b	nd a
	SL		ns	**	*	**	**	*		
Pyridines										
79	1203	pyridine ^A								
		25 °C	462 ± 136 c	237 ± 18 ab	220 ± 42 ab	172 ± 7 a	224 ± 68 ab	322 ± 3 b	261 ± 40 ab	178 ± 32 a
		4 °C	462 ± 136 c	233 ± 78 ab	140 ± 38 a	141 ± 38 a	249 ± 102 ab	210 ± 0 ab	328 ± 70 bc	103 ± 10 a
	SL		ns	ns	ns	ns	***	ns	ns	
Pyrazines										
80	1231	pyrazine ^A								
		25 °C	nd a	44 ± 0 bc	36 ± 7 b	36 ± 3 b	43 ± 12 bc	58 ± 9 c	54 ± 9 c	43 ± 4 bc
		4 °C	nd a	51 ± 9 c	23 ± 4 b	21 ± 0 b	47 ± 7 c	61 ± 9 c	57 ± 16 c	22 ± 2 a
	SL		ns	*	*	ns	ns	ns	**	
94	1288	2-methylpyrazine ^A								
		25 °C	418 ± 74 c	296 ± 3 abc	269 ± 22 a	272 ± 15 a	316 ± 65 abc	388 ± 20 bc	380 ± 52 abc	296 ± 26 abc
		4 °C	418 ± 74 b	292 ± 76 ab	212 ± 43 a	196 ± 38 a	316 ± 58 ab	358 ± 68 ab	403 ± 96 b	247 ± 65 ab
	SL		ns	ns	*	ns	ns	ns	ns	
108	1347	2,5-dimethylpyrazine ^A								
		25 °C	50 ± 2 bc	45 ± 1 abc	39 ± 3 a	41 ± 1 a	45 ± 5 abc	51 ± 4 c	52 ± 5 c	42 ± 2 ab
		4 °C	50 ± 2 bc	46 ± 7 abc	37 ± 3 ab	31 ± 4 a	45 ± 5 abc	51 ± 6 bc	55 ± 9 c	38 ± 6 ab
	SL		ns	ns	*	ns	ns	ns	ns	
108	1353	2,6-dimethylpyrazine ^A								
		25 °C	69 ± 19 c	32 ± 6 ab	28 ± 6 ab	24 ± 0 a	36 ± 11 ab	48 ± 0 abc	52 ± 11 bc	29 ± 6 ab
		4 °C	69 ± 19 d	26 ± 3 ab	4 ± 0 a	traces a	40 ± 11 bc	40 ± 11 bc	60 ± 26 bc	traces a
	SL		ns	**	*	ns	ns	ns	*	
107	1359	2-ethylpyrazine ^A								
		25 °C	59 ± 21 bc	43 ± 0 abc	39 ± 9 abc	32 ± 3 a	41 ± 0 ab	74 ± 6 c	62 ± 11 c	39 ± 4 abc
		4 °C	59 ± 21 cd	41 ± 9 bc	traces a	traces a	52 ± 6 cd	35 ± 9 bc	61 ± 26 d	18 ± 0 ab
	SL		ns	**	**	**	*	ns	**	
Others										
67	624	1,3-pentadiene ^A								
		25 °C	484 ± 19 b	284 ± 5 abc	217 ± 14 ab	205 ± 10 ab	287 ± 0 c	194 ± 19 a	367 ± 9 bc	204 ± 14 ab
		4 °C	484 ± 19 c	367 ± 0 b	284 ± 5 a	270 ± 4 a	280 ± 3 a	364 ± 24 b	317 ± 24 ab	481 ± 38 b
	SL		**	***	*	ns	***	**	***	
45	1480	acetic acid ^A								
		25 °C	369 ± 76 a	538 ± 1 bc	647 ± 129 abc	540 ± 98 ab	653 ± 148 abc	950 ± 98 c	850 ± 29 bc	727 ± 142 bc
		4 °C	369 ± 76 ab	385 ± 104 ab	563 ± 167 bc	572 ± 110 bc	496 ± 11 abc	327 ± 55 a	690 ± 138 c	630 ± 59 c
	SL		*	ns	ns	ns	***	ns	ns	

^a All values are shown as means ± standard deviations. In each row, different letters indicate statistically significant differences ($p < 0.05$) throughout the time. SL, significance level between the two storage temperatures in the same day: ns, nonsignificant ($p > 0.05$); *, significant ($p < 0.05$); **, very significant ($p < 0.01$); ***, highly significant ($p < 0.001$). nd, not detected. ^b Ion used for the quantification of the compound. ^c Kovats index calculated for the HP-Wax capillary column. ^d The reliability of the identification proposal is indicated by the following: A, mass spectrum, KI, and retention time according to standards; B, tentative identification (no reference compound available).

For all of these reasons, and taking into account the very limited knowledge of the behavior of the volatile compounds of coffee brews during storage at low temperatures, this study was conducted to monitor and characterize changes in the composition of the volatile fraction of coffee brews stored at room and refrigeration temperatures for one month by means

of HS-GC-MS. Moreover, the behavior of the volatile compounds was examined to find some aroma indices that could be good indicators of the sensory staling of Arabica coffee brews by the application of several multivariate statistical techniques. The results of this study would give us an idea about the viability of obtaining coffee brews with a long shelf life, destined for

Table 2. Compounds That Follow the Linear Regression Model with Excellent Linear Correlation Coefficients ($R^2 > 0.75$) for at Least One of the Storage Temperatures, Together with the Values of These Coefficients at Both Temperatures

compound	R^2	
	25 °C	4 °C
dimethyl disulfide	0.8211	0.1596
acetaldehyde	0.8097	0.8379
formic acid, methyl ester	0.7672	0.0198
2-methylfuran	0.8306	0.3145
3-methylfuran	0.7537	0.1109
2-vinylfuran	0.8122	0.4629
2-furfurylfuran	0.8271	0.4904
2-butanone	0.8122	0.3239
2,3-butanedione	0.9124	0.7698
2,3-pentanedione	0.9254	0.7504
1-methylpyrrole	0.5654	0.8010
N-furfurylpyrrole	0.4668	0.9224

being consumed as hot coffee brews at any time, and the possibility of the control of its quality by measuring only a few volatile compounds.

MATERIALS AND METHODS

Materials. Vacuum-packed Colombian Arabica ground roasted coffee (2.25% water content, $L^* = 19.57 \pm 0.09$) was provided by a local factory. The L^* value was analyzed by means of a tristimulus colorimeter (Chromameter-2 CR-200, Minolta, Osaka, Japan) using the D65 illuminant and the CIELab scale. The instrument was standardized against a white tile before sample measurements. Ground roasted coffee was extended in a 1 cm Petri plate, and the L^* value was measured in triplicate.

Pure reference standards of acetaldehyde, 2-methylpropanal, 2-methylbutanal, 3-methylbutanal, 2-methyl-2-butanal, 3-methylfuran, 2,5-dimethylfuran, 2-methyltetrahydrofuran-3-one, furfural (2-furancarboxaldehyde), 2-furfuryl methyl sulfide, 2-acetylfuran, 2-furfuryl acetate, 2-propanone, 2-butanone, 2,3-butanedione, 2,3-pentanedione, 2-ethyl-1-hexanol, 1-methylpyrrole, pyridine, pyrazine, 2,5-dimethylpyrazine, and 2-ethylpyrazine were purchased from ACROS (Springfield, NJ); dimethyl sulfide, dimethyl disulfide, propanal, hexanal, furan, 2-methylfuran, 5-methylfurfural, furfuryl alcohol, thiophene, 2-methylthiophene, 1H-pyrrole, 2-methylpyrazine, 2,6-dimethylpyrazine, 1,3-pentadiene, and acetic acid were obtained from Sigma-Aldrich (Steinheim, Germany).

Coffee Brew Preparation. The ground coffee packages were opened immediately before the preparation of the coffee brew to avoid aroma loss. Coffee brews were prepared from 90 g of ground roasted coffee in a water volume of 1 L, using a French press coffeemaker. Extraction time was 3 min and water temperature 90 ± 2 °C (pH 7.0). The freshly prepared coffee brews were immediately poured into 330 mL sterilized glass flasks, up to a volume of 135 mL, and hermetically closed. The filling of the flasks was carried out aseptically in a laminar flow cabin, to avoid microbiological contamination of the samples. Afterward, coffee brews were cooled with ice and stored at 4 and 25 °C for 30 days. This experiment was made in duplicate.

Volatile Compound Analysis. The profiles of volatile compounds were obtained with the method described by Sanz et al. (20), adapted to coffee brew by Maeztu et al. (21), and using static headspace–gas chromatography–mass spectrometry (SH-GC-MS).

After the flask was opened, 6 mL of a homogenized coffee brew was introduced into a 10 mL vial, which was immediately sealed with a silicone rubber Teflon cap. Each vial was equilibrated at 40 °C for 60 min in the static headspace sampler (model 7694, Agilent Technologies, Palo Alto, CA). Each vial was pressurized with carrier gas for 12 s, and 3 mL of the coffee headspace sample was injected into an HP-Wax glass capillary column (60 m \times 0.25 mm \times 0.5 μ m film thickness) in an HP 6890 gas chromatograph (Agilent Technologies). Injector temperature was 180 °C, and carrier gas was helium (1 mL/min linear speed). The oven temperature was maintained at 40 °C for

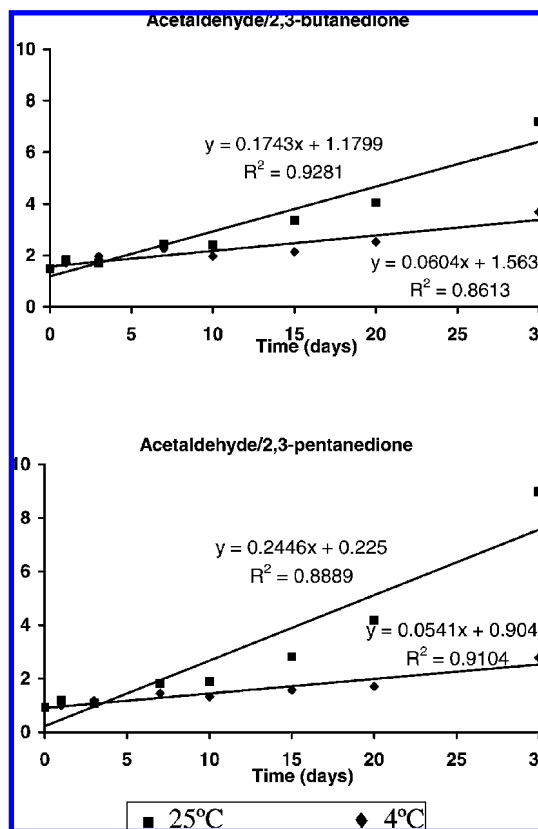


Figure 1. Aroma indices for coffee brews stored at 4 and 25 °C for 30 days.

6 min and then raised at 3 °C/min to 190 °C. Mass spectrometry analysis was performed with a Hewlett-Packard mass selective detector model 5973 (Agilent Technologies) operating in the electron impact ionization mode (70 eV), with a scan range of 33–300 amu. Ion source temperature was set at 230 °C. Each sample was analyzed in triplicate.

Identification and Quantification of the Volatile Compounds. The volatile compounds were identified by comparing their mass spectra with those of the pure reference compounds and the Wiley library and also by comparison of their Kovats indices with those of standard compounds and with data from the literature. The Kovats indices were calculated according to the method of Tranchant (22) and compared with available literature data (23). Peak areas were measured by calculation of each volatile total area based on integration of a single ion. The quantification ion of each volatile compound is given in **Table 1**.

Sensory Descriptive Analysis. Twenty judges were recruited among members of the Nutrition, Food Science, Physiology, and Toxicology Department at the University of Navarra. Selection and training were carried out as described by Maeztu et al. (21, 24) to have a 10-member panel. Although judges had experience in the sensory evaluation of coffee brews, they were retrained during four sessions to adapt their evaluation to the detection and quantification of parameters related to staling. Attention was focused on rancidity and the distinction of acidity, typical of a Colombian coffee, and sourness, typical of an old coffee brew. Reference coffee brews were prepared with a Colombian Arabica ground roasted coffee stored for 1 year for rancidity, other stored for less than 1 month for acidity and the positive aromas and flavors, and a coffee brew prepared with the same coffee but stored for 2 months for sourness. A scorecard with the most frequently perceived sensory attributes was developed during training. Two lines for “other” aromas and flavors were added. All of the descriptors were rated on 11-point scales from “none” (0) to “very high” (10).

Each coffee brew sample was heated in a microwave oven at 90 ± 2 °C immediately before tasting and served monadically in a white porcelain coffee cup. The order of presentation was randomized among sessions. A freshly prepared coffee brew was evaluated first, as a reference and to avoid first impressions. All evaluations were conducted

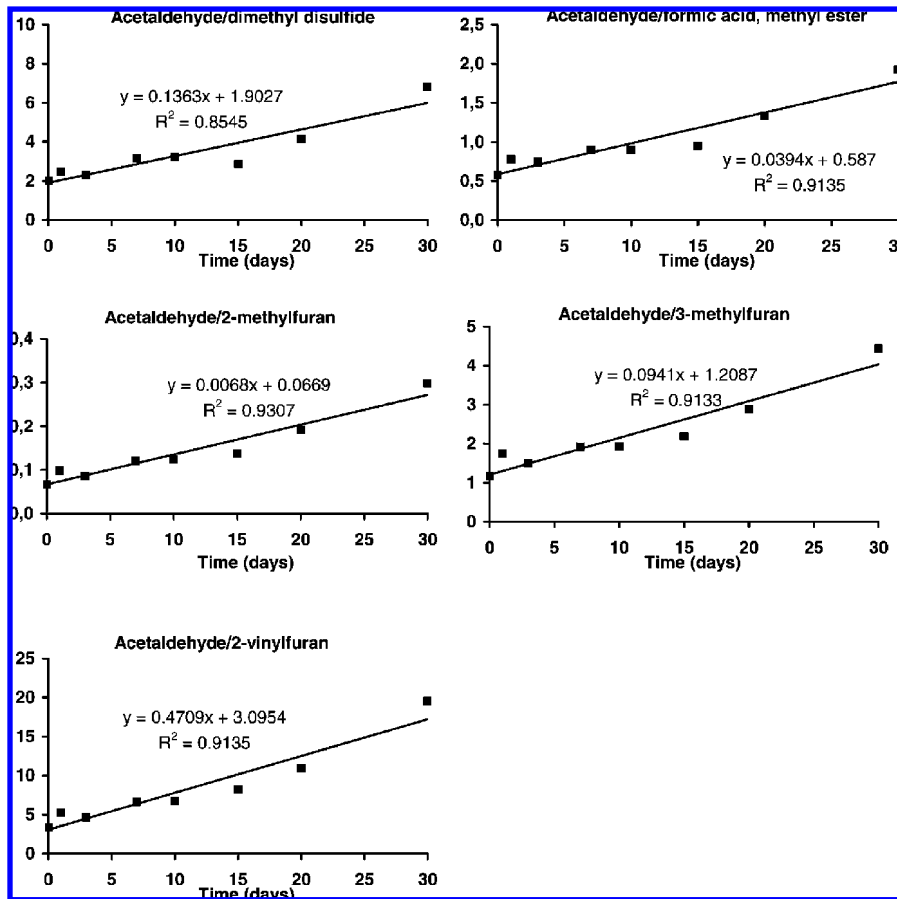


Figure 2. Aroma indices for coffee brews stored at 25 °C for 30 days.

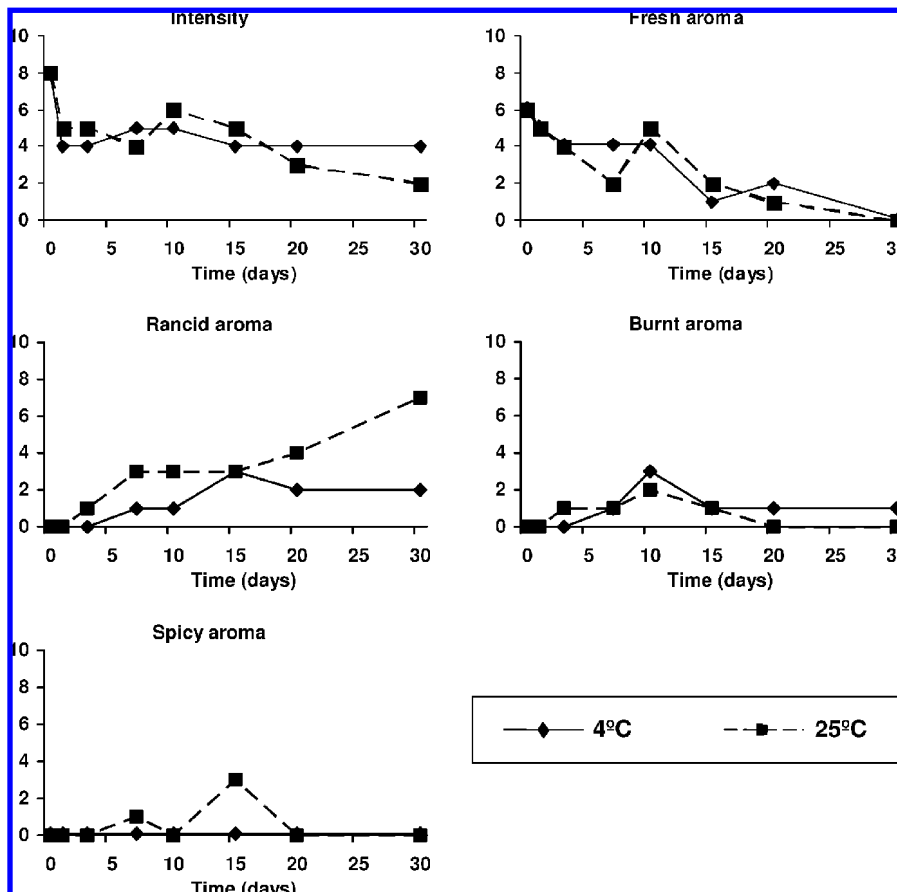


Figure 3. Changes in aroma sensory attributes of coffee brews throughout storage at 4 and 25 °C.

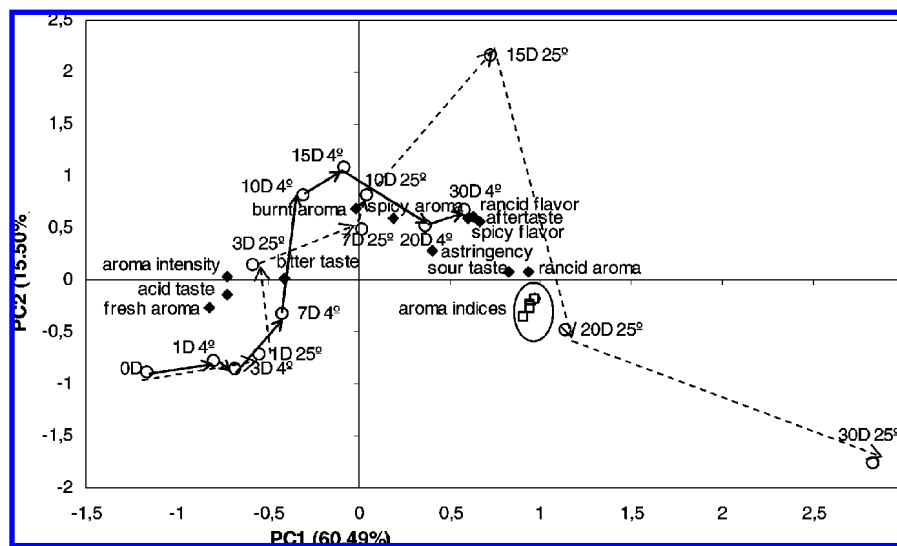


Figure 4. Principal component analysis of the coffee brew stored at 4 and 25 °C for 30 days.

Table 3. Correlations between the Aroma Indices Proposed and the Sensory Notes of Coffee Brews throughout Storage at 4 and 25°C^a

sensory note	A/B	A/P	A/D	A/F	A/2MF	A/3MF	A/2VF
aroma intensity	-0.663**	-0.617*	-0.632**	-0.686**	0.674**	-0.684**	-0.656**
fresh aroma	-0.744**	-0.665**	-0.785**	-0.662**	-0.702**	-0.693**	-0.659**
rancid aroma	0.889***	0.884***	0.901***	0.877***	0.911***	0.898***	0.918***
burnt aroma	-0.151	-0.197	-0.080	-0.104	-0.075	-0.072	-0.106
spicy aroma	0.117	0.068	0.000	0.040	0.079	0.075	0.097
acid taste	-0.652**	-0.581*	-0.610*	-0.607*	-0.648**	-0.651**	-0.623**
sour taste	0.789**	0.720**	0.708**	0.691**	0.766**	0.755**	0.740**
bitter taste	-0.473	-0.419	0.030	-0.421	-0.386	-0.373	-0.352
astringency	0.253	0.186	0.283	0.274	0.337	0.337	0.284
aftertaste	0.511*	0.444	0.526*	0.510*	0.564*	0.558*	0.539*
spicy flavor	0.491	0.459	0.450	0.427	0.506*	0.499*	0.514*
rancid flavor	0.540*	0.471	0.424	0.403	0.480	0.461	0.495

^a A/B, acetaldehyde/2,3-butanedione; A/P, acetaldehyde/2,3-pentanedione; A/D, acetaldehyde/dimethyl disulfide; A/F, acetaldehyde/formic acid, methyl ester; A/2MF, acetaldehyde/2-methylfuran; A/3MF, acetaldehyde/3-methylfuran; A/2VF, acetaldehyde/2-vinylfuran. *, **, and *** indicate significance at the 0.05, 0.01, and 0.001 probability levels, respectively.

in isolated sensory booths illuminated with white light in the sensory laboratory under standardized conditions by UNE 87-004-79 (25). Rinse water was provided between samples. After the individual evaluation of each sample, results were discussed and established by panel consensus.

Statistical Analysis. Analysis of variance (ANOVA) was applied for each storage temperature. The source of variation was the time. *T*-Tukey was applied as the test a posteriori with a level of significance of 95%. Student *t* test analysis was applied to the results of the two storage temperatures in each analysis time.

Correlations among variables were assessed by means of the Pearson correlation test. Principal component analysis (PCA), based on the Pearson correlation matrix, was applied to the obtained data of aroma attribute values, to the aroma indices, and to the taste values (13). All statistical analyses were performed using the SPSS v. 13.0 software package for Windows.

RESULTS AND DISCUSSION

Volatile Profile. A total of 47 volatile compounds were identified and quantified in the headspace of Arabica coffee brews throughout 30 days of storage at 4 and 25 °C. Table 1 shows the evolution during storage of 2 sulfur compounds, 7 aldehydes, 3 esters, 15 furans, 5 ketones, 1 alcohol, 2 thiophenes, 4 pyrroles, 1 pyridine, 5 pyrazines, 1 alkene, and 1 acid. Most of the volatiles were present in the coffee brews stored at both temperatures (4 and 25 °C).

Sulfur compounds are of high importance for the freshness of roasted coffee (1). Methanethiol, one of the key odorants responsible for the aroma freshness in ground roasted coffee (1) and in espresso coffee (21), was not detected in our samples. However, its oxidation product, dimethyl sulfide, also used as a marker compound for the aroma freshness (26, 27) of coffee brews, was detected in small amounts at the beginning, decreasing to undetectable levels during the first day in the brews stored at 25 °C and during the first 3 days in those stored at 4 °C. Dimethyl disulfide did not show significant variations throughout storage in the brews stored at 4 °C, whereas in those stored at 25 °C, a progressive and significant decrease was observed. Other authors have also observed losses of these compounds during the storage of roasted coffee beans (1, 28) and ground roasted coffee (6, 8) in air conditions.

Among the aldehydes detected, Strecker aldehydes (2-methylpropanal, 2-methylbutanal, and 3-methylbutanal), propanal, and acetaldehyde were the most abundant. Acetaldehyde and propanal, compounds related to the fruity flavor of coffee brews (14, 21), increased significantly during storage in the headspace of the brews stored at 25 °C. In those stored at 4 °C, a significant increase was observed only for acetaldehyde.

Strecker aldehydes were proposed as being responsible for the malty flavor in brewed coffee (14, 15). In general, these key odorants decreased significantly until day 1 at 25 °C and

day 7 at 4 °C, increased to days 10–15 at 25 °C and day 15 at 4 °C, and finally decreased again at 25 °C or remained practically constant at 4 °C. A decrease in Strecker aldehydes was observed by Czerny and Schieberle (9) in ground roasted coffee stored at 20 °C in the presence of oxygen; in contrast, other authors (6) reported an increase ranging from 13 to 36% in hermetically sealed ground coffee during 117 days at 37 °C.

The formation of hexanal, an odor impact compound related to rancid flavor, due to the oxidation of polyunsaturated fatty acids such as linoleic acid (29, 30), seems to have a certain influence on the staling of ground coffee. However, in the headspace of the coffee brew, hexanal remained at low levels. This could be because of the low proportion of fat in coffee brews. Thus, although this volatile compound may contribute to staling, it should not be considered as a good marker by itself in coffee brews, because rancid aroma and taste increased during storage.

Three volatile esters were identified and quantified during the storage of Arabica coffee brews. The most abundant was formic acid methyl ester, followed by acetic acid methyl ester. These esters were also the most abundant in roasted coffee (31). The ester most affected by the storage temperature was formic acid methyl ester, which exhibited a significant decrease, mainly at 25 °C. A loss of this compound was also observed by Steinhart et al. (12) in a coffee beverage after 4.5 h of hot storage at 80 °C.

Fifteen volatile furan compounds were identified in the coffee brews throughout storage, 2-methylfuran, furan, furfural (2-furancarboxaldehyde), 2,5-dimethylfuran, and 3-methylfuran being the most abundant. Numerous furans have been proposed as responsible for the burnt sugar, burnt, and caramel aromas in roasted coffee (32) and also for the caramel flavor in coffee brews (26). Furan did not exhibit significant changes throughout storage time, either at 25 or at 4 °C. With regard to 2-methylfuran, 3-methylfuran, and 2,5-dimethylfuran, nonsignificant differences were observed throughout storage in the coffee brews stored at 4 °C, whereas in those stored at 25 °C, there was a significant decrease, mainly during the first day. Other furans, such as 2-vinylfuran, 2-vinyl-5-methylfuran, 2-furfuryl acetate, and 2-furfurylfuran, showed, in general, a significant decrease at both storage temperatures, faster and more intense at 25 than at 4 °C. In contrast, storage temperature hardly seemed to affect the amounts of 2-methyltetrahydrofuran-3-one, furfural, 2-acetylfuran, and 5-methylfurfural. Therefore, it is clear that, even though the total amount of furan compounds tends to decrease during storage, not all of the compounds are affected in the same way either by time or temperature.

Within the group of ketones, special mention should be made of the significant decrease of 2,3-butanedione and 2,3-pentanedione, key odorants responsible for the buttery flavor in ground coffee (14, 32, 33) and in coffee brew (15, 21). This drop was faster and more intense in the headspace of the coffee brews stored at 25 °C, mainly from day 10. The degradation of these two diketones has also been observed during the storage of roasted coffee (1, 6, 9).

A significant decrease of thiophenes, volatile compounds present in heat-treated foods, was observed in the headspace of the coffee brews stored at 25 °C, mainly during the first day. A loss of thiophene in the presence of air in roasted coffee was also reported by Vitzthum and Werkhoff (19).

Pyroles, responsible for coffee burnt flavors (26), were some of the volatile compounds most affected by storage in coffee brews, because their amounts decreased significantly, mainly at 25 °C, during the first week. Some of the minor volatiles,

such as 1*H*-pyrrole and 1-ethyl-1*H*-pyrrole were undetectable after 1 day of storage at 25 °C or after 3 days at 4 °C.

Pyridine, another volatile compound formed during coffee roasting that contributes to the smoky flavor in coffee brews (26), exhibited a significant decrease at both storage temperatures, mainly during the first day, when it was reduced by half.

Pyrazines are related to roasty and earthy/musty flavors in ground roasted coffee and coffee brews (16, 21, 33, 34). The most abundant pyrazine in our coffee brews was 2-methylpyrazine, which showed a behavior similar to that of pyridine. However, some of the most aromatic pyrazines in coffee brews, such as 2-ethylpyrazine, 2-ethyl-6-methylpyrazine, and 2-ethyl-3,5-dimethylpyrazine (14, 21, 35), were in very small or undetectable amounts.

Among the other volatiles detected, acetic acid showed an increase throughout the storage time, which was faster at 25 °C, reaching more than twice the initial amount at 15 days.

Looking for Aroma Indices. After characterizing the volatile profile of the coffee brews at each storage time, following the mathematical method proposed by Kallio et al. (6), we tried to find some indices that could allow us to estimate the age and the quality of stored coffee brews. For this purpose, the area of each volatile compound was drawn against the storage time (data not shown). The objective was to know if the evolution of these compounds followed the linear regression model, considering appropriate for the calculation of indices only those compounds with excellent linear correlation coefficients ($R^2 > 0.75$). Only 12 volatiles complied with this criterion for at least one of the storage temperatures. As can be observed in **Table 2**, only three compounds showed R^2 values >0.75 for both storage temperatures: acetaldehyde, 2,3-butanedione, and 2,3-pentanedione. Moreover, 1-methylpyrrole and *N*-furfurylpyrrole had an R^2 value >0.75 in the coffee brews stored at 4 °C, and dimethyl disulfide, formic acid methyl ester, 2-methylfuran, 3-methylfuran, 2-vinylfuran, 2-furfurylfuran, and 2-butanone had R^2 values >0.75 in the brews stored at 25 °C.

According to Kallio et al. (6), the calculation of aroma indices requires that one compound decreases with time and the other increases at the same time. Thus, two indices were calculated by combining acetaldehyde, the only volatile for which the area rose linearly at both storage temperatures, with the other two compounds that exhibited a linear decrease, also at both temperatures (2,3-butanedione and 2,3-pentanedione). In **Figure 1**, it can be observed that these two indices (acetaldehyde/2,3-butanedione and acetaldehyde/2,3-pentanedione) follow the linear regression model with R^2 values >0.75 , being good indicators of coffee brew quality at 4 and 25 °C.

Additional indices were calculated by combination of acetaldehyde with the rest of the volatiles with $R^2 > 0.75$ for only one temperature (**Table 2**), with the exception of 2-butanone, for which the area rose, too. Among the six possible indices for the coffee brews stored at 25 °C, five (acetaldehyde/dimethyl disulfide, acetaldehyde/formic acid methyl ester, acetaldehyde/2-methylfuran, acetaldehyde/3-methylfuran, and acetaldehyde/2-vinylfuran) were found to be useful ($R^2 > 0.75$) (**Figure 2**). In contrast, neither of the potential indices for the coffee brews stored at 4 °C (acetaldehyde/1-methylpyrrole and acetaldehyde/*N*-furfurylpyrrole) complied with the linearity criterion.

Some of the compounds in **Table 2**, such as 2,3-butanedione or 2-methylfuran, have been also used by other authors to calculate aging indices for ground roasted coffee (6). However, acetaldehyde had not been previously used either for whole coffee beans or ground roasted coffee. Moreover, although the ratios methanol/2-methylfuran, methanol/2-butanone, dimethyl

sulfide/2-butanone, and dimethyl disulfide/2-butanone were found to be useful as aging indices in coffee beverages maintained at 60/80 °C (12), they do not work at room and refrigeration temperatures.

Relationship between Instrumental and Sensory Analysis in Coffee Brew Aroma. Taking into account that sensory quality is the ultimate criterion for the acceptance of food products, sensory scores of the most relevant aromas of the stored coffee brews are shown in **Figure 3**. Aroma intensity and freshness, typical of a good fresh coffee brew, tend to go down throughout storage at 25 and 4 °C. In contrast, rancid aroma started to be perceived at day 3 in the coffee brews stored at 25 °C and at day 7 in those stored at 4 °C, and the scores increased with storage time, higher and faster at 25 °C. Also, burnt aroma was first detected by the judges at days 3 and 7 in the coffee brews stored at 25 and 4 °C, respectively, reaching a maximum at day 10. The spicy aroma was perceived only in the coffee brews stored at 25 °C, at days 7 and 15.

Multivariate statistical techniques were applied to combine instrumental and sensory analyses of coffee aroma. Thus, PCA was applied to examine the efficiency of the obtained aroma indices in relation to the sensory characteristics of the coffee brews. Together with the aroma attributes, data corresponding to some taste attributes were also added to have at a glance a global vision of the changes in the coffee brew sensory attributes. The latter were taken from a previous work (13). Four principal components (PC), with eigenvalues >1, explaining 88% of the total variance of the data, were obtained. **Figure 4** shows the bidimensional representation of the scores corresponding to the two first PCs for all of the variables and coffee samples.

PC1 explained 60.49% of the total variance. Typical and good quality attributes of Colombian Arabica coffee brews, such as aroma intensity, fresh aroma, or acid taste, are on the left half of the graphic, whereas low-quality attributes, such as rancid aroma or sour taste, related to the staling of coffee brews, and aroma indices are on the right half. Burnt aroma and spicy aroma characterized PC2 (15.5%). These attributes are more typical of the coffee samples at intermediate storage times, mainly of those stored at 25 °C. When storage time increases, these notes become less perceivable, maybe because aroma intensity decreases and other low-quality sensory attributes, such as rancid aroma, increase.

During the first days, coffee brews are on the left half of the graphic, but they move to the right with the storage time, acquiring at the end of the storage sensory notes such as rancid flavor, aftertaste, spicy flavor, astringency, sour taste, and rancid aroma. It must be noted that the changes in the coffee brews stored at 4 and 25 °C follow the same behavior. However, there is a higher number of coffee samples stored at 25 °C on the right half of the graphic, showing that good sensory characteristics were lost more quickly than at 4 °C. In addition, the coffee brews stored at 25 °C were closer to the top and right extremes of the PC axes in comparison with those stored at 4 °C, indicating that changes are more intense in the former.

PCA also shows that the proposed aroma indices are very correlated with some sensory attributes. The correlations between all of the sensory notes of the coffee brews and the seven aroma indices are shown in **Table 3**. Very highly significant ($p < 0.001$) excellent ($r > 0.75$) correlations between the seven aroma indices and rancid aroma were found. Furthermore, fresh aroma showed an excellent ($r > 0.75$) very significant ($p < 0.01$) negative correlation with the acetaldehyde/dimethyl disulfide (A/D) index but only good correlation (0.5

$< r < 0.75$) with the others. Similarly, aroma intensity exhibited significant ($p < 0.05$) or very significant ($p < 0.01$) good (0.5 $< r < 0.75$) negative correlations with the seven aroma indices.

On the other hand, all of the indices proposed in this work showed very significant ($p < 0.01$) positive correlations with sour taste, these correlations being excellent ($r > 0.75$) for A/B, A/2MF, and A/3MF indices and significant ($p < 0.05$) or very significant ($p < 0.01$) good (0.5 $< r < 0.75$) negative correlations with acid taste. In addition, significant ($p < 0.05$) good (0.5 $< r < 0.75$) positive correlations were found between most of the aroma indices and aftertaste. Therefore, it could be said that the proposed aroma indices are very good indicators of the sensory staling of stored coffee brews.

In summary, in this work, the chemical changes occurring in the volatile fraction of Arabica coffee brews during storage at 4 and 25 °C for 30 days have been characterized for the first time, showing that no new volatile compounds were detected by HS-GC-MS at the end of the storage time. Furthermore, seven aroma indices are proposed as useful indicators of coffee brew staling, not only for aroma but also for overall sensory quality.

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