ELSEVIER

Food Chemistry 70 (2000) 99–106

Food Chemistry

www.elsevier.com/locate/foodchem

Analytical, Nutritional and Clinical Methods Section

Representativeness of coffee aroma extracts: a comparison of different extraction methods

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Received 19 August 1999; accepted 17 November 1999

Abstract

Analyses for the investigation of aroma components are routinely performed on coffee aromatic extracts. Various extraction methods exist. Ideally, the extraction method used should provide an extract with sensory characteristics as close as possible to the complete product. This is particularly relevant in the case of coffee, as no single key compound has been demonstrated as being responsible for the typical flavour of roasted and ground coffee. The purpose of this study was to compare various methods to see which provided an aromatic extract most representative of coffee. Five different extraction methods were compared: supercritical fluid extraction with carbon dioxide, simultaneous distillation extraction, oil recovery under pressure and vacuum steam-stripping with water or with organic solvent. In addition, Arabica Colombia coffee was used at three different roasting levels, i.e. green coffee as well as the same coffee light-roasted and medium roasted. Sensory testing of the extracts showed that vacuum steam-stripping with water provided the most representative aroma extract, for all three coffees.

Keywords: Coffee; Aroma isolation technique; Sensory analysis; Odour; Quality

1. Introduction

Studies of aroma composition of food traditionally involve aromatic extracts. It is essential that these extracts have sensory characteristics as close as possible to the complete food. Etiévant, Moio, Guichard, Langlois, Lesschaeve, Schlich and Chambellan (1994) recommended the validation of extract representativeness by preliminary sensory testing before analysis by gas chromatography–olfactometry (GC–O) and gas chromatography–mass spectrometry (GC–MS). A few groups (Abbot, Etiévant, Langlois, Lesschaeve & Issanchou, 1993; Guichard, Schlich & Issancohou, 1990; Moio, Chambellant, Lesschaeve, Issanchou, Schlich & Etiévant, 1995) have found this systematic approach to be appropriate on products such as apricot, wine and beer, respectively.

Coffee aroma is very complex, involving more than 800 volatile compounds (Maarse & Visscher, 1996) with a wide range of functional groups. In recent years, various studies have focussed on the most potent odorous constituents responsible for coffee aroma (Blank, Sen & Grosch, 1992; Grosch, 1995; Tressl, Grünewald & Silwar, 1981). Grosch et al. (1992, 1995, 1998) found that 29 volatile compounds were mainly responsible for roast and ground coffee aroma (R&G), 13 of which had a particularly important contribution. Since this work concerned only one degree of roasting and a single extraction method, we decided to compare various extraction methods on green, light and medium roast coffee, in order to try and identify one method which was suitable for a range of coffee roasts.

The following five methods were investigated: vacuum-stripping with water or organic solvent; simultaneous distillation-extraction; oil pressing and supercritical fluid extraction. The sensory evaluation of the aroma extracts allowed selection of the method providing the most representative extract. In addition, the overall composition of the aroma recovered will be discussed as a function of the extraction method used.

2. Materials and methods

2.1. Coffee sample

The same batch of Arabica Colombia coffee was used throughout this study. The beans were roasted using a

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0308-8146/00/$ - see front matter
PII: S0956-7135(99)00116-4

Neotec roaster (batch of 500 g; roasting temperature: 230°C; 2 min for light roast and 6 min for medium roast). Samples of the different roasted and green beans were packed in 100 g portions in bags of synthetic material (polyester/aluminium/polyethylene, 12/12/70). The bags were sealed under vacuum and stored at −80°C until analysis.

2.2. Analytical reagents

Purified water was prepared with a Büchi system (Labortechnik AG, Flawil, Switzerland). All solvents used (ethanol, methylene chloride) were analytical grade (Fluka, Switzerland). Smelling strips were purchased from Granger-Veyron (Privas, France). MCT oil [Delios®, $C_{8:0}$ (60%) and $C_{10:0}$ (40%) triglycerides] was used as neutral co-solvent for press oil aroma extraction. Sodium chloride (purity level 99.5%) was purchased from Merck (Darmstadt, Germany).

2.3. Methods

Aroma extracts were prepared using five different methods (Fig. 1).

2.3.1. Vacuum steam-stripping with water (VSS water)

Fifty grammes of ground coffee were placed in a 250 ml round-bottom flask. Thirty-five grammes of sodium chloride were dissolved in 100 ml of water, added to the coffee and mixed. The flask containing the mixture was frozen with liquid nitrogen for 20 min and then connected to the vacuum stripping apparatus (0.5×10^5 Pa). The stripping was carried out for 4 h at room temperature followed by 2 h at 50°C. The distillate was condensed in two cold traps cooled with liquid nitrogen. Then it was successively extracted with three portions of methylene chloride (3×33 ml). The aromatic extracts were pooled, dried over anhydrous sodium sulphate and then concentrated to 1 ml in a Kuderna-Danish apparatus.

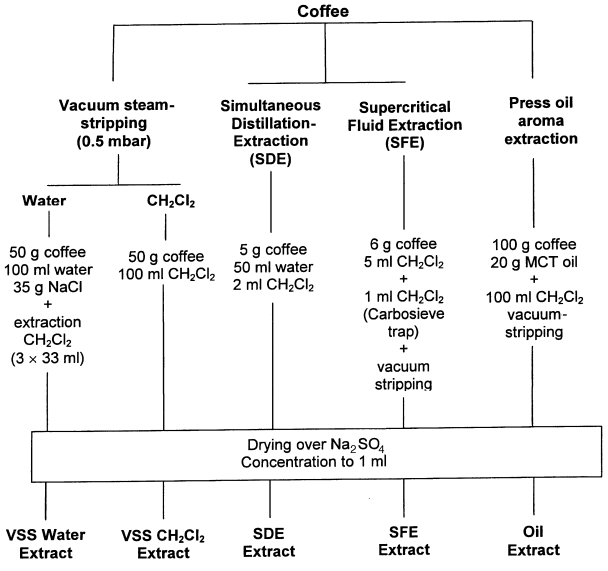

Fig. 1. Extraction methods used for the isolation of coffee aroma.

2.3.2. Vacuum steam-stripping with methylene chloride (VSS CH_2Cl_2)

The method was the same as the VSS-Water, except that the 100 ml water +35 g sodium chloride were replaced by 100 ml methylene chloride.

2.3.3. Simultaneous distillation–extraction (SDE)

Volatile compounds were isolated using a micro steam distillation apparatus, as described by Godefroot, Sandra and Verzele (1981). A mixture of ground coffee (5 g), water (50 ml) and a few drops of silicon antifoam (Siegfried Handel AG, Switzerland) was heated at 116°C. At the same time, 2 ml of methylene chloride were distilled at atmospheric pressure (63°C). The extraction was performed over 2 h (temperature of the cold finger −5°C). The aromatic extract was dried over anhydrous sodium sulphate and concentrated to 1 ml in a Kuderna-Danish apparatus.

2.3.4. Press oil aroma extraction (oil)

One hundred grammes of ground coffee were mixed with 20 g of MCT oil. The mixture was stirred for 1 h at 45°C. About 4×10^7 Pa of pressure was applied using a laboratory hydraulic press (CARVER, New Jersey, USA). After 2 h of pressing, the recovered oil was mixed with 100 ml of methylene chloride, stirred and then stripped under vacuum during 2 h at room temperature. The aromatic extract was dried over anhydrous sodium sulphate and concentrated to 1 ml in a Kuderna-Danish apparatus.

2.3.5. Supercritical fluid extraction (SFE)

A supercritical fluid extractor from ISCO (Geneva, Switzerland) was used. Six grams of ground coffee were extracted with carbon dioxide and ethanol as co-solvent (conditions of extraction: 2×10^7 Pa, 60°C and 1% ethanol). The aromatic extract (conveyed by a silica capillary tube, 50 μm ID heated to 60°C) was collected in a test tube containing 5 ml of methylene chloride. A Carbosieve™ trap (Supelco, Bellefonte, USA) fitted to the test tube collected the escaping volatile compounds. The Carbosieve™ was further desorbed with 1 ml of methylene chloride in a sonic bath for approximately 5 min. The two aromatic fractions were pooled, stripped under vacuum at room temperature, then dried over anhydrous sodium sulphate and concentrated to 1 ml in a Kuderna-Danish apparatus.

All aromatic extracts were stored in sealed vials at −80°C until evaluation.

2.4. Sensory evaluation

In spite of the different initial weight of coffee used for each method, a preliminary sensory evaluation in the

laboratory confirmed that the aromatic extracts provided a similar global odour intensity. This meant that we are able to perform a similarity test directly on the extracts. The similarity test was done by a panel of 19 assessors (7 females and 12 males) trained to evaluate coffee aroma. Samples were prepared by dipping smelling strips in the aromatic extracts, holding them in the air for 30 s to allow the solvent to evaporate and then placing each strip in a brown flask (25 ml).

For each type of coffee, a series of six coded brown flasks were presented together (one coffee reference consisting of 2 g of freshly ground coffee and five flasks for each of the five aromatic extracts in random order). Assessors opened the flask, waited 30 s, then smelled the samples.

The assessors were asked to score the samples using an unstructured 100 mm scale anchored at the right end with "far from the reference" and at the left end with "identical to the reference". In addition, they had to comment on the aroma characteristics using their own descriptors. For each of the three coffee samples (green, light and medium roast), four sessions were run: one for training and three for the evaluation.

The data acquisition and statistical treatments (statistic descriptive analysis, variance analyses and a Duncan test to threshold 5%) were performed with FIZZ software (Biosystemes, Couternon, France).

2.5. Investigation of the influence of different parameters on aroma recovery on the VSS water extract

The influence of different parameters on aroma recovery was investigated (for the VSS water method only, Fig. 2).

1. Sodium chloride impact was tested at two different stages of the aroma recovery: when mixed with the ground coffee and in the distillate as an aid to CH_2Cl_2 extraction.

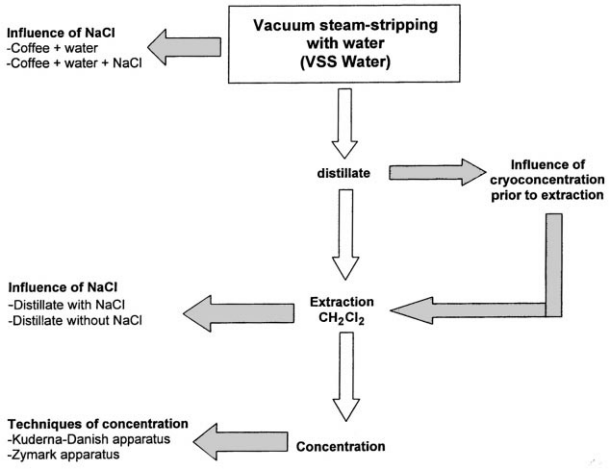

Fig. 2. Steps studied for the quantification of global aroma recovered.

2. In order to minimise the volume of organic solvent, aqueous distillates were cryoconcentrated (Langlois, Maltere & Etiévant, 1997) prior to CH_2Cl_2 extraction. The trials were performed with three different freezing temperature programmes: a constant temperature of −5°C and two temperature rates of −1°C/h and −5°C/h, respectively. 100 ml of aqueous distillate were placed in a 250 ml round-bottom flask with 3–5 carborundum pieces in order to facilitate crystallisation with a cryostat (Hahling, Aigle, Switzerland). The flask was rotated using a rotavapor at atmospheric pressure (Büchi, Labortechnik AG, Flawil, Switzerland). The final volume of aqueous distillate recovered corresponded to a concentration factor of 10 in volume.
3. The Kuderna Danish evaporator was compared with a Turbo Vap®500 concentrator. Kuderna-Danish is an evaporator-concentrator fitted with a Snyder column (Berdague, Denoyer, Le Quéré & Semon, 1991), and the Turbo Vap®500 is a concentrator (Zymark®, Brechbühler, Geneva, Switzerland) based on a vortex-assisted evaporation of the solvent at 40°C.

2.6. Gas chromatography (GC) analysis, VSS water-medium roast sample only

GC analyses were carried out using a Hewlett–Packard 5890 chromatograph equipped with a split-splitless injector and a flame ionisation detector. The detector and injector temperatures were set to 250°C. A DB-FFAP fused silica capillary column (50 m×0.32 mm; film thickness 0.35 μm; Macherey-Nagel, Dúren, Germany) was used with helium as carrier gas (36 cm/s). The oven temperature was programmed from 40 to 220°C at 8°C/min. The semi-quantification (expressed in ppm/R&G coffee) of overall global aroma for the aromatic extract was calculated from the GC-FID trace using methyl stearate as an internal standard (Fluka, Buchs, Switzerland), assuming the same response factor for all components.

3. Results and discussion

3.1. Sensory results

Fig. 3 shows the results for the mean values and Duncan tests (threshold at 5%), Tables 1 and 2 show the descriptors and Table 3 the variance analysis.

The aim of the similarity test was to compare the mean scores of each aromatic extract with the odour of the corresponding reference (reference coffee sample scores 0 on the scale). The shorter the distance to the reference, the better is the representativeness of the aromatic extract.

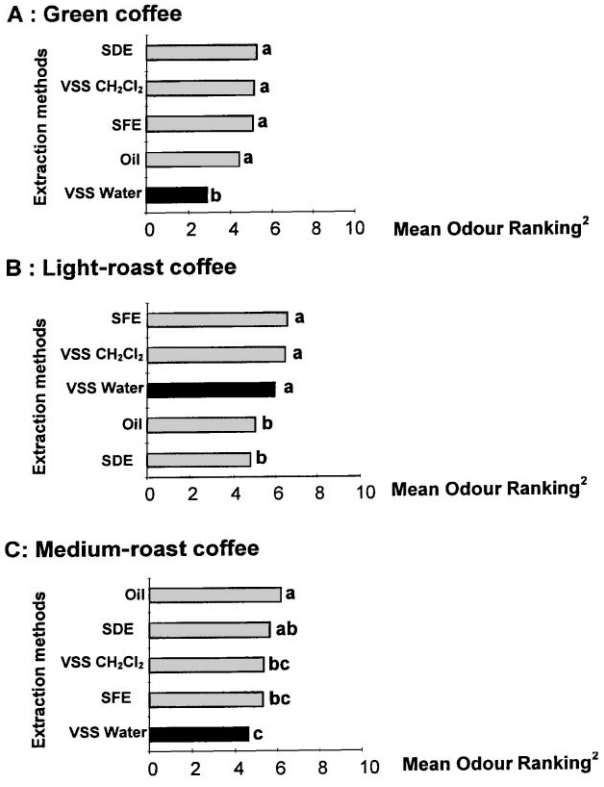

Fig. 3. Odour similarity test of aroma extracts with different extraction methods (A: green coffee, B: light-roast coffee, C: medium-roast coffee).

3.2. General scoring of the coffees — mean values

For the three graphs in Fig. 3, all mean scores for the aromatic extracts were rather far from the reference. This shows the difficulty of achieving an aromatic extract with all the typical sensory characteristics of the coffee. The best mean scores were 29, 48 and 46 mm for the three coffee samples, respectively (green, light, medium). Similar results were observed on goat cheese extracts (Le Quéré, Demaizières, Septier & Salles, 1996) where the best mean odour obtained was 44 mm (in a similarity test using an unstructured 100 mm scale). Green beans had the best score out of the three coffees. There are two possible reasons for this. First, green coffee has less volatile compounds which can be lost during the different steps of the aroma extraction and particularly during the concentration step. This is discussed later. Second, we noticed that, of the three coffees, the aroma of the green beans was the least difficult for the assessors to define. R&G coffee aroma gives roasting process odours, which are more complex than green coffee aromas. Overall, the difficulty in describing the odours may be due to a support effect, as the aromatic extracts were not put back in a support similar to the reference (Grosch, 1998). The incorporation of the aromatic extract into a support other than smelling strips may improve the odour evaluation.

3.3. Difference between extraction methods — similarity test

From the results shown in Fig. 3, we can see which aromatic extract was perceived as being the most representative of the coffee for each sample. For the green coffee (Fig. 3A), the results indicated that the most representative extract was achieved with the vacuum steam-stripping method (VSS water). According to the Duncan test, no significant difference in scoring odour was observed among the other aromatic extracts (SDE, Oil, SFE and VSS CH_2Cl_2).

For the medium roast coffee (Fig. 3C), the best mean score was also achieved by the vacuum steam-stripping method (VSS water). In this case, the aromatic extracts formed three over-lapping groups (one with the oil and SDE extracts, a second with the SDE, VSS CH_2Cl_2 and SFE extracts and a third with the VSS CH_2Cl_2, SFE and VSS water extracts).

For the light roast coffee sample (Fig. 3B), the methods indicated to be the best in this case were the SDE and the press oil aroma extraction. The VSS water extract scored in the middle of the five aroma extracts.

3.4. Variance analysis

The two-way variance analysis (see Table 3) showed that all five aromatic extracts were significantly different. These results highlighted a method effect and also an assessor effect. Moreover, as often found in sensory analysis, the assessor effect (which is usually significant) indicated a different use of the scale or a partial disagreement on the odour evaluation of these aromatic extracts, within the assessors. The interaction (method×assessor) allows the removal of this ambiguity. Therefore, when the interaction was not significant, the assessors were in agreement. In our results, no significant interaction (method×assessor) was observed for green and medium roast coffee. For the light roast, a significant interaction (method×assessor) showed a disagreement on the odour evaluation of the aromatic extracts by the assessors. This meant that it was difficult to conclude which aromatic extract was really the most representative of the complete coffee in this case. Therefore, we looked at the descriptors used by the assessors.

3.5. Validation of the most representative extract based on the descriptors

A further advantage of the similarity test is that the assessors provide descriptors of the aromatic extracts (Table 1).

An overall view for the three coffee samples showed that most of the descriptors quoted were already close to the descriptors used for the reference (Table 2). These

Table 1
Descriptors[a] associated with the aromatic extracts

Coffee	SDE extract	SFE extract	Oil extract	VSS CH_2Cl_2'' extract	VSS water extract
Green	Green (3) Cereal (3) Fatty (3) Smoke (3) Earthy/mouldy (3) Floral (2) Vegetable (1)	Green (6) Fatty (4) Floral (3) Pungent (3) Cereal (2) Vegetable (2) Earthy/mouldy (2)	Green (6) Cereal (3) Earthy/mouldy (4) Pungent (3) Sweet/butter (3) Floral (2) Potato (2) Fatty (2)	Pungent (5) Green (4) Earthy/mouldy (4) Vegetable (2) Fatty (2)	Green (8) Cereal (3) Fatty (3) Pungent (3) Vegetable (3) Earthy/mouldy (3) Floral (2) Potato (2) Fruity (2)
Light-roast	Green (4) Fatty (4) Pungent (4) Roasted (3) Smoky (3) Caramel (2) Fruity (2) Cereal (2) Earthy/mouldy (2)	Green (6) Cereal (2) Fatty (2) Roasted (2)	Cereal (3) Green (2) Roasted (2) Smoky (2) Pungent (2) Floral (2) Earthy/mouldy (2) Caramel (2) Smoky (2) Sweet (2)	Green (6) Fatty (3) Rubbery (2) Smoky (2) Earthy/mouldy (2) Roasted/toasted (2) Phenolic (2)	Green (5) Peanut (4) Roasted (3) Earthy/mouldy (2) Toasted bread (2) Caramel (2) Floral (2) Smoky (2)
Medium-roast	Old coffee (6) Phenolic (4) Fatty (3) Burnt (3) Rubbery (3) Earthy/mouldy (3) Sulphurous (3) Pungent (2) Smoky (2)	Earthy/mouldy (4) Smoky (4) Green (3) Phenolic (3) Rubbery (2) Pungent (2) Roasted (2) Peanut (2)	Earthy/mouldy (3) Fatty (3) Sweet/vanillin (3) Honey (2) Smoky (2) Phenolic (2)	Old coffee (4) Smoky (3) Earthy/mouldy (2) Pungent (2) Rubbery (2) Sulphurous (2) Phenolic (2) Burnt (2)	Roasted (6) Earthy/mouldy (4) Fatty (3) Caramel (2) Pungent (2) Smoky (2) Sulphurous (2) Phenolic (2) Toasted bread (2)

[a] Between brackets: frequency of citation per assessor. Only descriptors mentioned by more than 1 assessor are reported.

Table 2
Descriptors associated with the coffee samples

Coffee	Descriptors			
Green	Smoky	Soft	Vegetable	–
	Green	Potato		
	Floral	Earthy		
Light-roast	Smoky	Roasted	Almond	Cereal
	Soft	Biscuit	Caramel	Malt
	Pungent	Peanut	Earthy	Toasted bread
Medium-roast	Smoky	Curry	Fruity	Sulphurous
	Roasted	Burnt	Floral	Toasted bread
	Fatty	Pungent	Caramel	Coffee
	Earthy			

results confirmed the validation of the aroma extraction methods and also the procedure applied to evaluate the extracts.

For medium roast coffee, Grosch (1995) and Semmelroch and Grosch (1996) have demonstrated that, typically, the main notes in R&G coffee are sulphurous/roasted, musty/earthy, sweetish/caramel, green/peas and smoky/phenolic. In our case, for the medium roast coffee, and for the five extraction methods, the descriptors used for the VSS water extract were the most similar to these main notes. This gives a further indication that the VSS water extract was the most representative extract.

For the light roast coffee, we referred to Parliment and Stahl (1995) and Silwar and Lüllmann (1993) who showed that during roasting the green coffee develops green and pungent notes, followed by peanut, biscuit, cereal to roasted coffee finishing with fish, sulphur notes in dark roast. The SDE and oil extracts of group b were characterised by green, fatty, roasted, cereal and caramel notes (Table 1). For group a, the descriptors used were, respectively, green, roasted, peanut, toasted bread, cereal and caramel for the VSS water extract and more rubbery and phenolic notes for the two other extracts (SFE and VSS CH_2Cl_2 extracts). We observed that the sensory notes for light roast coffee (Table 2) named by the assessors, were roasted biscuit, peanut, caramel, toasted bread and cereal. Consequently, although the oil and SDE extracts were characterised cereal, only the VSS water aromatic extract provided simultaneous ''peanut'' and ''toasted bread'' notes. The presence of these notes reinforces the representativeness of the VSS water method, as indicated by the results for green and medium roast coffee.

Therefore, despite the results of the statistical test on light roast coffee, VSS water was retained as a suitable method to recover representative coffee aroma extracts from green coffee and coffees roasted at different levels.

Table 3
Two-way analysis of variance of the odour scores on the aromatic extracts for the three coffee samples

	Green coffee		Light-roast coffee		Medium-roast coffee	
Variation	F^a calc.	Prob.[b]	F^a calc.	Prob.[b]	F^a calc.	Prob.[b]
Extraction method	8.43	< 0.0001[d]	12.25	< 0.0001[c]	4.61	0.0014[c]
Assessor	3.46	< 0.0001[d]	14.18	< 0.0001[d]	10.94	< 0.0001[d]
Interaction: method×assessor	1.36	0.0557	1.77	0.0021	1.01	0.4593

[a] *F* calc, means the *F*-values.
[b] Prob, indicated the significance.
[c] Significant at 1%.
[d] Significant at 0.1%.

Vacuum steam-stripping is a time-consuming method but it is a powerful means of recovering volatile compounds under soft conditions and has already been used successfully in various food applications such as, wine aroma (Moio et al., 1995), tomato (Langlois & Etiévant, 1996), ham (Guillard, Le Quéré & Vendeuvre, 1997) and yellow passion fruits aroma (Werkhoff, Güntert, Krammer, Sommer & Kaulen, 1998) etc.

3.6. Sensory differences with respect to the extraction method

Some interesting sensory differences were observed among the different extracts of the same coffee sample.

Notes such as sulphurous, old coffee, burnt and phenolic were used more frequently in the SDE extract of medium roast coffee. This could be explained by heat degradation during the extraction or because there was a higher extraction of compounds giving these aromas. Although the SDE has frequently been used in coffee (Boosfeld et al., 1993; Holsher, Vitzthum & Steinhart, 1990; Silwar, Kamperscheoer & Tressl, 1987), the major drawback of this method (Buttery & Ling, 1996) is the high temperature required during extraction, which generates artefacts.

Vacuum-stripping allowed recovery of volatile compounds while avoiding the thermal degradation associated with SDE. The VSS CH_2Cl_2 and water extracts were mainly characterised by the descriptors "old coffee-smoky" and "roasted-earthy/mouldy", respectively. These sensory differences could be based on the preference of some volatile compounds for one process according to their hydrophobicity and volatility. Among the methods tested, the oil aromatic extracts revealed the highest score of sensory descriptors such as sweet, vanillin, honey and floral. Despite the good ranking for the green and light roast coffee samples, the "oil" extract for medium roast coffee was very far from the reference. We suspect that not all volatile compounds taken up by the oil were recovered at the vacuum-stripping step. This may be due to the more lipophilic components remaining in the lipid phase. Therefore, they were missing from the final aromatic extract.

3.7. GC analysis — quantification of the aroma recovery on the VVS water extract

GC analysis was done on the medium roast coffee only to investigate the influence of the different steps on aroma recovery (see Table 4 and Fig. 2).

Using this method, we obtained an aqueous distillate containing 1358 ppm aroma for the medium-roast coffee. After the extraction and concentration steps, the volatile compounds of the final aromatic extract amounted to 830 ppm. These results are in agreement with the findings of Silwar, Kamperscheoer and Tressl (1987) and Silwar and Lüllmann (1993) which suggested aromatic compounds of 700–800 ppm in R&G coffee. Consequently, our results tend to prove that this extraction method achieved an aromatic extract with aroma recovery close to the results from the literature. Aroma losses seemed to occur at two different steps (Table 4a): during the CH_2Cl_2 extraction (35% aroma remaining in water) and during the concentration step (approximately 5% aroma losses). Despite unavoidable losses of highly volatile compounds, the aromatic extract was still representative and had typical sensory notes.

To confirm the influence of the different parameters involved during the extraction method, the global aroma recovery was investigated. The first important step was the addition of sodium chloride which allowed an important increase in the recovery of hydrophilic compounds in the distillate, 1358 ppm/R&G coffee with NaCl compared with 738 ppm/R&G coffee without

Table 4a
Quantification by GC analysis of aroma recovery in medium roast coffee according to the influence of the different parameters involved in the vacuum steam-stripping[a]

Phase	ppm/R&G coffee	Aroma recovery %
Distillate	1358	100
Aqueous phase	472	35
Organic phase	884	65
Aromatic extract	830	61

[a] Aroma recovery during the different steps involved in the aroma extraction.

NaCl (Table 4b1). The influence of salt during the CH_2Cl_2 extraction was also checked. No significant improvement was revealed, less than 5% (Table 4b2).

The second important step for aroma recovery was the solvent extraction. The technique of cryoconcentration with three different freezing temperature programmes, prior to the CH_2Cl_2 extraction, was studied. As reported in Table 4c1, the appropriate temperature programme was $-1°$/h with 70% of aroma recovery. Our results were consistent with the work of Langlois et al. (1997) for a cryoconcentration technique in a model solution, where the aroma recovery of all volatile compounds was at least 75% for a final concentration ratio of 8. This is in the same range as our results (ratio of 10).

Unfortunately, the amount of aroma in the aromatic extract obtained with cryoconcentration was 500 ppm/R&G coffee, whereas the amount recovered in the extract with direct CH_2Cl_2 extraction was 830 ppm/R&G coffee (Table 4c2). In this case, the cryoconcentration technique was not suitable for aroma recovery. Some volatile compounds remained in the ice.

Finally, the concentration is a critical step in the preparation of aroma extracts from foods. The aroma losses were checked by comparison of two concentrators (high-speed distillation: Kuderna-Danish and a vortex evaporation system: Turbo Vap®500). As reported in Table 4d, the aroma losses were less than 4% in both cases. We concluded that both types of apparatus could be used for a concentration down to 1 ml. Guillard et al. (1997) and Langlois and Etiévant (1996) working with ham and tomato, respectively, have already shown that the Kuderna-Danish concentrator is suitable for small volumes, such as 500 μl and 300 μl, without significant losses.

Table 4b
Influence of NaCl

b1.Medium roast coffee	
Aroma in the distillate	ppm/R&G coffee
With NaCl	1358
Without NaCl	738
b2. CH_2Cl_2 extraction	
Aroma in the aromatic extract	ppm/R&G coffee
With NaCl	830
Without NaCl	818

Table 4c
Influence of the cryoconcentration technique prior to the CH_2Cl_2 extraction

c1. Freezing temperature programme

Freezing temperature programmes	Aroma in the cryoconcentrated distillate ppm/R&G coffee	Aroma in the ice
−5% constant	102 (8%)	1167 (92%)
−5°C/h	140 (11%)	1129 (89%)
−1°C/h	935 (70%)	334 (30%)

c2. Influence of the cryoconcentration technique

Cryoconcentration	Aromatic extract ppm/R&G coffee
With	500
Without	830

Table 4d
Influence of the concentration step

Apparatus	Aromatic extract ppm/R&G coffee
Zymark apparatus	790
Kuderna-Danish apparatus	830

4. Conclusion

When performing GC–O analysis to identify key flavour compounds, the objective is to characterise the aroma of the complete product. This may be executed on a suitable aromatic extract. The choice of extraction method depends on the type of food and the information needed. It is first essential to recover an aromatic extract as representative as possible of the product. The present work is aimed at selecting a method capable of extracting volatile compounds from green and roasted coffees.

Five techniques were investigated and a sensory test was applied to evaluate the extract odour quality. Of the five methods studied, vacuum steam-stripping with water, of ground coffee, followed by methylene chloride extraction and concentration, provided an aromatic extract with sensory characteristics the most representative of the initial coffee. During these steps, 2/3 of the aromas were recovered in the solvent extraction and approximately 5% aroma was lost in the concentration step. Finally, this aroma extraction provided an aromatic extract containing 800–900 ppm aroma. Although some of the volatile compounds were lost during the aroma extraction, the results indicated that this final concentrated extract possessed the sensory characteristics of roasted coffee suitable for subsequent GC–O. The aroma extraction was also successfully applied to coffee with different roast levels and to green coffee.

Acknowledgements

We are grateful to the sensory team for this collaboration and technical assistance in this work.

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