Chahan Yeretzian, Alexia Glöss, Sergio Petrozzi, Lucio D'Ambrosio, Kaja Knöpfli-Lengweiler, Flurin Wieland, Andrea Fridolin Wild and Ruedi Anliker
Coffee Research Group, Zürich University of Applied Sciences

The smell of coffee – an analytical perspective

Within the last century, coffee has become one of the world's most popular beverages and represents a major economic factor for many coffee-producing countries and a significant business sector in consuming countries. The success of this fascinating brew has been overwhelming and its future seems even more exciting[1,2]. The ever transforming landscape of coffee-shops and coffee-houses, the various trends and fads in the growing speciality coffee sector, the mounting awareness about origins, the steady innovations in the coffee machine sector and last but not least, the rising media-hype around the Barista-scene are all just the tip of the iceberg for a steadily growing coffee-lover community and an astonishingly recession-resistant industry.

Fuelled by high-level media and PR coverage and endorsed by celebrity 'ambassadors' such as George Clooney (for *Nespresso* coffees) and Roger Federer (for *Jura* coffee machines), this trend is here to stay and grow. Today, coffee is a brew full of passion, pleasure and plenty of myths.

The prime reason for the increase in the popularity of coffee is its unique set of sensory qualities, particularly the strong and distinctive flavour. The perception elicited from drinking a freshly prepared coffee involves all of our senses, such as olfaction, taste, texture, trigeminal and visual sensation. Furthermore, emotions and cognitive processes constructed during drinking experiences, such as interactions between senses and product familiarity, modulate perception. Among the various sensory modalities, the aroma, also known as the smell, is of the greatest importance to the consumer experience of a cup of coffee.

The aroma of a freshly prepared cup of coffee is the final expression and perceptible result of a long chain of transformations which link the seed to the cup. It results from the interplay between genetic predispositions, environmental factors, harvest practices, roasting, grinding and extraction techniques. Among all these factors, three appear of particular importance. First – the predisposition: the green coffee variety (genetics) with its specific set on chemical precursors lays out the stage for later aroma development during roasting – these can be slightly modulated by harvest practices and post-harvest treatment. Yet the green beans give no clue as to what they might become once roasted. They convey neither the characteristic smell nor the taste of a cup of coffee. Second: the aroma generation: the roasting process unlocks the aroma potential of the green coffee beans and creates the coffee aroma that is so appreciated by coffee aficionados. Third: the extraction into the cup: the grinding and extraction, allow the right mix of compounds to be extracted by the hot water and transferred into the cup.

How is coffee aroma really made?

In order for coffee to be sensed by our nose (i.e. to have a smell), aromatic volatile compounds have to be released from the brew and reach the olfactive epithelium, a region in the upper part of the nasal cavity which contains the nerve endings that allow us to smell.

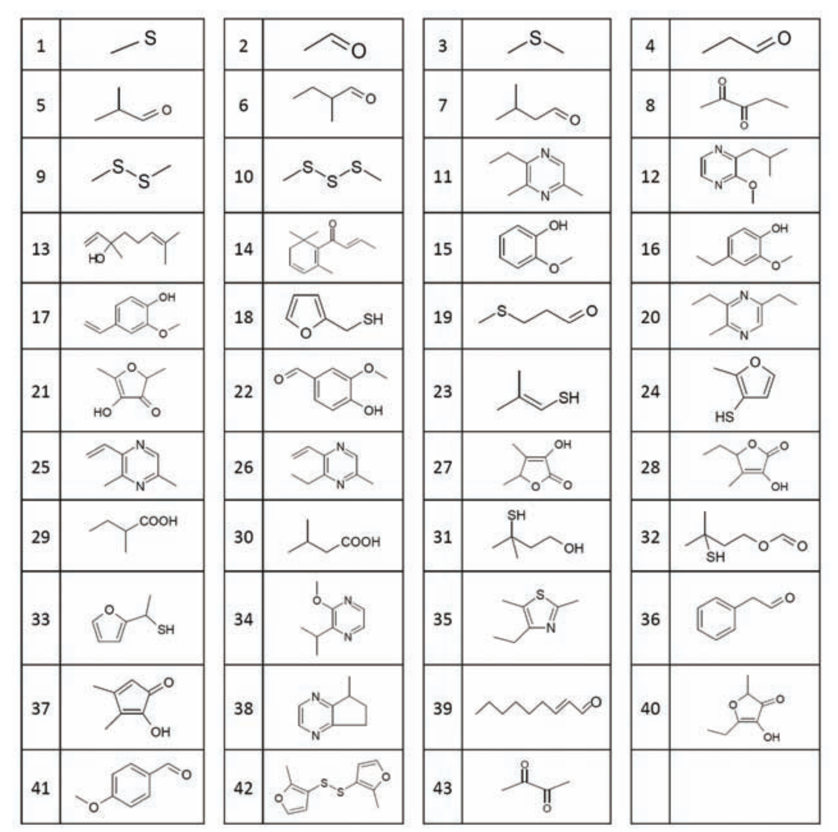

Figure 1 43 volatile compounds that contribute to the aroma of coffee

Scientific efforts to elucidate the origin of the rich and distinctive aroma of coffee, and ultimately to understand "what makes that coffee smell so good?"[3,4] can be traced back to 1880 when Bernheimer, a German scientist, identified the first few volatile compounds of coffee[5]. But the first significant progress can most likely be attributed to Reichenstein & Staudinger, who, in 1926, identified and patented several important aroma active compounds of coffee[6,7]. Mainly fuelled by progress in analytical techniques, in particular gas chromatography (GC), the number of publications on coffee aroma and concomitantly the number of identified coffee volatile compounds has rapidly increased since then. Today, almost 1000 volatile compounds have been reported in coffee, which includes the compounds from both green and roasted[8].

» Without doubt, the classical 'work-horse' in coffee aroma analysis has been gas chromatography «

For many years, scientists have concentrated on identifying volatiles in coffee. As a result, the number of identified volatiles has steadily increased. However, by the 1970s, it became clear that only a small fraction of these volatiles – perhaps five per cent – are odoriferous and hence relevant to the aroma. The focus shifted towards these few sensory relevant aroma-active compounds in the headspace (HS, the air-space just above the coffee).

Several instrumental methods were developed which should achieve the following objective: identify and quantify the odour-relevant volatiles, assess their odour impact and recombine a coffee aroma with the minimum number of impact compounds. Figure 1 gives a collection of 43 coffee aroma compounds that have been repeatedly found in coffee. Currently, less than 30 volatiles are believed to be important aroma compounds of roasted coffee[9-17]. Omission experiments from Grosch, a renowned German coffee aroma scientist, and his co-workers suggest that the actual number of key coffee aroma compounds could be as small as nine[18-22].

Gas Chromatography

Without doubt, the classical 'work-horse' in coffee aroma analysis has been gas chromatography (GC). Depending on the application, it can be equipped with different sample injection and detection systems, and has led over the past 30 years to the elucidation, characterisation and

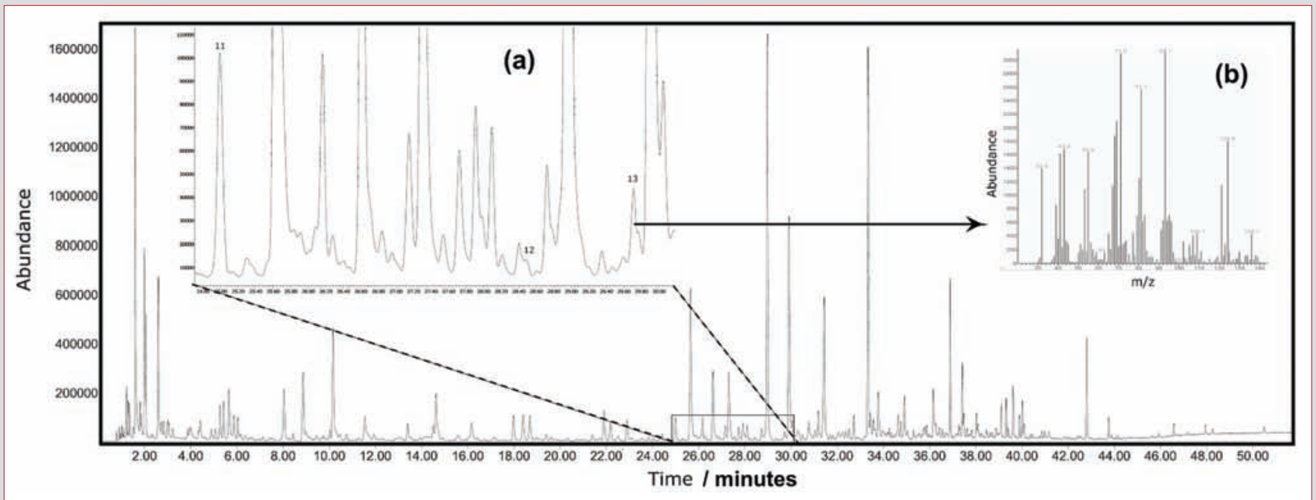

Figure 2 Gas Chromatogram of a coffee HS analysed by Solid Phase Micro Extraction Gas-Chromatography, and detected in a mass spectrometer (HS-SPME-GC-MS). Intercept (a) shows a magnified section between 25 to 30 minutes. The mass spectrum of compounds #13 is show in intercept (b)

quantification of the compounds relevant to coffee aroma. The most important element of any GC analysis is the chromatographic capillary column. On one end of the column, the sample is introduced onto the head of the column in the form of a small vapour plume. While passing through the column, whose walls are covered with an organic solvent, the different chemicals in the sample have different affinities for the solvent, and hence travel at different speeds. Consequently, they separate as they pass through the column, and individual compounds reach the end of the column at different times. At the exit side of the column, a detector identifies and eventually quantifies these compounds in the order of their arrival, and allows reconstruction of the complete aroma as the sum of the individual aroma active compounds eluting at the end of the column.

Obviously, the major weakness of such an approach is that it neglects possible interactions between aroma compounds, such as masking and enhancement, and also does not consider interactions with other sensory modalities, such as aroma-taste or aroma-texture interactions. While such interactions are known to exist, GC analysis is still considered a good approach to understand and reconstitute the flavour of coffee. The ultimate proof that we 'understand' the aroma of coffee is when we are able to reconstitute it[20,21].

In Figure 2 on page 12, a typical GC trace is shown, in which the slowest compounds have up to 50 minutes to travel through the column used here. Prior to injection, the volatile compounds were enriched by a technique called Solid-Phase Micro Extraction (SPME). This is often necessary as the concentration of many compounds in the HS of coffee is very low and barely detectable even in the most sensitive analytical instruments. At the exit side of the column, the detector used here is a mass-spectrometer. This allows a mass spectrum to be recorded to assist in the identification. In the chromatogram shown in Figure 2, several hundreds of compounds have been detected, of which approximately 150 were identified via their mass spectra. To illustrate the very large number of volatile compounds identified in such a method, we show a magnified intercept (a) of Figure 2 for the time-window between 25 to 30 minutes. In this small time-window, probably more that 50 compounds are eluting, many of which are partially or completely overlapping. Three compounds that are included in the list of Figure 1 are marked with their respective numbers.

Identification was accomplished via mass spectral detection. This is demonstrated in intercept (b), which shows the mass spectrum of compound #13. Matching the recorded mass spectrum in intercept (b) with a collection of thousands of mass spectra in a database, compound #13 was identified as Linalool.

Olfactometry: when the human nose becomes a detector

Many aroma active compounds in coffee appear in the HS at only very low concentrations, some of which can hardly be detected by analytical detectors. The only detector capable of sensing these highly potent coffee aroma compounds is our nose. Hence, aroma chemists use their nose (or a panel of 'sniffers') to detect compounds eluting at the end of the GC column. If trained, such sniffers can also give a sensory description of the compound being sniffed, which is of help for their identification. Techniques which combine separation

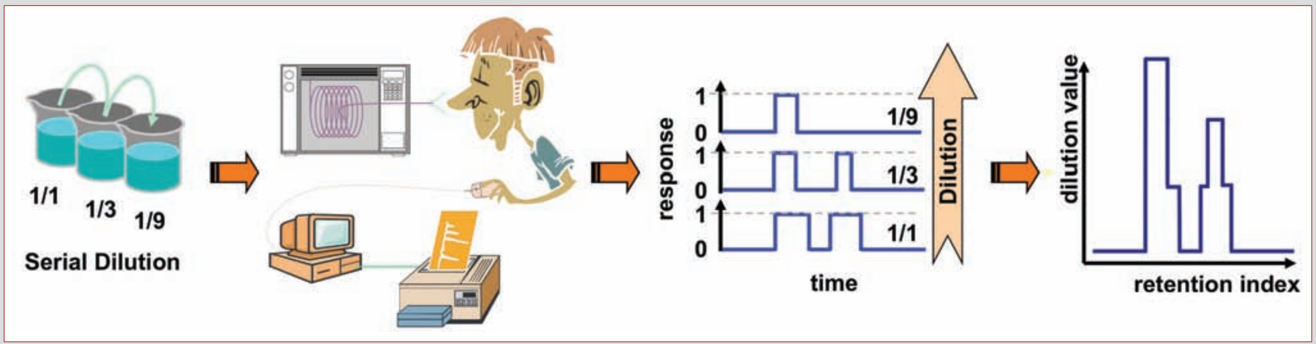

Figure 3 Schematic description of Gas Chromatography Olfactometry, GC/O. Aroma extract dilution analysis (AEDA) and CHARM analysis are the two most widely used realisations of GC/O methods.

by capillary column chromatography and the human nose as detector are called GC-Olfactometry – GC/O[23-29].

Two major GC/O screening techniques have been developed; one by Grosch and co-workers (called the Aroma Extract Dilution Analysis)[22-27] and the other by Acree and co-workers (called CHARM analysis)[23,26,30]. They are shown schematically in Figure 3. Both evaluate by GC/O a dilution series of an original aroma extract. Note is taken of the occurrence of an aroma in each dilution. With increasing dilution, the compounds with lower odour potency are successively not sensed anymore, and only the most potent are detected at higher dilutions. One then adds the occurrences of an odorant across dilutions. The greater the number of dilutions an odorant is sensed, the higher the odour potency in both AEDA and CHARM analysis. This leads to plots of Dilution- or CHARM-values. Both AEDA and CHARM analysis have originally

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proposed that the larger the Dilution- or CHARM-value, the more important the contribution of the respective aroma compound to the overall aroma is. While this interpretation has slightly evolved since its introduction, both techniques are still widely used to estimate the relative importance of various volatile aroma compounds to the aroma of coffee.

An interesting application of GC/O was the identification of the chemicals responsible for the mouldy/earthy off-notes found in lots of green Mexican coffee[28,29]. GC-profiles obtained from the reference sample – a sample free of off-notes – and the mouldy sample showed minor differences and no indication for the off-note was found. The same samples were then subjected to a GC/O sniffing analysis. This allowed identification of several important differences between the extracts that are related to the off-flavour – see Figure 4. In particular, earthy, green, chemical and mouldy chromatographic zones were located that could be identified as being 2-Methyl isoborneol, 2,4,6-Trichloroanisole, Geosmin and various Pyrazines.

From the example in Figure 4, it is clear that GC/O is particularly efficient at identifying the main olfactive defaults of the defective sample relative to the reference. It offsets the lack of sensitivity for low concentration flavour active compounds encountered with other detection systems. In this study, it was clear that instrumental detection failed to recognise the defect documented in the sensory profile.

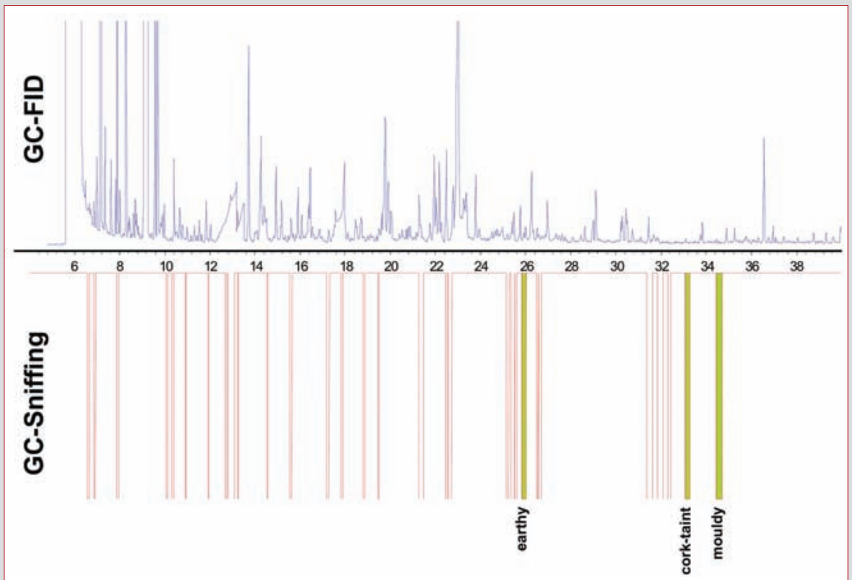

Figure 4 Comparison of a GC Chromatogram on the top with the GC/O profile on the bottom, for a sample with a mouldy/earthy defect, analysed by one sniffer. All sniffed signal without sensory descriptor represent signals that are typical to coffee and are not related to the off-note.

What's next?

Through a sustained and concerted research effort over more than 30 years, our understanding of the aroma of coffee has steadily grown. Today, we believe that the list of aroma active compounds is essentially complete, and we do not expect major discoveries in this field. Trends that will shape the future of (coffee) aroma research can be classified into two main categories. On the one hand, novel analytical technologies and strategies are being introduced that go beyond chromatography and integrate, for example, optical and laser technologies,[31,32] on-line methods,[33-41] nose-space analysis,[42-46] as well as advanced computational calculations[47]. On the other hand, research is expanding into new fields. This includes studies on the interactions between aroma compounds and between different sensory modalities. A critical development is research on improved strategies and methods

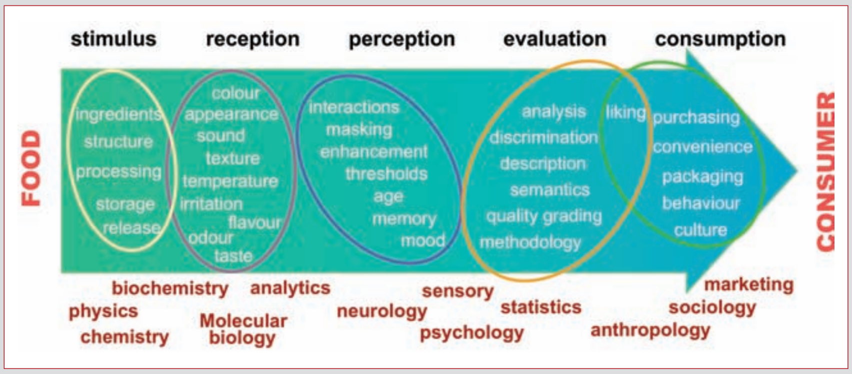

Figure 5 Linking the food to the consumer, a very large number of diverse technical expertises and scientific disciplines are joining forces

for the correlation of sensory and instrumental analysis[39,48], but most significantly, aroma research is increasingly aligning itself towards the consumer, developing new customer centred and individualised approaches to aroma science[46,49,50]. Linking the food to the consumer, as schematically shown in Figure 5, is indeed one of the new paradigms for aroma research. It has also become obvious that what was initially a discipline restricted to the realm of food science is today a highly multidisciplinary research field of the life sciences. This was impressively demonstrated at the last Weurman Flavour Research Symposium in 2008, in Interlaken, Switzerland, where scientists from all disciplines met to discuss the latest developments in flavour sciences[51,52].

Today, flavour science is moving into a discipline that is truly multidisciplinary and that requires a new breed of scientists. What was once the playground of food and flavour scientists and analytical chemists is today a complex scientific platform where experts from biology, psychophysics, psychology, organic chemistry, analytics, material sciences, physics, mathematics and health meet with food and flavour scientists to work in concert.

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Dr. Chahan Yeretzian

Dr. Chahan Yeretzian is Head of the Analytical Chemistry & Technology Group of the Zürich University of Applied Science in Wädenswil. His current research includes a focus on the chemistry, technology and flavour of coffee. He is the initiator and coordinator of the Certificate of Advances Studies on "The Science and Art of Coffee" – www.icbc.zhaw.ch/coffee – the first university degree on coffee. Additionally, he is a Board Member of the Swiss Chapter of the Speciality Coffee Association of Europe (SCAE) – http://www.swissscae.ch.

Dr. Yeretzian received his PhD in Chemistry from the University of Bern, Switzerland, for research on material sciences and laser spectroscopy. A two year post-doctoral position at the University of California, Los Angeles (UCLA), and a subsequent Alexander von Humboldt Junior Award for a two year Fellowship at the Technical University of Munich, complemented his academic education. In 1995, he accepted a position at the Nestlé Research Centre in Lausanne, Switzerland, where over a period of 12 years, he held various R&D management positions, mainly in both product categories of Coffee and Culinary. During this period, he also completed an MBA degree at the University of Lausanne. In 2007, he joined the Corporate Communication Department of Nespresso, developing communication platforms on the science of coffee. Since 2008, he has been teaching analytical chemistry at the Institute of Chemistry and Biological Chemistry at Zürich University of Applied Science, and has also initiated a research platform on coffee.

Dr. Yeretzian is co-author of more than 80 peer-reviewed publications and book chapters, co-editor of a book entitled "Expression of Multidisciplinary Flavour Science" and regularly invited by both specialised and main stream media to contribute articles and interviews around the subject of coffee.

Contact the author: yere@zhaw.ch