Back tracing environmental toxicants in animal-derived food chain based on food metabolomics

Erwan Engel, Jérémy Ratel, Christelle Planche

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Issue 6 · 2013

Sodium reduction in ready meals

Martin G. Wilkinson,
Department of Life Sciences,
University of Limerick

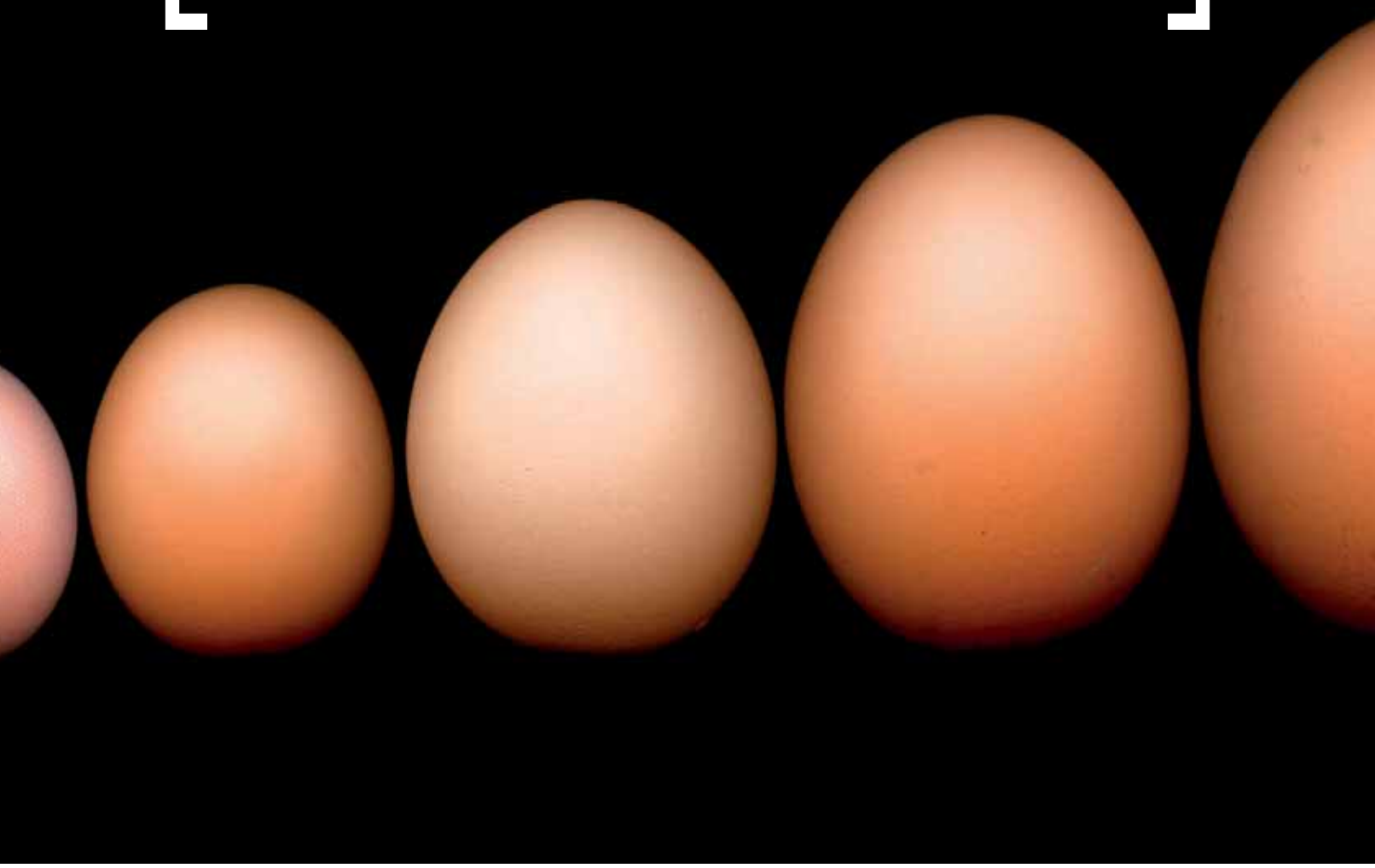
Pasta production

Alexis Freier, Research & Development
& Technical Services Manager,
Dakota Growers Pasta Company

Mass spectrometry for the food industry

Angela Calder, Leatherhead Food Research

No balance sheet has a line for Salmonella.



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Helen Bahia
Editor

Looking ahead at 2014

Innova Market Insights have revealed their Top 10 Trends that research has indicated will impact the food industry in 2014, many of which are unsurprising given the negative headlines that have beleaguered the food industry in 2013. The trends include building consumer confidence, analysing and reducing food loss or waste, moving towards simpler foods, small innovators with high quality and distinct products will flourish, and nutritious food and beverage solutions as a possible answer to healthcare budget crises globally. Other trends include new superfoods, the rise of hybrid foods, protein in new product launches (especially in the dairy sector), competition for alternative products such as soy, and what has been termed stealth or health, a choice manufacturers will have to make when they market products with reduced sugar, salt and/or saturated fats contents of foods.

None of the identified trends are surprising, but perhaps the most important for companies to survive will be building and or retaining consumer trust. With the meat adulteration scandal still affecting the industry today, consumer relationships are of the utmost importance. In this issue of *New Food*, we have a bumper supplement that looks at both mass spectrometry and rapid methods for detecting and analysing food samples to ensure the safety of the consumer and the integrity of the food manufacturer. Please turn to page 19 for the supplement.

From the team at *New Food*, we wish all our readers, contributors and advertisers a very happy holidays and a prosperous new year!

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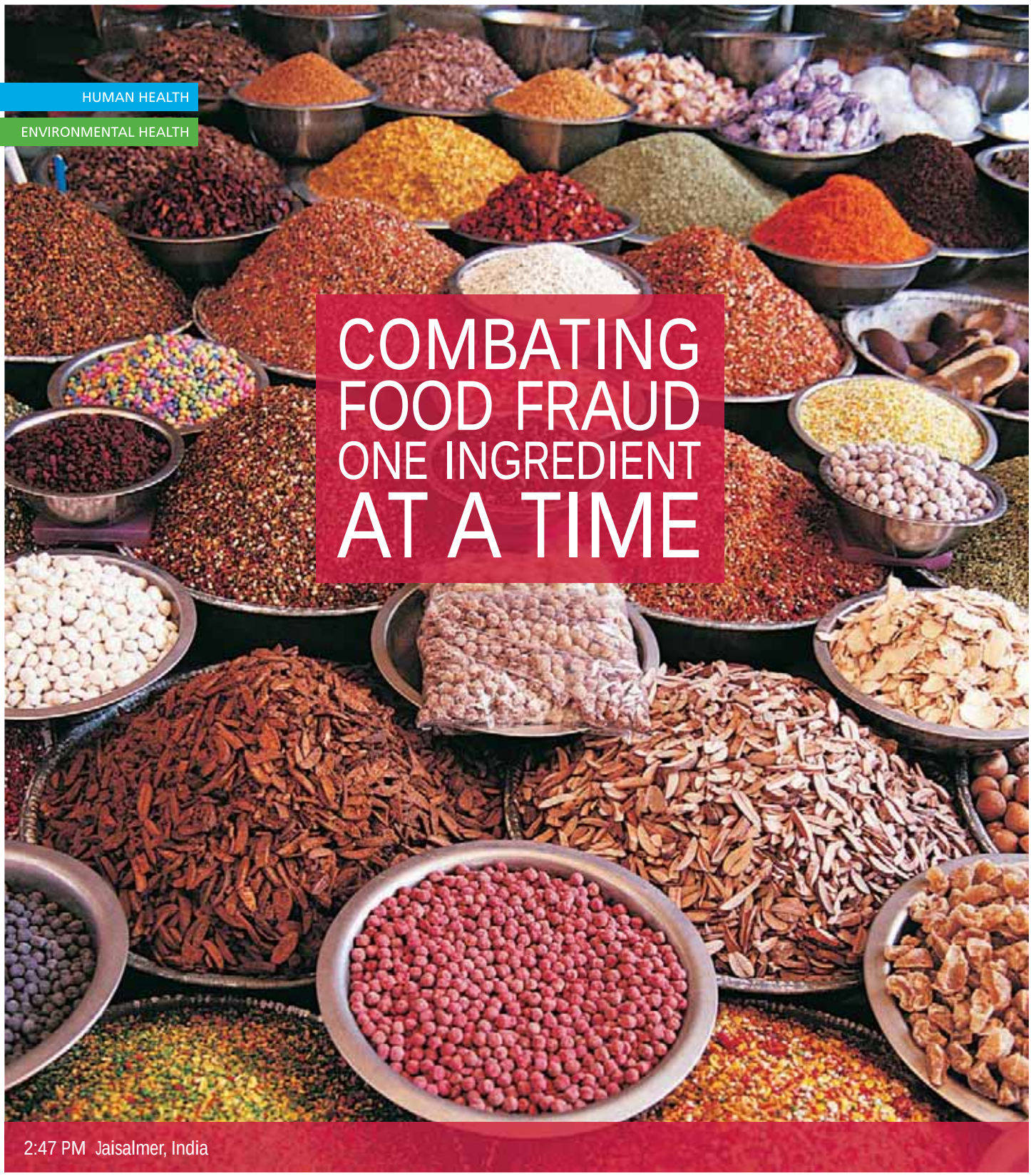

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2:47 PM Jaisalmer, India

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Events

JANUARY 2014

The 100th Annual NW Food Processors Expo & Conference
Date: 12 – 15 January 2014
Location: Portland, Oregon, USA
e: info@nwfpa.org
w: www.nwfpa.org/events/expo-2014

IFPAC 2014 – Fifth Annual Food Quality, Safety & Analysis Symposium
Date: 21 – 24 January 2014
Location: Arlington, Virginia, USA
e: info@ifpacnet.org
w: www.ifpac.org/food

FEBRUARY 2014

Cfia Rennes 2014
Date: 11 – 13 February 2014
Location: Rennes, France
t: +33 (0) 55336 7878
w: www.cfiaexpo.com

International Ice Cream Science & Technology Training Workshop
Date: 18 – 20 February 2014
Location: Cork, Ireland
e: mccarthybuckley@ucc.ie
w: www.ucc.ie/fitu

Gulfood 2014
Date: 23 – 27 February 2014
Location: Dubai, UAE
e: gulfood@dwtc.com
w: www.gulfood.com

4th Annual European Food Manufacturing & Safety Summit 2014
Date: 25 – 26 February 2014
Location: The Netherlands
e: laura.kill@wtgevents.com
w: www.foodmanufacturingevent.com

MARCH 2014

World Food Technology & Innovation Forum
Date: 3 – 4 March 2014
Location: St. Paul's, London, UK
e: chris.chapman@wtgevents.com
w: www.foodinnovate.com

4th Annual Food Manufacturing & Safety Forum
Date: 10 – 11 March 2014
Location: Dallas, Texas, USA
e: kelly.lowe@wtgevents.com
w: www.foodmanufacturingsummit.com

Foodex 2014
Date: 24 – 26 March 2014
Location: NEC Birmingham, UK
e: beki@stormcom.co.uk
w: www.foodex.co.uk

Food Structure and Functionality Forum Symposium from Molecules to Functionality
Date: 30 March – 2 April 2014
Location: Amsterdam, The Netherlands
e: arozenberg@elsevier.com
w: www.foodstructuresymposium.com

APRIL 2014

Food Technology and Innovation Forum 2014
Date: 7 – 9 April 2014
Location: Chicago, Illinois, USA
e: kelly.lowe@wtgevents.com
w: www.thefoodsummit.com

15th Food Colloids Conference
Date: 13 – 16 April 2014
Location: Karlsruhe, Germany
e: karsten.koehler@kit.edu
w: www.ift.org

MAY 2014

AOAC Symposium on Chemical & Microbiological Analytical Challenges in Food Safety
Date: 5 – 7 May 2014
Location: Istanbul, Turkey
e: aoace.ist2014@gmail.com
w: www.aoaceurope-istanbul2014.org

Vitafoods Europe 2014
Date: 6 – 8 May 2014
Location: Geneva, Switzerland
t: +44 (0)20 7017 6482
w: www.vitafoods.eu.com/lsnewfood

IAFP's European Symposium on Food Safety 2014
Date: 7 – 9 May 2014
Location: Budapest, Hungry
e: info@foodprotection.org
w: www.foodprotection.org/europeansymposium

XII International Conference on the Applications of Magnetic Resonance in Food Science: Defining Food by Magnetic Resonance
Date: 20 – 23 May 2014
Location: Cesna, Italy
e: foodmr2014@unibo.it
w: www.foodmr.org

3rd International ISEKI-Food Conference
Date: 21 – 23 May 2014
Location: Athens, Greece
e: office@iseki-food.net
w: www.isekiconferences.com/athens2014

JUNE 2014

Salt-Sugar-Lipids Reduction
Date: 17 – 18 June 2014
Location: Nantes, France
e: bpop@tritecc.ro
w: www.pleasure-fp7.com/conference/index.html

IFT Annual Meeting 2014
Date: 21 – 24 June
Location: New Orleans, LA, USA
e: info@ift.org
w: www.am-fe.ift.org

JULY 2014

3rd International Conference and Exhibition on Food Processing & Technology
Date: 21 – 23 July 2014
Location: Las Vegas, USA
e: foodtechnology2014@conferenceseries.net
W: www.foodtechnology2014.conferenceseries.net

If you have a diary event you wish to publicise, send details to Martine Shirtcliff at: mshirtcliff@russellpublishing.com

New Nestlé freezers to help tackle climate change

Nestlé is systematically introducing more environmentally efficient ice cream freezers using natural refrigerants across Europe. The company has pledged that from now on all new commercial horizontal ice cream chest freezers bought by Nestlé across Europe will use only natural refrigerants. These are naturally occurring substances – like carbon dioxide, ammonia, water or air and hydrocarbons, like propane and isobutane – which do not harm the ozone layer and have no or negligible global warming effects. The new horizontal ice cream chest freezers will reduce energy consumption by more than a third, in comparison with existing systems.

Replace and renew

Nestlé has already introduced 18,000 natural refrigerant hydrocarbon freezers worldwide.

The company has also invested more than CHF 240 million to replace synthetic refrigerants with natural alternatives in more than 92 per cent of its industrial refrigeration systems. The decision to systematically introduce these commercial horizontal ice cream chest freezers across Europe is part of Nestlé's commitment to progressively phase out the use of high global warming potential refrigerants, such as hydrofluorocarbons (HFC). It also moves the company beyond meeting the Consumer Goods Forum Resolution on Refrigeration, which encourages businesses to take action towards phasing out some HFC refrigerants from 2015.

www.nestle.com

Hanna Instruments launches new mini titrator for wine and fruit juice analysis

Hanna Instruments is pleased to announce the release of a new mini automatic titrator designed specifically for determining the formol number in wines and fruit juices.

The new titrator, HI 84533, is designed with a high precision piston dosing system with dynamic dosing and compact, space saving footprint. It also features automatic stirrer speed control, a graphic mode with exportable data and a Good Laboratory Practices (GLP) feature which allows users to view calibration data.

Hanna Instruments manufactures a variety of analytical instrumentation for the food and beverage industry, including juice meters, dairy meters, wine meters and more. Hanna's new mini titrator is available through any of our 40 international locations. These offices provide Hanna customers with local service, support and training and are part of Hanna's goal to provide an exceptional customer experience.

www.hannainst.com

ACQUITY QDa Detector: leveraging mass detection for food analysis

The release of the ACQUITY® QDa Detector has brought mass detection within the reach of a much broader scientific base. Mass spectrometry has traditionally required some degree of understanding of the process through which the technology operates – until now.

The ACQUITY® QDa Detector has been designed to operate with little user intervention, much like the PDA detector that is familiar to food scientists. Unlike with a PDA however, it offers all the discrimination and sensitivity associated with mass detection to provide opportunities for consolidation of analytical methods. This delivers an ease of use for dealing with the complex matrices that commonly plague food analysis, thereby also providing scope for simplification of sample preparation (the single biggest challenge in food testing).

The ACQUITY® QDa was specifically designed to meet the robustness essential to make it a routine instrument in a laboratory where instrument uptime is essential to meet turnaround times, like a busy food manufacturing facility. From its inception, the specification for the system includes a delivery cost that is as revolutionary as the technology itself, making it very attractive to food quality scientists.

www.waters.com/qda

Merck Millipore Singlepath® Direct Campy Poultry Kit for faster, easier on-site *Campylobacter* testing

Merck Millipore's Singlepath® Direct Campy Poultry Lateral Flow Kit enables rapid immunological screening of *Campylobacter* in poultry flocks. The kit requires no prior enrichment step or additional laboratory equipment, and can be completed on-site, at the farm. Test results are available within two hours of sample collection.

The 'pregnancy test' assay design is easy to use and displays results in a clear yes/no format. A built-in control reaction ensures greater accuracy and reliability. The kit contains all materials and reagents needed for testing: the test device, tubes, a pipette, sample buffer and sample diluent.

Because *Campylobacter* can spread to an entire flock in a matter of a few days, testing should be done frequently. Until now, testing has been cumbersome and time-consuming. The Singlepath® Direct Campy Poultry Kit allows screening for *Campylobacter* immediately prior to slaughter, which enables financial decisions, such as the separation of *Campylobacter* highly positive and lower risk flocks for slaughter, to be made on the basis of real-time information.

Merck Millipore is offering free samples of the Singlepath® Direct Campy Poultry Kit to the first 100 people who register at www.merckmillipore.com/poultry.

www.merckmillipore.com

Newsbites

New release JuiceScreener™

With the third release of the push button NMR-based JuiceScreener in October 2013, juice quality and authenticity analysis has been further enhanced in quantification and statistical results. Now high-valued products such as passion fruit juice, pomegranate juice and mango puree are covered. The reference database contains more than 16,000 samples, which not only further enhance the sensitivity and specificity of statistical models but also enable important new statistical parameters (in non-targeted screening).

The enhanced representation of reference distributions of compound concentrations is now based on more than 300,000 NMR values (and is now available for 19 types of fruit). For validation, more than 12,000 conventional values are available which show good agreement. The inclusion of compound normal concentrations further enhances the judgment on normality for those compounds, which is only available by NMR.

The JuiceScreener now also determines, in addition to the existing country models, the origin of mango puree (available origins are Mexico and India). A further non-targeted result (all within a single measurement of about 15 minutes) is the detection of the addition of peach and apricot, in respect to other types of puree.

A check of fruit content for more than 10 types of juice is available with this release (e.g. for pomegranate, passion fruit, grapefruit). This enables the detection of sugar and/or water addition, a major fraudulent practice that occurs especially in high value juices. The substantial added value now available to everyone in juice screening is further raising the method's prominence.

www.buker.com/sgf

Tate & Lyle wins FiE Beverage Innovation award

Tate & Lyle, a global provider of ingredients and solutions to the food, beverage and other industries, has been crowned the winner of the Food Ingredients Europe Beverage Innovation of the Year Award in Frankfurt, for its innovative PROMITOR® Soluble Gluco Fibre. Tate & Lyle showcased the award winning ingredient in the form of a Raspberry Lime Tea drink, demonstrating that sugar and calorie reduction is achievable while maintaining body and taste through the addition of fibre.

The prototype was developed by replacing sugar with Tate & Lyle's, no-calorie, TASTEVA™ Stevia Sweetener to provide the clean sweetness and fresh taste from a natural source adding PROMITOR® Soluble Gluco Fibre to maintain the right mouth feel and increase fibre content. Tate & Lyle's PROMITOR® Soluble Gluco Fibre provides transparent bulking and fibre fortification as it is highly soluble and has very little colour or taste making it easy to incorporate in a wide range of products.

The FiE Excellence awards, which take place every two years, rewards the major innovations in the food industry in seven categories and are judged by a panel of 10 industry experts. The awards took place at FiE in Messe Frankfurt on Tuesday 19 November 2013.

www.tateandlyle.com

Advantages of Haas-Meincke ovens have made them extremely popular

Consumers all over the world are increasingly demanding high quality products at low cost. Depending on the product type, the delicious bakery products produced on the world market today require different methods of heat transfer and moisture addition. With our large range of ovens, comprising convection ovens, directly fired gas ovens, radiation ovens and hybrid ovens, we can meet the requirements for the most sophisticated baking profiles. Traditionally, the Haas-Meincke ovens have been known for high quality baking. Even and reliable baking of the products is primarily obtained through an extreme precise control of the moisture, the airflows and the heat distribution inside the oven. This precise control also makes our ovens flexible. We can combine different oven types and quickly adjust them according to the customer's wishes.

Haas-Meincke ovens are well insulated and rank among the best when it comes to efficiency. In recent years, we have focused on energy saving efficiency in order to meet the world's energy issues. As a result, we can offer our customers a 15 per cent reduction of energy consumption with our Heat Recovery Unit for the convection oven.

For our customers, short installation times are very important. Every day that goes by without the line running equals lost production time. The Haas-Meincke ovens are pre-manufactured in two metre modules to facilitate quick installation. Concretely speaking, this means that installation time is reduced to only a few weeks compared to the normal one to two months for a complete assembly. Pre-manufacturing of the oven modules also ensures that production on nearby lines can continue during the installation without health issues or discomfort of any kind.

www.haas-meincke.com

Campden BRI key appointment

Food and drink research company Campden BRI has appointed Steve Spice as their new Head of Regulatory Affairs. He joins Campden BRI after 15 years at Waitrose, where he was Regulatory Affairs Manager and previously Analytical Services Manager. Steve will lead the Campden BRI regulatory affairs team, which continues to expand to meet a growing demand for its legislative advice, training and consultation services.

Steve brings over 30 years' industry experience in legislative and technical roles at Nestle, Leon Frenkel, Cow & Gate, QPeanuts and Sol Tenco. His expertise in food law will be invaluable as the team prepare for a challenging year ahead with the biggest change in labelling legislation for a generation – through the enforcement of the Food Information to Consumers (FIC) Regulation in December 2014.

www.campdenbri.co.uk

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Davide Fissore, Mauricio M. Coletto and Antonello A. Barresi
Department of Applied Science and Technology, Politecnico di Torino

Atmospheric food freeze-drying: challenges and opportunities

Freeze-drying is a process that can be used to remove water from foodstuff, thus increasing their shelf-life, avoiding deterioration of aroma and flavour compounds as well as nutrient degradation. In a vacuum freeze-drying process, product temperature is firstly decreased in such a way that all the free water freezes, then the product is exposed to low pressure and the ice sublimates (primary drying). At the end of the sublimation stage, the amount of residual water can be further decreased by removing the bound water (secondary drying); this stage is generally carried out increasing product temperature and decreasing the operating pressure. This way, all the characteristics of the product, e.g. shape, appearance, colour, taste and texture, are retained in the final product. Moreover, the high specific surface area of the final product allows a fast and easy rehydration.

The main drawback of a freeze-drying process is the cost: in particular, the energy required by the process is higher with respect to that of other drying processes. Claussen *et al.*[1] reported a value of specific moisture extraction rate of 0.4 kilograms of water per kWh in a vacuum freeze-drying process.

In 1959, Meryman[2] evidenced that the water flux from the ice front to the drying chamber depends on the pressure gradient in the dried product, and not on the absolute pressure of the system. As a consequence, it can be possible to carry out the drying process at atmospheric pressure if a water vapour pressure gradient is maintained in the dried product.

In an atmospheric freeze-drying process, the product to be dried is

firstly frozen (at about -10°C) and then it is dried with air at temperatures generally ranging from -10 to -3°C, as this appears to be a good compromise between costs and final product quality. In this case, the specific moisture extraction rate can range from 1.5 to 4.6 kilograms of water per kWh[1], and 35 per cent of energy savings can be achieved with respect to vacuum freeze-drying, as reported by Wolff and Gibert[3] for drying potato slices. Besides, almost the same product quality with respect to vacuum freeze-drying is obtained, as shown for many products like strawberries and potatoes, fish products, lactic acid bacteria and many others.

The atmospheric freeze-drying process can be carried out in a tunnel dryer[4]. The product is placed over a belt or a shelf and dry air flows over the product to remove the water. Air temperature, relative humidity and velocity can play a relevant role on drying time. Figures 1A and 1B show the effect of the various operating parameters on drying time of apple samples (*Malus domestica*) in a tunnel dryer. It appears that the drying time is significantly reduced when decreasing air relative humidity or increasing air temperature, while the effect of air velocity is poor. This is due to the fact that in a tunnel freeze-dryer, the velocity of heat and mass transfer is not very high and thus the sublimation flux can be increased mainly acting on the driving force of the sublimation process (i.e. on the difference between water vapour partial pressure at the ice front, dependent on product temperature and in the chamber, dependent on relative humidity).

As an alternative, the product can be freeze-dried in a fluidised bed (Wolff and Gibert[3]), a type of device where a fluid is passed through a granular solid material at a sufficiently high velocity to suspend the solid, and cause it to behave as it were a fluid. When using a fluidised bed it is necessary to granulate the frozen product before drying. Size reduction caused by mechanical cracking is a potential drawback, while the main

advantages are represented by the high velocity of heat and mass transfer processes that can be obtained. In Figures 1C and 1D, the drying time calculated for the atmospheric freeze-drying of apple samples (*Malus domestica*) in a fluidised bed are compared to those obtained in a tunnel freeze-dryer, evidencing that the drying time can be significantly reduced in the first type of equipment.

Spray freeze-drying is a valuable alternative to produce a free-flowing powder, with high surface area, enhanced solubility, and a uniform and ultrafine particle size. Spray freeze-drying into gases (e.g. a refrigerated air stream), liquids and gases over a fluidised bed have been described in the literature. The inclusion of a heat pump has also been proposed to reduce the energy consumption and to carefully control drying temperature and air humidity[5,6].

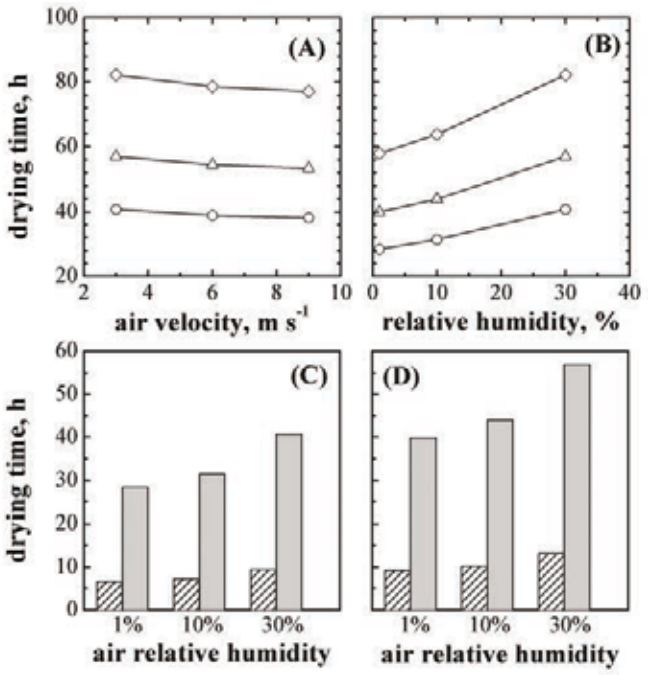

Figure 1: Upper graphs: Effect of air velocity (graph A, air relative humidity = 30 per cent) and air relative humidity (graph B, air velocity = 3 m s^{-1}) on the drying time of apple (Malus domestica) samples (product thickness = 5 mm) in a tunnel dryer for different values of air temperature (○ = -5°C, □□ = -10°C, ◇ = -15°C). Lower graphs: Comparison between the drying time calculated for a tunnel dryer (▩, air velocity = 3 m s^{-1}, product thickness = 5 mm) and in a fluidised bed dryer (▨, air velocity is equal to 1.1 times the minimum fluidisation velocity) for apple (Malus domestica) samples as a function of air relative humidity and temperature (graph C: -5°C; graph D -10°C)

High product quality can be obtained through atmospheric freeze-drying, but the process still entails long drying times. Different alternatives were proposed in order to accelerate the process, such as combining atmospheric freeze-drying in a fluidised bed with the application of microwaves or ultrasound. When using microwaves, the drying rate can be highly increased[7], although corona discharge and non-uniform heating can occur and produce high energy zones; thus the use of microwaves as an additional heat source is beneficial to the process, but further studies are required to improve the control of the energy supplied by microwaves. With respect to the use of ultrasounds, it does not constitute an alternative source of heat for the product, and the increase in the water removal rate is due to mechanical actions on both gas-solid interfaces and product structure[8]. The use of this technique in production units is still limited, probably because of practical difficulties to satisfy industrial-scale requirements.

Another possibility to reduce drying times is to mix the product in the fluidised bed with an adsorbent material. The humidity of the air stream passing through the system is reduced as water adsorbs on this material. In this way, the rate of the drying process can be increased as the product is exposed to dry air; moreover, as water adsorption produces heat, this can be used for ice sublimation. A further advantage related to the use of an adsorbent medium in the system is that a dry air stream leaving the system is obtained. As the air is recycled (due to the low temperature), this is beneficial as the heat load for air cooling and drying (before recycling) is reduced.

Wolff and Gibert[9] employed starch as adsorbent, while Di Matteo *et al.*[10] tested different materials like zeolites, bran and corn flour.

Zeolites appeared to be the best adsorbent material, but the compatibility with the foodstuff has to be taken into account. In this respect, corn flour appeared to be the most efficient adsorbent. Unfortunately because of its irregular shape and hardness, it causes mechanical damages to the product due to the attrition. Due to its compatibility with food products, and it's very low price since it is a by-product of wheat processing, wheat bran seems to be an interesting material to be applied in atmospheric freeze-drying.

Various problems were detected by Di Matteo *et al.*[10] when carrying out these experiments. Segregation might take place when two or more different particles are mixed in a fluidised bed at some stage of the drying process, and the adsorbent material can form agglomerates and channels, which causes uneven behaviour of the bed of fines during the process. With the goal being to take advantage of the presence of the adsorbent medium, it is necessary to get a uniform distribution of solid particles in the bed. If this is not the case, then water vapour could not be removed effectively by the adsorbent medium and heat transfer to the product could be impaired.

Wheat bran shows a particular behaviour during fluidisation. Unlike sand or other materials which present regular bubble formations, it exhibits canalisation or preferential air paths formation, with non-regular formation of bubbles. Furthermore, channels and unstable bubbles formation, as well as limited increase of bed height, are quite clear signs of mechanical particles interaction due to their characteristics such as rough surface and rest of grain brushes.

Unlike other cases where a binary mixture is fluidised in a bubbling fluidised bed and bubbles are the main mixing agent, in channelling fluidised bed mixing is due to the shaking of the bed, and to channels generation and collapse. Thus, three main types of product particle movement mechanisms can be observed: passive transport (downward), active transport (upward) and movement blocking. Moreover, the presence and frequency of these mechanisms depends on air superficial velocity. Passive transport is the downward direction movement of foodstuff material as a result of the dragging of adsorbent material below product particles by air through channels and its deposition on the bed surface. On the opposite side, a food particle is actively transported when it is dragged by air and other bran particles

through the channels in upward direction. Finally, as a result of interaction between adsorbent particles, two main effects causing particle blocking may be identified: floor effect and roof effect. The former is caused by agglomerates of bran particles or more compact zones avoiding the fall of product particles. The compaction level decreases augmenting air superficial velocity mainly because of a greater motion in the fluidised bed and a larger number of channels.

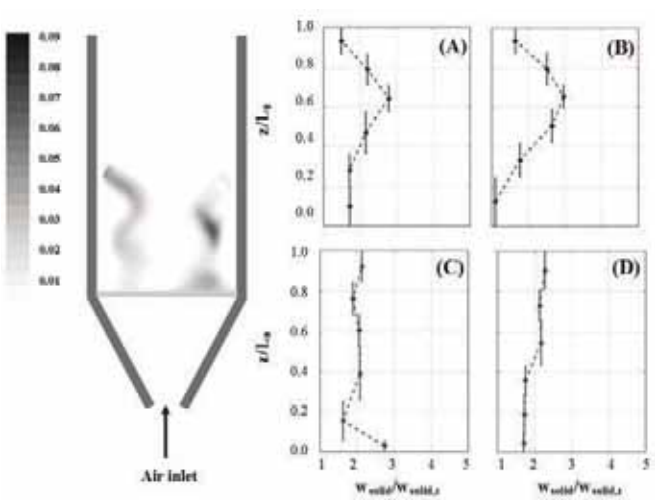

Figure 2: L.H.S.: Carrots volume fraction at the end of the atmospheric freeze-drying of carrot samples in a fluidised bed using wheat bran as adsorbent. The reactor has a square section, whose side length is 0.35 m; the ratio between adsorbent and fresh product is 20, and the air velocity is 1.5 times the minimum fluidization velocity. R.H.S. Influence of air velocity (v_{air}, given as multiple of the minimum fluidisation velocity, v_{mf}) and the ratio between the amount of adsorbent and of foodstuff (A/F) on the carrots axial distribution: this is given as ratio between mass fraction of carrot samples (w_{solid}) divided by the mass fraction of carrot samples at the end of the drying process ($w_{solid,t}$). Graph A: v_{air} = 1.5 v_{mf}, A/R= 20; graph B: v_{air} = 1.5 v_{mf}, A/R= 80; graph C: v_{air} = 2.6 v_{mf}, A/R= 20; graph D: v_{air}= 2.6 v_{mf}, A/R= 80. The carrot disks have a diameter of 1.8 cm, and a thickness of 5 mm; The total bed height at rest, L_0 = 0.38 m

The latter blocking effect occurs when the path of a food particle is obstructed due to the collapse of a channel wall.

Atmospheric freeze-drying of carrot-bran mixtures was studied by means of simulation, using Computational Fluid Dynamics, and by means of experiments in a laboratory-scale unit. Some of the results obtained are shown in **Figure 2** (page 11). Mathematical simulation clearly evidenced a non-uniform distribution of carrot samples in the bed (**Figure 2**, on the left), and this was confirmed by experiments (**Figure 2**, on the right). Various operating parameters can affect the food distribution in the bed, namely the ratio between the air superficial velocity to bran minimum fluidisation velocity, and the ratio between the amount of food and of bran. As is shown in **Figure 2,** on the right, when the air velocity is increased, an even distribution of the food samples in the bed is obtained. With respect to the ratio between the amount of adsorbent and of the foodstuff, this parameter seems to have a weak effect on the mixing of the system, at least for the case study investigated. In fact, as reported by Di Matteo *et al.*[10] if the amount of adsorbent is high, then there is a significant portion of the freeze-drying chamber that is unavailable for product processing, while in case the amount of adsorbent is low, a poor contact between product and adsorbent is obtained.

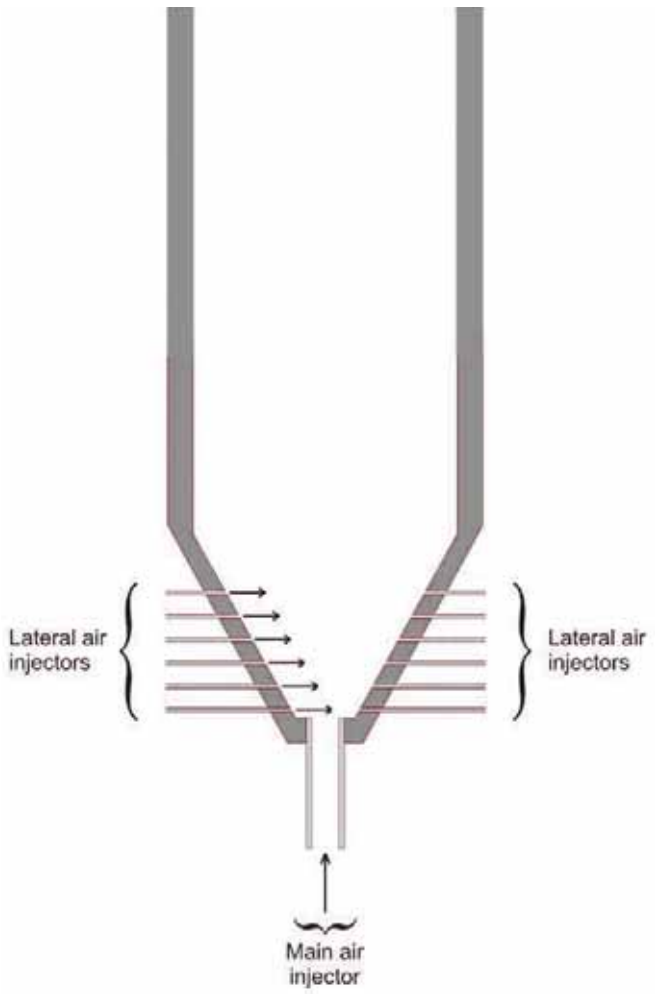

Figure 3: Sketch of the modified spouted bed with lateral air injections

Atmospheric freeze-drying of carrot-bran mixtures was studied by means of simulation, using Computational Fluid Dynamics, and by means of experiments in a laboratory-scale unit

One possible solution for improving mixing in the fluidised bed with adsorbent might be to employ a hybrid spouted bed which possesses lateral air injectors in the bed bottom, besides its central air jet (**Figure 3**). Thus, the food particles and adsorbent would be transported actively in the central channel to bed surface, and passively in downward direction in channel laterals. Lateral air injectors would avoid product concentration in bed bottom pushing it to the central channel, particularly during the first stages of the drying process. Therefore, a better contact product-adsorbent may be reached and food particle blocking effects might be mitigated.

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How much sodium is in your food?

Claudia Haller and Christian Walter Mettler Toledo

Sodium enhances the taste of food, but may lead to hypertension if consumed in excess. Measuring sodium content has, therefore, become imperative for food producers. The new sodium analyser from Mettler Toledo provides a simpler and more cost effective analytical method than existing spectroscopic or chromatographic methods.

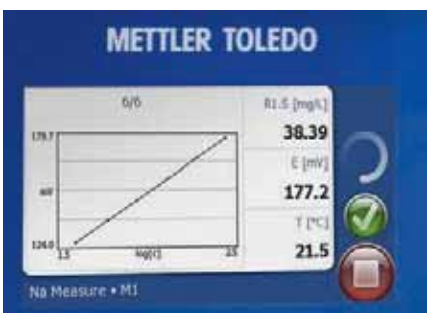
Sodium: important, but not easy to measure

In recent years, society has become aware of the dangers of excessive sodium intake. Pressure is mounting for food manufacturers to accurately determine, and subsequently reduce, the sodium content of their food produce. Direct sodium measurement is commonly performed by atomic absorption spectroscopy, ion chromatography or inductively coupled plasma spectroscopy. These techniques demand a significant capital investment, ultrapure reagents and lengthy sample preparation and system calibration. Therefore, direct sodium determination is often substituted by argentometric chloride titration, which is simple and very accurate. The results are, however, not comparable due to other sodium - (glutamate, citrate) and chloride compounds (KCl).

A lab technician is preparing a snack food sample for the sodium analyser by shredding with piston and mortar

A new approach to sodium analysis

The new sodium analyser from Mettler Toledo provides a very simple, accurate and cost effective tool for the direct determination of sodium. It uses a sodium-specific glass electrode (ISE) and applies a multiple standard addition with a built-in dosing burette. Thanks to a special evaluation algorithm, it delivers highly accurate and repeatable sodium analyses. It is possible to obtain accurate sodium results in just three steps:



The touch control of the sodium analyser at the end of a multiple standard addition, showing the linearity of the test as well as the result

- Weigh or pipette sample
- Add water and Ionic strength adjuster (ISA) solution
- Measure and display calculated sodium content with one click.

One of the great advantages of the multiple standard addition technique is that system calibration and adjustment are unnecessary. Therefore, it is no longer necessary to prepare multiple dilutions and daily, lengthy procedures to prepare the system are a thing of the past.

Great applicative versatility

Most food products containing sodium can be determined directly without extensive sample preparation. Potato crisps, ketchup, sauces, soups, biscuits, yoghurt, seasoning, salad dressing or mineral water are just some of the examples that can be tested with the sodium analyser. Table 1 shows some of the results obtained:

Typically, samples are simply prepared by dilution (for some samples, warm water is advantageous) and adding diisopropylamine solution as Ionic Strength Adjuster (ISA). The operator starts the measurement and the sodium analyser works completely independently from this point; stirring the sample, performing the sodium standard additions and computing the result.

The sodium analyser consists of a control unit with touch screen, a dosing burette and a magnetic stirrer stand with electrodes and stirrer

Conclusion: the right tool at the right time

The simplicity and economical price of the sodium analyser will allow many small food companies to perform their own analyses rather than having to rely on external service labs. This not only significantly reduces costs, but also provides much better test coverage of the whole production process. Considering the increased public awareness of the health implications of excess sodium intake, the new sodium analyser from Mettler Toledo has arrived just at the right time.

Table 1

Sample	Result	Precision (srel)
Potato Crisps	0.587%	1.8%
Ketchup	0.465%	2.1%
Mineral Water	273.6ppm	4.2%
Biscuits	0.330%	2.7%
Yoghurt	0.641%	0.064%
Broccoli Soup	40131ppm	1.2%
Ice Tea	0.008%	0.05%
Salad Dressing	0.876%	0.6

Martin G. Wilkinson Department of Life Sciences, University of Limerick

Sodium reduction in ready meals

Daily salt intake, mainly in the form of sodium chloride, is obtained from either discretionary (salt cellar) or non-discretionary sources (processed ready meals, snack foods, restaurants and takeaways). Consumers are now obtaining the vast majority, around 75 per cent, of their sodium intake from non-discretionary sources such as ready meals, many of which may be high in salt[1,2]. It has been reported that consumption of ready meals may contribute up to 70 - 80 per cent of an excess dietary salt intake[5]. Consequently, health and regulatory agencies have become concerned with the high intake of sodium from frozen or chilled ready meal products. The principal public health issue associated with an excess daily salt intake (about 10 - 12 grams) is the increase in blood pressure (≥ 140mmHg systolic or ≥ 90 mmHg diastolic) leading to hypertension and cardiovascular disease (CVD)[3,4].

Public health agencies are currently endeavouring to reduce the adult daily intake to six grams of salt. However, consumers are increasingly purchasing ready meals as a result of lifestyle pressures, leaving less time available to cook meals in the home. A key driver of growth in this sector is the availability of a range of frozen or chilled products ranging from ethnic dishes such as Indian, Italian and Mexican to more conventional products such as Cottage Pie and Vegetable Soups. All of these products are quite complex to manufacture and are typically formulated using blends of meat, sauce, cheese, pasta, rice, salt and vegetables, with added flavouring ingredients such as spices and herbs; most of which contain sodium, generally in the form of salt. Currently, ready meal manufacturers are under pressure to reduce the sodium content in their product range. In Ireland, the Department of Agriculture, Food and the Marine (DAFM) funded two large multi-institutional research projects under the Food Institutional Research Measure (FIRM) one of which dealt with salt reduction in frozen ready meals and the other with salt reduction in chilled ready meals. This programme was co-ordinated from the University of Limerick and involved a consortium of academic, industrial and state research partners, each of which provided a particular skill set and research expertise. The industrial research

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partners included a major ready meals manufacturer and a supplier of spices, herbs and flavour ingredients. This article attempts to summarise the research approach taken and the key outputs and findings from both projects.

Consumer awareness of salt issues associated with ready meals

The project initially conducted a survey of Irish consumer's perception of salt content of ready meals and found that it was overwhelmingly negative, with 76 per cent of respondents considering ready meals to be high in sodium. Interestingly, 75 per cent of consumers would opt for a chilled meal over a frozen meal as they were considered healthier. Only six per cent of respondents regularly checked the salt / sodium contents of the ready meals and many were unaware of the hidden salt contents within these meals. Despite this, consumers regularly purchased ready meals despite their health concerns regarding these products[5].

Salt in food technology

To take a step back, food technologists add sodium to food formulations for the sensory (flavour and texture) and microbiological impacts on the quality of the product. Salt reduces water activity (a_w) in foods which can slow down or disrupt vital microbial processes, thereby inhibiting the growth of pathogens (*Staphylococcus aureus, Escherichia coli*) or spoilage microorganisms (e.g *Pseudomonas* spp) and increasing product shelf life. Sodium chloride is added to ready meals for its specific taste properties and also for its ability to enhance or modify the flavour of other ingredients within the product. However, in the case of ready meals, the final sodium content is a composite of individual ingredients each of which can contain salt either as a primary ingredient e.g. in cheddar cheese or as a secondary ingredient e.g. within a powdered spice blend. The list of ingredients contributing to the final sodium content in the product formulations typically includes processed meats, natural cheese, cheese sauces, meat sauces, dried spice and herb blends, concentrated bouillons and flavour enhancers.

Reformulation: the key to sodium reduction

In the first project, three frozen ethnic ready meals were chosen for study namely; Chicken Curry, Chilli Con Carne and Beef Lasagne. In the second project, the focus was on sodium reduction in the following chilled ready meals: Cottage Pie, Vegetable Soup and Chicken Supreme. All products were selected on the basis of being mainstream, high volume products, each of which differed significantly in the complexity of their manufacturing process, ingredient formulation and sensory attributes. In conjunction with the industrial ready meals manufacturer, each of the product formulations were assessed for the contribution of individual ingredients to the final declared sodium content. Thereafter, reformulation was undertaken to significantly reduce their final salt contents. This involved reviewing each product ingredient, its addition rate and computing its contribution to salt / sodium levels. Reformulation was then undertaken by: (a) reduction in addition levels of highly salted ingredients e.g. bouillon, cheese or meat sauces, (b) sourcing of commercial low sodium alternatives such as low sodium spices / herbs, and low sodium processed meats, (c) evaluation of commercial salt replacers and flavour enhancers, and, (d) the development of novel low sodium ingredients such as reduced sodium cheddar cheese[6] used both as a grated cheese topping and as an ingredient in the Bechamel sauce layer in Lasagne. Using this approach, Chicken Curry, Chilli con Carne and Lasagne were produced at industrial scale with sodium contents reduced by 66, 60 or 50 per cent, respectively, compared with regular sodium commercial counterparts. In the case of the chilled ready meals Cottage Pie, Chicken Supreme and Vegetable Soup, reduced sodium variants were generated with sodium contents reduced by 50 per cent, 37 per cent and 30 per cent, respectively. Thereafter, the microbiological and sensory aspects of salt reduction were evaluated and compared with regular salt counterparts.

Microbiology of regular and reduced sodium ready meals

Most microbiological studies concentrate on finding inhibitory concentrations of salt for food pathogens using elevated levels up to 10 per cent or beyond. However, relatively little work has been undertaken on the effects of variation in salt at lower levels. Salt concentrations up to three per cent are typically found in commercial ready meals and the rationale was to determine whether bacterial growth was markedly affected by varying salt levels below this value. We commenced a detailed study of the effects of salt in the range three per cent downwards on bacterial growth and survival in broths and following inoculation of ready meals of varying salt contents[7]. In broth systems with up to three per cent added salt, *E. coli* increased by approximately 3 - 5 $\log_{10}$ cycles over the 24 hours in comparison to other bacterial species which experienced a 1 - 2 $\log_{10}$ cycle increase in population over the same period. Little or no difference was found for growth of a range of bacterial species at lower salt levels (~1.5 per cent) including *Bacillus subtilis, Listeria innocua* and *Staphylococcus aureus*. However, at three per cent salt, growth of *E. coli* appeared to be somewhat reduced in broths. These findings were then examined in four thawed ready meals spiked with *E. coli* or *S. aureus* at ~10^5 – 10^6 cfu/g. These meals included; regular salt commercial frozen Chicken Curry, Chilli Con Carne and Beef Lasagne meals, reduced salt equivalents of these three meals and reduced salt meals adjusted by the addition of 0.4 per cent or 2.4 per cent salt (w/v). Growth was followed over 24 hours at various incubation temperatures. In three of the meal types, no lag period was noted for the growth of *E. coli*, which appeared to readily adapt to the compositional conditions

within these products. In agreement with data from broth studies, the salt levels present in all meals during this study did not significantly affect the growth of *E. coli* or *S. aureus*. We noted some moderate inhibition of growth for *E. coli* in Chicken Curry and Beef Lasagne meals adjusted with 2.4 per cent, but growth of *S. aureus* was unaffected by any of the salt levels in the meals[6].

We undertook a further investigation of the microbial quality of regular and reformulated reduced sodium products following thawing and subsequent storage at 4°C, 8°C or ambient (23°C) temperatures for eight days[8]. At 4°C, Beef Lasagne and Chilli Con Carne had unacceptable microbial quality after eight days storage. When these products were stored at 10°C, bacterial populations exceeded recommended levels by Day Three[9]. At ambient storage, rapid increases in bacterial populations were found in all meals and by Day Three, were well in excess of recommended levels. Microbial populations of reduced salt meals at 4°C storage were within the microbiological guidelines of aerobic plate counts (APC) of 4 - < 5 and ≥ 5 $\log_{10}$ CFU/g[9]. We concluded that under the experimental conditions of these studies a salt reduction of ~50 per cent by reformulation did not adversely affect microbiological quality as microbial populations in the reduced salt meals were generally similar to those in regular salt counterparts. Industrial manufacture of layered products such as Beef Lasagne can involve a higher operator input at depositing stations. Therefore we explored the implications of contamination of individual layers and demonstrated that migration of *E. coli* occurred during storage to adjoining layers. We concluded that overall microbial safety of the multi-layered products product relies heavily on ensuring the safety of individual layers of the product at each point of production[10].

Sensory and bioactive aspects of herbs, spices, salt replacers, salt substitutes, flavour enhancers in reduced sodium ready meals

In both research projects, we examined the role of herbs, spices, protein hydrolysates, commercial salt replacers, flavour enhancers or salt substitutes for sensory improvement of various reformulated reduced sodium products. The application of flavour enhancers, salt replacers or salt substitutes in Beef Lasagne[11], Chicken Curry[12] or Chilli con Carne[13] meals was quite successful with positive sensory impacts. In general, inclusion of these ingredients allowed additional reductions in salt contents of reformulated reduced salt ready meals. In the case of Chilli Con Carne, product reformulation to a 40 per cent salt reduction could be achieved without consumers noticing a significant taste difference. However, the inclusion of salt substitutes such as a commercial nucleotide yeast extract allowed for an extension to 60 per cent in the extent of salt reduction in this product[13]. A direct comparison of the instrumental and sensory flavour profile was undertaken between a commercial regular salt and a reformulated 50 per cent reduced salt Vegetable Soup[14]. Reduced salt vegetable soup had the weaker overall sensory profile with more prominent sensory attributes such as 'green colour', 'sweet flavour' and 'pepper flavour'. Other sensory differences found in the reduced salt soup included a more pronounced 'sweet' and 'pepper' type flavours with a reduced 'aftertaste'. Volatile headspace analysis using SPME-GC-MS also detected differences due to salt reduction including a much lower level of terpenes in the reduced salt vegetable soup. It was therefore clear that salt reduction may generate flavour deficits in products which may be remedied using flavourings such as herb or spice blends.

Microbial quality

We examined the role of commercial herbs and spices as potential routes for contamination in ready meals. Briefly, many of the industrial spices and herbs had some degree of bacterial contamination while around 80 per cent of spices contained spore forming bacteria from about 10^2 – 10^6 cfu/g. We examined the survival of bacteria in the more heavily contaminated spices following a simulated industrial heating process and compared spices suspended in water and those added to a chicken supreme ready meal. We noted a marked protective effect of the meal matrix with significant survival of both mesophilic and thermophilic bacteria despite exposure to 90°C for 30 minutes. This study suggests that the quality of spices and herbs added into ready meals may influence shelf life and care must be taken in the microbiological quality specification when spices and herbs are selected for addition into reduced sodium products[15].

Bioactivity

Herbs and spices are included in most ready meals and may provide an avenue for salt replacement or as functional bioactive preparations with anti-microbial or antioxidant activities. The anti-microbial potential of extracts of industrial herbs and spices was investigated against a number of food pathogens including *L. innocua, E. coli, S. aureus* and *Pseudomonas fluorescens.* Ethanol or hexane extracts of oregano, clove, sage, rosemary and celery displayed bacteriostatic or bactericidal activities against four foodborne bacteria and could be potentially added into reduced salt ready meals for enhancement of shelf life[16]. In terms of antioxidant potential, we screened 30 spices used in ready meal manufacture and compared them with synthetic antioxidants. Clove had highest antioxidant potential while cinnamon, bay, rosemary, oregano, sage and marjoram also displayed some activity. In contrast, garlic powder had minimal antioxidant activity[17]. Based on the above information, we selected a number of herbs and spices for inclusion into reformulated reduced sodium chilled ready meals produced at 500 kilograms batch scale[18]. In total, five products were manufactured; (1) commercial regular salt Vegetable Soup; (2) reformulated reduced sodium soup; (3) reduced sodium soup with 0.15 per cent added rosemary; (4) reduced sodium soup with 0.1 per cent added Lactoferrin and (5) reduced sodium soup with 0.05 per cent inclusion of a spice blend of rosemary, sage and oregano. Consumer acceptance testing was undertaken and revealed that all products were acceptable. Highest acceptability was found for the regular sodium soup but was not significantly different for three out of the four reduced sodium products. It was concluded that the addition of rosemary or lactoferrin increased acceptability and allowed removal of around 50 per cent of sodium content without impacting negatively on consumer acceptability[18]. Both the latter soups were positively correlated with purchasing intent of consumers which indicates a successful outcome to the research approach taken. In a final study, a novel proprietary process for production of a lactoferrin hydrolysate (LFH) salt replacer was developed at the University of Limerick and this ingredient included in a trial with selected spice blends in the following reduced salt ready meals: Vegetable soup, Chicken Supreme and Cottage Pie. Positive sensory data from the reformulated meals with indicated the potential of the LFH ingredient as a natural salt replacer at addition levels of 0.1 per cent (w/w)[19].

Conclusions

The reformulation approach to salt reduction was shown to be technologically feasible, especially when combined with a microbiological and sensory optimisation process. This integrated multi-disciplinary research approach can provide the food industry with a template with which to generate ready meals having significant reductions in salt / sodium contents with benefits for both producers and consumers of these products. However, each product must be considered on case by case basis to ensure comparable microbial safety with commercially available regular salt counterparts. Finally, salt reduction by the food industry should be undertaken in conjunction with clear, understandable and unambiguous labelling of salt and/or sodium contents of ready-meals to enable consumers make informed health dietary choices.

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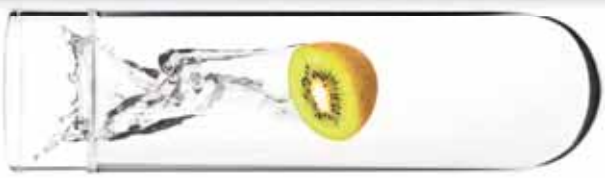

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Back-tracing environmental toxicants in an animal-derived food chain based on food metabolomics

Erwan Engel, Jérémy Ratel and Christelle Planche
INRA, UR370 QuaPA Research Unit, Microcontaminants, Aroma and Separation Science (MASS) Group

Food-producing animals are exposed to toxic micropollutants via their environment and feeds. These micropollutants represent a chemical human health hazard because they are capable of entering the animals and being transferred to edible tissues[1]. Most current approaches to assessing contamination levels in foods are based on high performance analytical methods designed to determine the concentration of targeted micropollutant residues present down to trace levels in the food. Alternative analytical approaches have been proposed that consist of seeking indirect markers of exposure to micropollutants, e.g. metabolites such as lipids, amino acids, simple sugars or cofactors.

Exposure to micropollutants, and the ensuing development of pathologies, gives rise to biological stresses that alter the metabolic profile of body tissues, thereby generating specific metabolic signatures. These metabolic signatures are currently obtained by techniques based on mass spectrometry, NMR spectrometry or 2D gel electrophoresis. The possibility that some micropollutants may be metabolised and thus become practically undetectable by direct measurement methods argues strongly for seeking persistent metabolic signatures of exposure to micropollutants in animal food products. If costless and fast analytical techniques based on metabolomics were developed to quantify these biomarkers, it would be interesting to use them for a routine control of food chain contamination to micropollutants.

Volatile compounds occur in the bodies of humans and animals and their presence is mainly the result of their biological activity. Some volatile compounds in blood were found to differ significantly between healthy and liver cancer patients[2]. A recent review[3] highlights the relevance of the volatile compounds to diagnose some diseases like cancer and proposes their use for personalised screening, diagnosis and treatment monitoring. Volatile compounds have been also identified in animal tissues and fluids like markers of the conditions of livestock production[4]. These compounds may thus be regarded as potential biomarkers of any deviation of the metabolism in response to nutrition, pathology or exposure to micropollutants.

A novel rapid approach based on a non-targeted analysis of volatile compounds in poultry liver was assessed to detect previous dietary exposure to different types of xenobiotic. The micropollutants under study include the main environmental micropollutants susceptible of being found in poultry, including dioxins (PCDD/Fs), polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs) and polycyclic aromatic hydrocarbons (PAHs).

Experimental livestock breeding for the animal exposure to micropollutants

Chicken was chosen as the experimental animal in this work to investigate the feasibility of using volatile compound signatures to detect previous dietary exposure to different types of xenobiotic. Five groups of broiler chickens were reared indoors in individual coops under controlled conditions. Within each group, the chickens were exclusively given a control feed that was either non-contaminated (control group) or contaminated with one of four different types of xenobiotic mixtures regarded as contaminants commonly found in the poultry production chain, namely PCDD/Fs (17 congeners), PCBs (Aroclor 1254), PBDEs (BDE-47, BDE-99) and PAHs (benzo[a]pyrene, pyrene, phenanthrene).

For the quantification of the micropollutants, a representative sample of each of the five experimental feeds was collected and the chicken livers were sampled immediately after slaughter according to Berge *et al.*[5] The micropollutants were quantified in all feed samples and in the liver samples. The PCDD/Fs, PCBs and PBDEs were quantified by gas chromatography coupled with high-resolution mass spectrometry (GC-HRMS) and the PAHs by gas chromatography coupled with tandem mass spectrometry (GC-MS/MS)[5].

For the generation of volatile compound signatures, the volatile compounds of liver samples were extracted by solid-phase microextraction (SPME) and the analysis was performed by GC-full scan MS. The chromatograms obtained in full scan by SPME-GC-MS were converted into virtual SPME-MS fingerprints[5]. SPME-MS signatures were

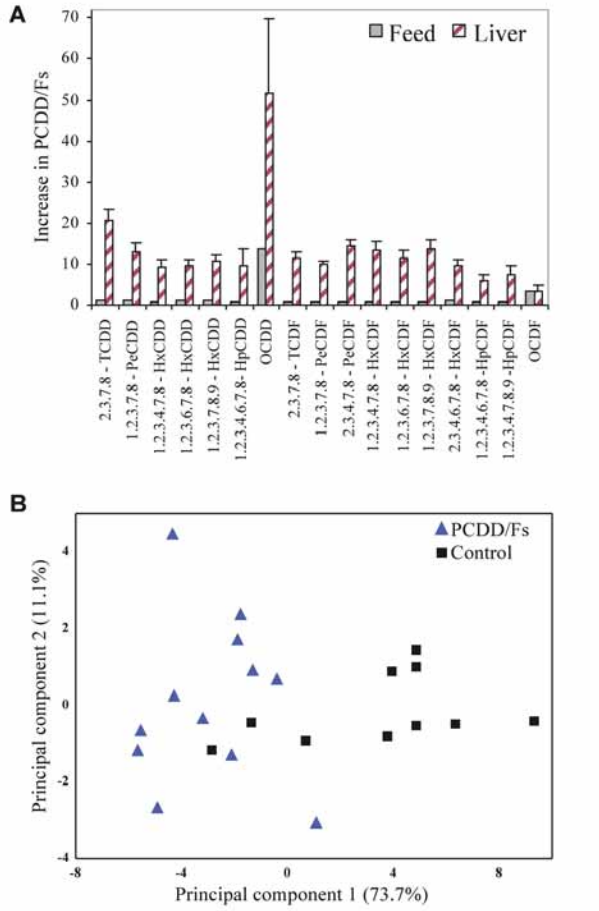

Figure 1: Detection of chicken exposure to PCDD/Fs by analysis of livers. **(A)** Increase in PCDD/F concentration in contaminated feed (pg/g) and liver samples (pg/g fat) compared with control samples. **(B)** Use of metabolic signatures in volatile compounds generated by SPME-MS: first map of normed PCA plotted on the most discriminant log ratios of mass fragments selected in the SPME-MS signatures

normalised according to Berge *et al.*[5] and data were processed using Statistica and R software.

Are the volatile compound signatures in animal liver usable for the detection of previous dietary exposure to micropollutants?

PCDD/Fs and PCBs

The feed given to the animals was properly contaminated with the 17 toxic PCDD/Fs congeners, the concentrations of which were found significantly higher in the deliberately contaminated diet than in the control diet. The PCCD/F level reached 3.14 pg WHO TEQ/g (toxic equivalency factor used by the World Health Organisation) on a 12 per cent moisture basis in the deliberately contaminated diet, which is higher than but within the same range of magnitude as the European Food Safety Authority (EFSA) standard set at 0.7 pg WHO TEQ/g on a 12 per cent moisture basis for the maximum levels of these contaminants[6]. Similarly, the concentrations of the 17 congeners were much higher in the liver of contaminated chickens than in the control group, evidencing their transfer from the feed to the liver tissue. Similar trends for Aroclor 1254 PCB congeners were observed, with higher levels in contaminated samples for both feed and liver than in corresponding controls. Dioxin-like PCB levels reached 0.08 WHO TEQ/g on a 12 per cent moisture basis in the deliberately contaminated diet, which was lower than EFSA standards set at 0.3 (action level) and 0.5 pg (maximum level) WHO TEQ/g on a 12 per cent moisture basis[6]. Previous studies showed that after animals had been exposed to PCDD/Fs and PCBs, these micropollutants were transferred in the blood to organs such as the liver and adipose tissues[7]. The examination of the ratios between liver PCDD/F increase and feed PCDD/F increase (Figure 1A) confirms previous reports showing that the transfer from feed to liver tends to decrease with an increasing number of chlorine substituent for both PCDDs and PCDFs[8], which is particularly significant when tetra-, hepta- and octa-chlorinated congeners are compared. Figure 1B presents the first map of the Principal Component Analysis (PCA) processed on the volatile compound signature in the liver of chickens contaminated or non-contaminated with PCDD/Fs. The overlaying of PCDD/F group plots with the control group plots suggests that the exposure to dioxins had no direct or indirect influence on the liver volatile compound composition and that these micropollutants with closely similar chemical structures are practically non-metabolised and so accumulate in this organ. Similar trends for PCBs were observed.

PAHs

The feed was also properly contaminated with the three PAHs, the average concentrations of which were found to be significantly higher in the deliberately contaminated feed than in the control feed. The feed contamination level (0.72 µg/g fresh matter) was five-fold higher than the average PAH level measured in real-life chicken feed[9], thus demonstrating that the PAH dose given to the chicken was significant

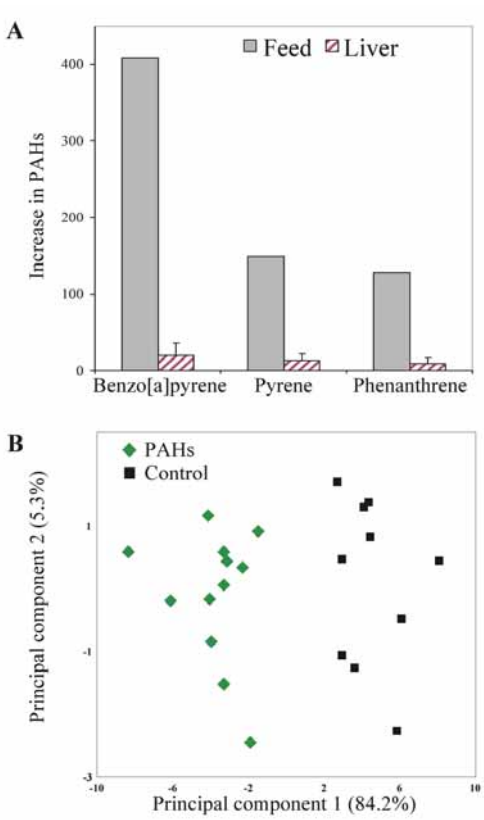

Figure 2: Detection of chicken exposure to PAHs by analysis of livers. **(A)** Increase in PAH concentration in contaminated feed (ng/g) and liver samples (×10 ng/g fresh matter) compared with their counterparts from control non-contaminated samples. **(B)** Use of metabolic signatures in volatile compounds generated by SPME-MS: first map of normed PCA plotted on the most discriminant log ratios of mass fragments selected in the SPME-MS signatures

and realistic. By contrast, **Figure 2A** (page 21) also evidences that none of these three PAHs was detected at a significantly higher concentration in the liver of contaminated chickens than in the control group. This can be explained by a rapid metabolic detoxification of these compounds. Animal tissues are reputed to have the capacity to metabolise PAHs quickly following dietary exposure[10]. The enzymatic systems that metabolise PAHs are widely distributed in the tissues of both humans and animals, and the liver is the organ that exhibits the highest capacity to metabolise PAHs[11]. **Figure 2B** (page 21) points out that changes in animal metabolism occur in response to dietary contamination by PAHs, and that these changes are perceptible through the clearly distinct volatile compound signatures found in the PAHs in contaminated and control non-contaminated groups.

PBDEs

As for the two other groups of micropollutants, the feed was again properly contaminated with PBDEs. While the concentration of BDE-99 was much higher in the liver of contaminated chickens than in controls, revealing its transfer from the feed to this organ, **Figure 3** shows a clear distinction of the PBDE group from the control group. As for PAHs, these results suggest that changes in metabolism occurred in response to dietary contamination by PBDEs, and that these changes were perceptible through the volatile compound signature.

Conclusion

Volatile compound signature evidenced a liver metabolic response to PAHs although these rapidly metabolised xenobiotics are undetectable in this organ by the reference methods. Similarly, the volatile compound metabolic signature enabled differentiation of the non-contaminated chickens from those contaminated with PBDEs. In contrast, no clear signature was pointed out for slowly metabolised compounds such as PCDD/Fs and PCBs, although their residues were found in liver. Further investigation undertaken to evaluate whether the use of more resolutive metabolic signatures, e.g. signatures based on volatile compound profiles generated by GC-MS or GC×GC-MS techniques, can reveal more subtle differences in the metabolic responses of chickens. In addition, these techniques are needed to identify robust biomarkers of exposure to a particular xenobiotic. By opening up the prospect of using these high-resolution techniques to improve the selectivity, sensitivity and specificity of the approach, the present study might pave the way to a new generation of monitoring methods based on metabolomics which are not based on the measurement of pollutant residues or their parent metabolites.

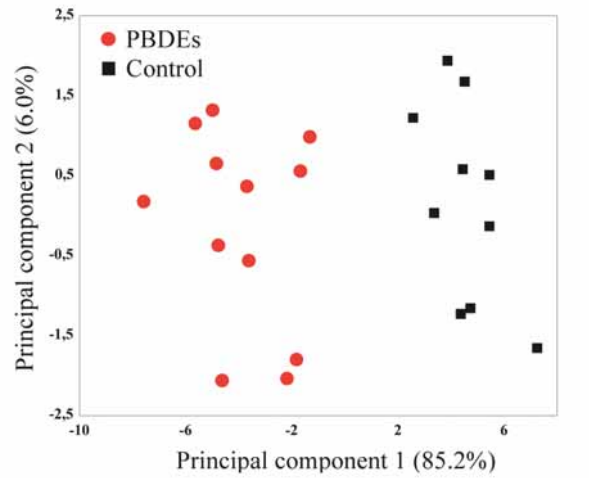

Figure 3: Detection of chicken exposure to PBDEs based on metabolic signatures in volatile compounds generated by SPME-MS analysis of livers: first map of normed PCA plotted on the most discriminant log ratios of mass fragments selected in the SPME-MS signatures

Acknowledgement

We would like to thank Patrick Blinet (INRA, UR370 QuaPA), Bruno Le Bizec (ONIRIS-LABERCA), Catherine Jondreville and Cyril Feidt (Nancy University, UR AFPA) for their contribution to this work. Adapted with permission from Berge et al.5, Copyright from American Chemical Society.

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Detecting bacteria in food: harder than searching for a needle in a haystack?

It would have been easier to find a needle in a haystack than to locate variables needed to 'simply' compute the relative difficulty of that task! Although I am employed by the US Department of Agriculture, before writing this article I never knew that there are at least three haystack configurations that vary in volume and weight depending upon the species of grass, drying time, rainfall, packing density, etc. and as for sewing needles, apparently the debate on which is the most common needle is about as mind boggling as figuring out the difference between European versus USA sizing conventions for needles!

Easier to find, bacteria or needle? Note: if one thought it an interesting challenge to develop biosensors for the purpose of detecting one of these bacteria in 1mL (or cm^3) of liquid, they too would have applied for my job with the USDA back in the mid-1990s. The problem first merits some guesstimates. Racking my brain, I recall that typical rod-shaped bacteria such as the second most prevalent *Salmonella* isolated from the US in 2009 foodborne pathogen *Salmonella* typhimurium (aka *Salmonella enterica* subspecies enterica serovar Typhimurium – i.e. a 'bug' that causes typhoid like symptoms in infected mice and can also, at the very least, cause humans to develop gastrointestinal issues) is about 5µm (micrometres or one millionth of a metre) or 0.0005 centimetres long and about 1µm (0.0001 centimetres) wide. Making some assumptions, ignoring close packing (assuming pegs fit nicely into square holes), a volume ratio of one bacterium in 1mL of water is roughly one in 64 billion parts. By comparison, a needle (also assumed to be a rod, but three centimetres in length and 0.1 centimetres wide) and haystack with attributes gleaned from a 1931 USDA technical bulletin[1]; 'wild' hay, dried 30 – 90 days; ~600 ft^3 per short ton (~19 m^3 per metric ton), and for the sake of simplicity, assumed to be in a non-water shedding square with flat top configuration with a base of 20 foot x 20 foot and height of 12.5 foot (~140 m^3) would have a volume ratio of one in 57 billion parts. So the odds of finding a needle are slightly better, but then again, a needle can be seen by eye.

Two competing teams on an episode of the popular TV show

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'Myth Busters' ingeniously tackled the needle in haystack problem via two approaches. One team used water to separate the denser stainless steel needles (four in all) from the 10 bales of dried hay. That team succeeded in locating the needles in a quicker time than the other team that processed their challenge through setting fire to the hay. Unfortunately, bacteria are mostly water and if the water 'burns', the bacteria will surely burn too so neither of these techniques could apply to the bacteria detection conundrum. I wonder if handheld microwave emitters are for sale.

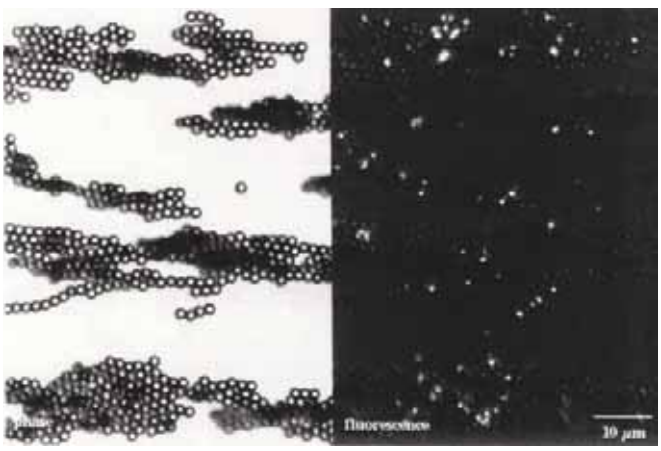

Figure 1: IMB captured fluorescent antibody-labelled bacteria. IMB associated *E. coli* O157:H7 bacteria were treated with a fluorescent, DNA-intercalating dye (4',6-diamidino-2-phenylindole or DAPI) and a typical sample on a microscope slide, located adjacent to a permanent magnet, was viewed using an epifluorescence microscope with phase contrast setup using visible (left) or UV (right; to induce fluorescence) light sources

For years now, it has been cautioned that a person, particularly young, old, or immune-compromised, is more prone to become sick from ingestion of a single, live pathogenic bacteria known as *E. coli* O157:H7 (strains of that serotype of Shiga toxin-producing *E. coli* or STECs) infamous for the Jack in the Box outbreak of 1993. Live bacteria can multiply in a host and thus cause an infection that can lead to devastating effects such as haemolytic fever, kidney failure and brain damage. After optional pre-enrichment or resuscitation in order to revive / recover potentially injured cells, very small amounts of bacteria are traditionally detected after a growth enrichment period typically at least overnight in nutrient-laden culture media. Then growth on solid agar in Petri dishes allows for the visual examination of bacterial colonies for enumeration and possibly cursory identification if selective and/or differential chemicals (e.g., antibiotics, dyes, salts, enzyme substrates, etc.) are used. Bacterial enrichment can be circumvented using microscopy (e.g., phase contrast light microscopy), but aside from the capital outlay for equipment, several concerns arise including: 1) microscopy requires advanced training, 2) analysis time is considerably longer if scrutinisation of multitudes of microscopic fields is performed manually as opposed to using an automated routine since bacteria in samples containing very low numbers of cells are viewed few and far between (e.g., a 5μL sample of bacteria applied to a microscope slide at a concentration of 100,000/mL under a typical 22x22 mm^2 cover slip will only yield about five cells per field of view at 400x magnification), 3) the bacteria may be stuck to and/or have to be distinguished from a large number of food particles, and 4) microscopy alone cannot usually be used to distinguish target pathogenic bacteria from an often relatively large (>1000×) number of relatively benign bacterial cells ('background flora') typically present in many food samples that may only contain a single pathogenic cell or less per gram.

So microscopy is a powerful technique that, given the circumstances, may readily allow for observation of a single bacterial cell. But one has to know where to look! Augmentations to microscopy have enhanced visual-based detection of bacteria. One such augmentation has included labelling bacteria with fluorescent antibodies and capture / concentration of the labelled bacteria on filter membranes for greatly improved, selective viewing using the method antibody-direct epifluorescence technique (Ab-DEFT). Another technique has used antibody-coated superparamagnetic particles (also known as immunomagnetic beads or IMBs – iron oxide encapsulated in plastic microparticles with surface-attached antibodies that can be manipulated by magnets) for a one to two combination of selective capture and concentration of targeted bacteria away from food matrices in a process known as 'immunomagnetic' separation (IMS). Utilising IMS, one simply places a magnet next to a microscope slide and can view 'glowing' bacteria (attached to IMBs) with the aid of a fluorescence microscope (a typical result is portrayed in Figure 1). However, a colleague at the USDA once pointed out a problem with this approach – even though the IMBs were approximately 3μm in diameter, the IMBs in a mere two drops or 100μL (used at approximately 13 million/mL in the detection mixture) could 'daisy-chain' together to a line of beads over one metre long (that makes visual analysis rather challenging when the field of view is only approximately 0.5 millimetres wide)!

What about less manual involvement? Because traditional microbiological and microscopic techniques are time-consuming and somewhat laborious, scientists have been seeking alternatives for decades. These newer, alternative techniques are termed 'rapid

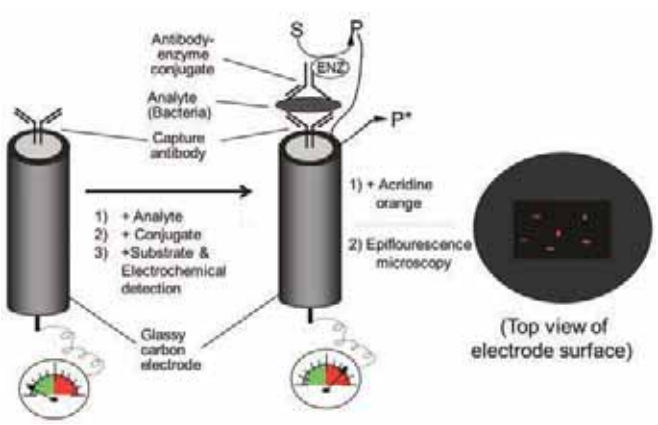

Figure 2: Immunoelectrochemistry (IEC). Attachment of antibodies directly to the surface of an electrode affords the capture and indirect enzymatic labelling of bacteria with a 'sandwich' immunoassay. Further addition of enzyme (ENZ) substrate (S) generates a product (P) that is directly electro-converted at the electrode surface. Captured bacteria can further be stained with fluorescent dyes (e.g., acridine orange) and enumerated with fluorescent microscopy

methods' given that their primary goal is to circumvent time-consuming enrichment culture thus not relying on bacterial growth for increasing concentrations to levels detectable by eye notable with turbid broth cultures or visualisation of macroscopic-sized colonies on agar-based growth media. Aside from clever partitioning of bacteria away from food matrices (e.g., using tandem filtration with multiple membranes of various pore sizes, polylysine- or polymyxin-coated surfaces, or IMS),

rapid methods often are biosensor[2]-based. Aside from relying on biorecognition elements for selectivity, biosensors also frequently employ signal amplification in addition to signal transduction and measurement. Though used for screening, rapid methods are not currently replacements for gold standards of microbial identification (e.g., biochemical testing, serological typing with antisera, and/or genetic typing in combination with selective plate culture) and more so, of confirmation that focuses on analysis of isolated (i.e., monoclonal) pathogens. Albeit, contemporary developments of academic researchers with the Center for Food Safety Engineering at Purdue University hold promise and include improved concentration via enhanced filtration (Ladisch lab) as well as microbial identification via infrared analysis (Mauer lab) and laser scanning of bacterial colonies (BARDOT system; Bhunia lab). The remainder of this article covers the trials and tribulations primarily observed in my biosensor research at the USDA.

A popular rapid method approach has employed relatively inexpensive, simplistic and potentially highly sensitive electrochemical detection. Highly sensitive since electrochemistry may exploit 'local concentration' reaction and detection technique that, depending upon the format, may involve interaction of analytes at electrode surfaces before they dilute into bulk liquid medium that occurs with many other analytical methods including the widespread colorimetric-based immunoassay ELISA (enzyme-linked immunosorbent assay). Such typical biosensors combine antibody selectivity with electrochemical detection and are referred to as 'immunoelectrochemical'(or IEC) techniques. IEC often employs enzyme-linked antibody 'conjugates' that serve as reporter or label molecules. These conjugates greatly enhance selectivity, especially when used to 'sandwich' bacteria between themselves and other biorecognition elements attached to capture surfaces. Chemicals, known as enzyme substrates, that are 'electro-

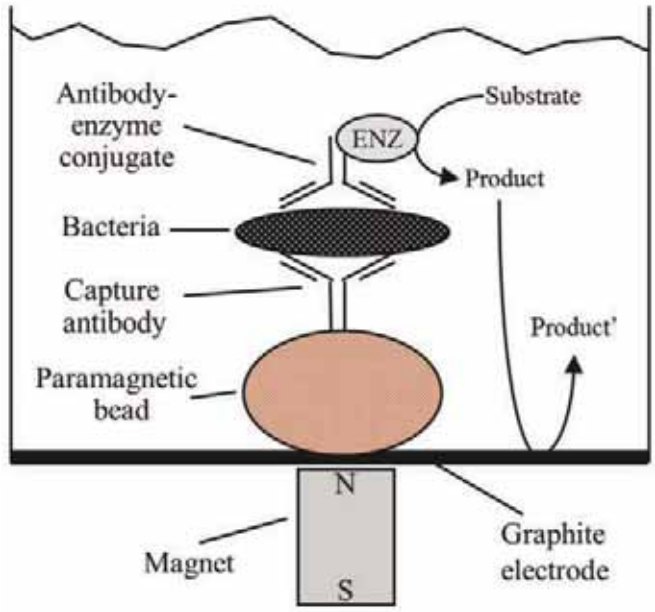

Figure 3: Enzyme-linked immunomagnetic electrochemical (ELIME) detection of bacteria. Similar to ELISA (enzyme-linked immunosorbent assay) in sandwich format that typically employs antibody-coated polystyrene microtitre plate well surfaces, ELIME employs IMS in which antibody-coated superparamagnetic particles (or IMBs) partition captured bacteria away from bulk [food in aqueous medium] mixtures and concentrates the bacteria to an electrode with the aid of a permanent magnet. After reaction with enzyme (ENZ)-antibody conjugate, enzyme-converted electroactive product is then detected directly at the electrode surface

active' can be selectively converted by the antibody-associated enzymes to readily electro-detected products. Furthermore, enzymes catalyse substrate conversion at a high rate and effectively amplify the biorecognition response. Ultimately, electro-converted product concentrations are directly proportional to the number of conjugates which are directly proportional to the number of bacteria captured and detected.

A rudimentary example of IEC, in which capture antibodies were directly adsorbed to a conductive carbon electrode surface, is depicted in **Figure 2** (page 26). This method was reported to detect, at best, 5,000 cells/mL in a total assay time of two hours. The performance was decent, but it could have been vastly improved with higher bacterial cell capture efficiency. Bacteria are mostly water and since they have a very similar density to that of water, they essentially do not move in aqueous mixtures. Therefore, unless they are close to a biorecognition capture surface such as an antibody-coated electrode, the majority of bacteria will not be captured in any semblance of a reasonable amount of

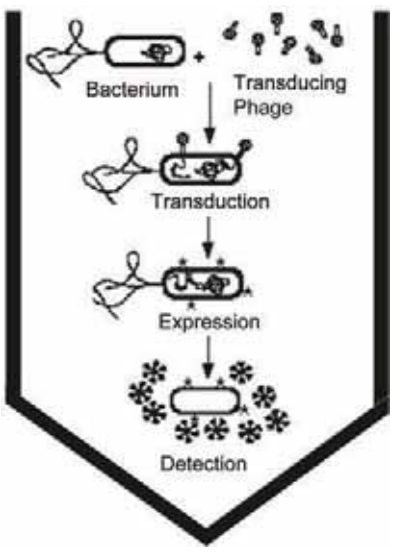

Figure 4: Bacteriophage ice nucleation detection (BIND) assay. Live bacteria are incubated with genetically engineered bacteriophage specific for the targeted bacteria. During three hour incubation, the bacteriophages infect the bacteria and insert DNA that produce ice nucleating proteins that integrate with the bacterial cell membrane. When the aqueous reaction mixture is supercooled (-9.3°C for 20 minutes), mixtures containing infected bacteria freeze

detection time. Using fluorescent dyes for labelling bacteria, it was possible to count the actual number of captured cells at the antibody-coated electrode surface using fluorescence microscopy. Though capture efficiency was observed to be approximately one in a 1000 cells or around 0.1 per cent, the electrochemical response was at a readily detectable level of around 1 nA (nanoamps or one billionth of an amp) per bacteria! Since electrochemical detection is possible to picoAmp (one trillionth of an amp) levels, there was much promise for IEC as a detection platform as long as cell capture was improved.

To enhance bacterial capture efficiency, several variations of IEC as well as other spectroscopic-based detection formats such as colorimetry have employed active capture of bacteria. One variation used filter membranes pushed up against electrodes (e.g., glassy carbon) while others have used IMB magnetically attracted to electrode surfaces (e.g., conductive graphite printed plastic sheeting such as Mylar) during separation and detection (ELIME, **Figure 3**, page 27). Though these two methods had similar detection limits as the previous, they exhibited markedly improved total assay times ranging from 25 – 80 minutes.

Another IEC concept for which a prototype was developed in my USDA lab but regrettably was never pursued for publication was an immunoelectrochemical technique that employed a conductive carbon felt electrode that was incorporated into a flow-through electrode assembly housing. Antibodies readily adsorbed to the surface of the felt, which served as a combination filtration / capture membrane as well as a detection electrode, with extremely high surface area yet very low background current. With the provision that the food matrix is not electroactive (a situation perhaps circumvented with the use of a non-conductive tandem pre-filter), a separated and captured bacterial target was detected after labelling with enzyme-antibody conjugate followed by addition of enzymatic substrate. Cursory results indicated that the system was capable of detection levels around tens of cells per milliliter in a ~15 minute assay.

Another area that was explored was bacteriophage ice nucleation detection (or BIND) that used bacteriophages (very small viruses that can specifically bind and infect bacteria) that infected live *Salmonella* bacteria. Borrowed from the production of artificial snow at ski resorts, this technology was adapted for bacteria detection taking advantage of bacteriophage, specifically reactive with *Salmonella*, which were genetically engineered to introduce DNA that generates a membrane embedded protein that serves as ice nucleating centres on the surface of infected bacterial cells. I had theorised that if unimpeded, a single infected cell in the typical assay reaction volume of around 200µL could initiate a 'branched chain reaction' of freezing during supercooling (approximately -9°C). BIND was indeed demonstrated to detect single cell/mL levels of *Salmonella*. Unfortunately, this technique is not particularly fast, taking at least three hours of reaction time. Furthermore, BIND failed during practical application for the detection of *Salmonella* in chicken carcass rinse water since either background flora or particulates in the rinse, on their own, induced ice nucleation! Also, it was later rumoured that the bacteriophage were shown to cross-react with *E. coli* thus exhibiting false positive results.

It is quite a challenge to detect rather low levels of bacteria, even more so to do it quickly, inexpensively, with high specificity, and of course as also desired by analysts, with a method that is also robust and simple to use. For decades now, research groups worldwide have spent countless hours trying to develop rapid methods as alternatives to traditional microbiological growth enrichment and plating techniques. However there appears to be a universal barrier to limit of detection for bacteria with respect to concentration and time. Advances have been made, but so far the best, most reliable state-of-the-art rapid methods take a few hours to detect levels no lower than approximately 100 cells/mL. Putting things into perspective, it appears relatively easier to find a needle in a haystack, by comparison making detection of a single bacterial cell in small samples such as 1 mL appear to be an insurmountable challenge!

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2. Biosensor= any combination of a biological recognition element (e.g., antibody, aptamers, enzymes, nucleic acids) with a measurement device via a transducer… think crab antennae attached to an electrode connected to a volt meter

Rapid detection methods for chemical hazards in foods

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Foodborne illnesses caused by chemical and microbial hazards are an important health concern in the United States. The new FDA Food Modernisation Safety Act emphasised the need for better detection of and response to food safety problems. Rapid detection methods are critical to prevent the contaminated foods into the market chain as well as to track contaminated food back to the source in an effective, timely manner. To detect the targets in a complex food matrix, there are two critical steps involved; separation and identification. A fast separation method with good recovery is the key for the following identification. This article focuses on the rapid detection methods for chemical hazards in foods including environmental toxins (e.g. pesticides and heavy metals), illegal drugs (e.g. antibiotics), adulterants (e.g. melamine), allergens and microbial toxins. Based on different natures of chemical hazards, the detection methods can greatly vary. Recent advances in method development are highlighted in this article.

Chromatographic techniques for chemical hazards

The most universal technique for detecting chemical hazards is chromatography coupled with different detectors (e.g. UV, fluorescent and mass spectroscopy). Chromatography is used to separate the target chemical from other compounds based on molecular weight, size, affinity and ionic strength. The detector identifies the target. The choice of the detection system is very important for selectivity and sensitivity. High performance liquid chromatography (HPLC) with high automation in injection, elution, washing and detection has been applied extensively as a screening technique.

Recent advances in HPLC technique derived from the evolution of packing materials used to effect the separation. By using smaller particles, speed and peak resolution can be greatly improved. The advanced technique is called ultra-performance liquid chromatography (UPLC). It has demonstrated superior capacity to rapidly and simultaneously quantification of multiple chemical hazards extracted from foods. For example, Kovalczuk *et al.* reported a UPLC-MS/MS method for simultaneous quantification and confirmation of 64 pesticide residues and their toxic metabolites in fruit extracts. The total time required for UPLC-MS/MS analysis was eight minutes plus two minutes for re-equilibration to the initial UPLC conditions[1]. Jia *et al.* used the UPLC-MS/MS method for the simultaneous quantitation of multiclass veterinary drugs (including seven tetracyclines and four types of quinolonein) in eggs within 15 minutes of total run time. The limits of quantitation ranged between 0.02 and 4.29 µg/kg (ppb)[2].

Sensors for chemical hazards: Recognition molecules targeting chemical hazards

A sensor is composed of a recognition element with a detecting element to provide a signal for identification. The recognition element can specifically capture the target, thus separating the target from a complex background. Antibodies are the most common recognition element. The antigen and antibody reaction is very specific and has been used for years to detect a wide variety of food contaminations. Various antibodies are available commercially. Aptamers have received increasing interest recently. Nucleic acid aptamers are selectively engineered single-stranded DNA or RNA that can bind to a specific target molecule. Compared with antibodies, aptamers are easier and cheaper to synthesise and manipulate (e.g. modify the molecule with functional groups). They often offer more stability as well as improved robustness compared to antibodies[3]. For small molecules which have challenges in triggering immunological responses to produce antibodies, aptamers are of particular interest in application. There is a review paper that listed and discussed all the aptamers specific to chemical and microbial food contaminants and their applications in various sensor techniques[4].

Sensor techniques: immunomagnetic separation (IMS)

The use of magnetic beads offers the capacity to rapidly and effectively concentrate and separate the captured target out of a solution with a magnet. There are many commercial IMS kits available for conjugating different antibodies on the protein G or protein A coated beads. Protein G or protein A can bind antibodies without chemical modifications. Streptavidin coated beads can be used for biotin modified aptamer. IMS is actually not a sensor technique but rather a separation method. It has been used prior to detection techniques such as PCR and Raman spectroscopy for concentration and separation of bacteria, toxins and allergens from food matrices[5-7].

Enzyme-linked-immunosorbent assay (ELISA)

ELISA utilises the antibody-antigen interaction and the detection is

based on the colour from the reaction between the enzyme linked on the secondary antibody and the substrate. There are different formats (direct and competitive) for antigen capture and quantification. Many diagnostic companies have marketed ELISA test kits for the detection of chemical residues such as antibodies, allergens and toxins.

Nanoparticle based colorimetric assay

Gold and silver nanoparticles have unique properties in light-scattering and absorption. The nanoparticle-based colorimetric assays have recently attracted considerable attention in detection and diagnostic applications due to their simplicity and versatility without the use of advanced equipment. The mechanism is simple. Dispersed gold nanoparticles with 10 – 50 nanometres in diameter in water appear deep-red colour. As the size of the nanoparticles increases by aggregation, colour will change to purple or blue. Therefore, a target analyte that can induce the nanoparticle aggregation can be detected by the colour change of the gold colloid, providing a simple, direct, rapid sensing assay. A sensitive and accurate detection depends on a strong and specific binding affinity between the target and the recognised molecule on the nanoparticles. For example, the colour change induced by triple hydrogen-bonding recognition between melamine and a cyanuric acid derivative conjugated on the surface of gold nanoparticles was used for detection of melamine. This assay was able to detect melamine in raw milk and infant formula even at a concentration as low as 2.5 ppb within several minutes[8]. This assay was also applied in detection of pesticides[9,10], toxins[11,12] and heavy metals[13-15].

Lateral flow assay

A lateral flow assay is based on the movement of a liquid sample along a strip of polymeric material passing the zones where recognition molecules have been attached to capture the analyte. Then the complex continues moving along the strip to the detection zone where the complex will be stopped and resulted in a colorimetric response. Coloured particles are commonly used as the reporter. This assay gives advantages of on-site and economic detection without an expensive device, similar to the previous described nanoparticle-based colorimetric assay, but offering more user-friendly operation and disposal. This assay has been used for detection of pesticides with excellent detection limits (1 - 10 nM) and rapid response times (about five minutes)[16]. It was also used for detection of Clostridium botulinum neurotoxin[17], aflatoxin[18] and peanut proteins[19].

Electrochemical techniques

The most typical part of electrochemical sensors is the presence of a suitable recognition in the layer providing electroactive substances for detection by the physico-chemical transducer providing the measurable signal[20]. Electrochemistry detection offers considerable promise with features that include remarkable sensitivity, inherent miniaturisation, low cost, and high compatibility with microfabrication technologies[21]. Electrochemical impedance spectroscopy (EIS) based biosensors are well-suited to the detection of binding events happening on the transducer surface since minute changes in analytes to a biosensor surface can be easily and rapidly detected. A simple aptasensor based on

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EIS for sensitive and selective detection of acetamiprid has been developed with a low detection limit of 1 nM[22].

Surface-enhanced Raman spectroscopy (SERS)

SERS is a combination of Raman spectroscopy and nanotechnology. SERS has received increasing interest recently due to its high sensitivity and rapid response time to give a molecular fingerprint spectrum. Raman spectroscopy is one of the vibration spectroscopies that measure the shift in the wavelength of the photons of a laser beam interacted with sample molecules. The advantage of Raman spectroscopy is that the chemical signatures of samples can be measured non-destructively and rapidly. However, spontaneous Raman scattering is typically very weak. The weak Raman scattering can be greatly enhanced by using a noble metal nanostructure[23]. Limit of detection of SERS can go down to single molecule level in some cases[24,25]. SERS has been demonstrating the feasibility of detecting different pesticides and antibiotics with a simplified procedure compared with HPLC[26-29].

To capture specific target from a food matrix, recognition molecules can be conjugated on SERS-active nanosubstrates directly. There are two types of SERS techniques used in sensor applications. One is an ELISA-like assay, which uses a Raman-active dye as a label, similar as the enzyme label in ELISA. The reading will be the molecular fingerprint spectrum of the dye. The advantage of using different dyes is that it can analyse multiple analytes simultaneously. However, the Raman reporter–modified nanoprobe provides only the signature of the reporters and tends to be an imaging tool rather than a detection probe[30]. The intrinsic target analyte information is missing. In addition, the use of a secondary label significantly increases analytical time. The other assay is called 'label free' SERS assay. The measure of intrinsic SERS patterns of the targets proved advantageous in terms of accuracy; this is because spectral changes, after capture, can be compared with the original intrinsic spectra, to confirm that captured target is the analyte and not a food component. This is so-called 'self-validation'. This is also a unique advantage of the label free SERS method, compared with other colorimetric or electric sensing techniques, which rely on colour or electric change only. The label free SERS method has been used in detection of allergens and toxins in different food matrix[7,31] within 20 minutes.

Summary

In summary, there are various techniques for rapid detection of chemical hazards in different food products. The choice of which technique to use depends on the specific chemical target, the food matrix, numbers of samples, cost, requirement for sensitivity and quantification, the availability of the equipment and so on. Nevertheless, the need for a more rapid, sensitive and cost-effective method is always driving the development of new and advanced technologies.

About the Author

Dr. Lili He is an Assistant Professor in the Department of Food Science at the University of Massachusetts. Her research focuses on development and application of advanced and innovative analytical techniques in solving critical and emerging safety and quality issues in food science.

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Mass spectrometry for the food industry

Mass spectrometry is a powerful analytical technique which can be used for the detection and subsequent identification of a wide range of compounds. In the food industry, it can be used in taint investigations, flavour profiling and shelf life studies as well as qualitative and quantitative analysis of specific compounds of interest.

Mass spectrometry is frequently combined with chromatographic systems. Chromatography refers to the separation of compounds in a mixture using a mobile phase to carry the compounds through a stationary phase. This process separates compounds by exploiting the rate the compounds move through a stationary phase. For example with gas chromatography, the mobile phase is an inert gas (such as helium) which carries a gas mixture through a column coated with a solid stationary phase. With liquid chromatography, a liquid mixture is carried through a column (solid stationary phase) by a liquid mobile phase. Separated compounds then pass out of the column into a mass spectrometer which is used for obtaining a response which includes data which can be used to identify compounds by comparison to a library database.

How a typical mass spectrometer works

The sample must first be introduced to the mass spectrometer which operates under vacuum. Once introduced, the sample then passes directly to the ion source where ionisation takes place. Electron ionisation is the most common ionisation technique used, although other ionisation techniques are possible. Chemical ionisation for example is sometimes used when the molecular ion is of interest but is not observed using electron ionisation. Ionised atoms and molecules are directed to the mass analyser where separation according to mass takes place. These separated ions are picked up by the detector and the data is acquired for review in the instrument's associated software.

GC-MS

There are a wide range of gas chromatograph-mass spectrometer systems available on the market, though the costs for instrumentation can vary widely. Two will be discussed briefly here.

With a typical quadrupole, Selective Ion Monitoring (SIM) is frequently used to obtain selective and specific detection while maintaining the sensitivity required for trace analysis. This sensitivity however is reduced when full scan data is acquired. The main advantage with these machines is that they are at the cheaper end of the market, robust and good for routine analysis.

A Time of Flight (TOF) mass spectrometer enables the collection of full scan data without compromising sensitivity. Obtaining full scan data also means that there is the flexibility to screen for other compounds without repeating the sample extraction process (providing the sample

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extraction was appropriate for the type of compound). This means that there is also no need to decide what ions need monitoring prior to sample injection. The TOF-MS is also capable of measuring accurate mass so elemental composition can provide further confirmation of any compounds identified.

LC-MS

When compounds are involatile or thermally instable, liquid chromatography-mass spectrometry is an option. Applications include the analysis of vitamins, pesticide screening and determining whether cleaning agents are present in food products. Only low amounts of sample are required and analysis times can be very short.

With LC-MS-MS, there are added advantages of specificity from the data obtained. Interferences which commonly arise when a liquid chromatograph is linked to a UV detector are removed, allowing for greater sensitivity and confidence that the response is related only to the compound of interest.

Other mass spectrometer techniques

Mass spectrometers are not only used when hyphenated to chromatography systems. Matrix assisted laser desorption ionisation (MALDI) can be used for the analysis of large molecules such as proteins and sugars.

Mass spectrometers can also be used for direct probe analysis. This approach requires a very small amount of sample and may be used for the analysis of plastic food packaging.

Taint analysis

In the food industry, taints refer to undesirable odours or flavours. The problem with taints in food is that they are not always picked up by the majority of people and the detectable odour and taste levels vary according to the type of food or drink. Tainting compounds may be detectable at concentration levels as low as parts per billion by the human senses. The most obvious cause of taints arises from the affected food product either being manufactured with tainted ingredients or coming into direct contact with the tainting source – tainted packaging for example. Food products can also pick up taints from nearby sources. Such sources include flooring, wooden pallets used for transport and storage, and volatile compounds released from diesel fumes into the air.

Common tainting compounds are halogenated phenols and anisoles. Halogenated phenols can be formed if the necessary chemicals are present – phenol will readily react with halogens. This can occur as a result of using a mixture of phenolic and chlorinated disinfectants which then react either in the atmosphere or drains leading to chlorophenols in mains water. Chlorinated disinfectants used for washing floors have also reacted with new polymer flooring to produce chlorophenols. Trichlorophenol and tribromophenol have been used as preservatives and have been found in wood used in crates, pallets and the floors of shipping containers.

Halogenated phenols can be converted to halogenated anisoles via microbial methylation so it is not uncommon to find a mixture of halogenated phenols and anisoles in a tainted product. A well-known example of a halogenated anisole is trichloroanisole in tainted wine. The taint originates from the process of bleaching cork with chlorine, resulting in the formation of trichlorophenol which is then converted to trichloroanisole. There are instances where halogenated anisoles have been the cause of contaminated packaging, leading to a taint in the packaged food. One of the most potent of the halogenated anisoles is tribromoanisole which is frequently found as a result of the methylation of tribromophenol.

Halogenated phenols and anisoles are only part of a whole host of tainting compounds however. The detection and identification of taints frequently depends on the matrix it is detected in. For example, an earthy, musty odour in water can often be attributed to geosmin, which can be produced by blue green algae. There are also cases where a compound responsible for a taint in one type of food would not be perceived as a taint in another. For example, decanoic acid is found in palm kernel oil but in other food products can form by the breakdown of lipids resulting in a soapy taint.

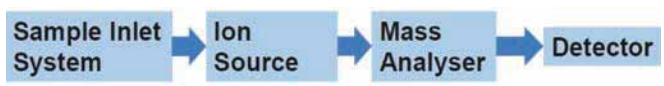

Figure 1: How a typical mass spectrometer works

Sensory analysis complements the chemical analysis of taints since the sensory descriptors can be used to target the analysis and subsequently support instrumental results. The sensory descriptions of the taint's odour and taste can give an indication of the types of compounds to be looked for. 'Antiseptic' and 'disinfectant' descriptors are characteristic of halogenated phenols while halogenated anisoles can be distinguished by their characteristic 'musty', 'mouldy' and 'earthy' descriptors.

Since most tainting compounds are volatile in nature, GC-MS is often used in investigations. Comparative analysis can be carried out using a sample with the perceived taint and a control sample which is similar to the tainted sample but has no taint detectable by taste or odour. The aim of the analysis is to detect compounds in the suspect sample that are not in the tainted sample. Differences are identified using a library search and the sensory characteristics of these compounds can then be checked to determine if they could be the cause of a taint.

Flavour profiling

GC-MS is a commonly used technique in flavour profiling since flavour compounds typically tend to be volatile. While identifications can be made by matching data against a library database, running a solution of the compound identified on the GC-MS would help confirm the compound's identity. This can be particularly useful when using blended flavours where although the ingredients are known, the volatile compounds making up the flavour are not provided by the supplier.

Prior to using GC-MS analysis, it is helpful to have some idea of the type of compounds to optimise the separation of them in a flavour matrix. This is because often many compounds come from the same chemical group e.g. terpenes, ketones, aldehydes. Some software packages enable the deconvolution of peaks which enables the user to determine whether more than one compound is present in a peak response observed. It is worth remembering that the largest peak observed instrumentally may not be the biggest contributor to flavour.

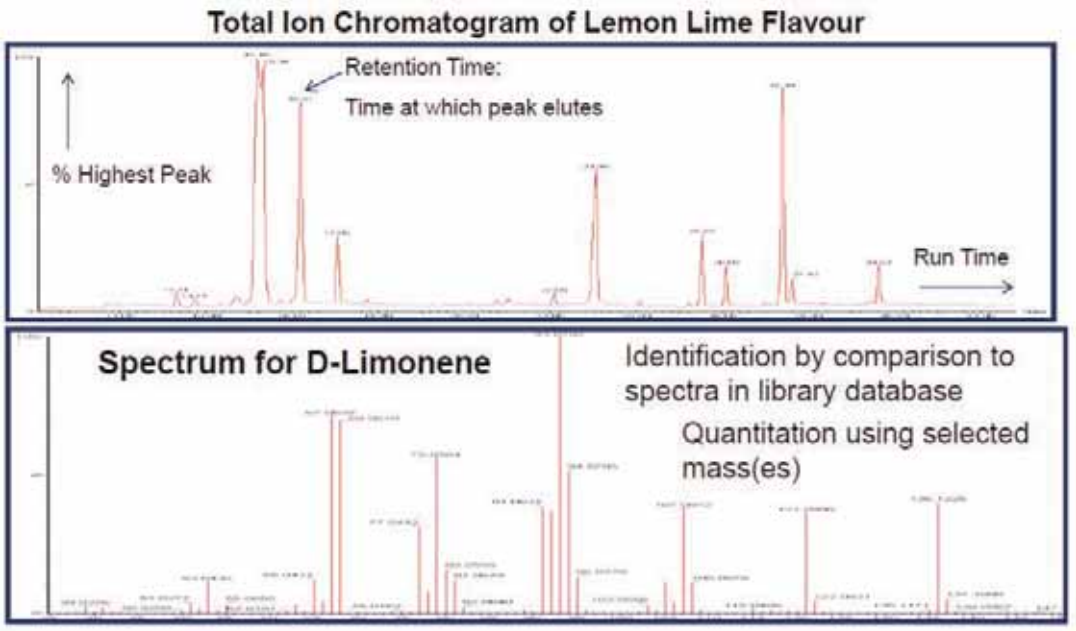

Figure 2: Total ion chromatogram of lemon lime flavour

Shelf life

The shelf life of a product may be defined as the length of time in which a food product remains of satisfactory quality to the consumer. In order for a food product to be of a satisfactory standard for consumption, it is necessary to retain certain sensory attributes such as flavour and colour. Other factors are also of importance such as microbiological stability. Retaining these factors in a food product for a long period of time can represent challenges to the manufacturer, particularly if they wish to change their ingredient list.

Shelf life testing typically involves the measurement of changes in product quality under defined storage conditions, which will involve the comparison of a 'fresh' food product with 'aged' food products. Flavour is one factor which can significantly influence product acceptability. Various compounds contributing to overall flavour may increase, decrease or even reduce to zero detectable levels at certain time points.

Before using a technique such as GC-MS to monitor flavour changes in a food product, ideally as much information regarding the ingredients present would be provided as possible. This is because the flavour compounds must first be extracted from the food product before they can be analysed. A common issue in the laboratory can be the production of other compounds as a result of the extraction technique used which can complicate the process of positively identifying compounds which are responsible for changes in the flavour. Parameters such as temperature used for extraction should be carefully considered to reduce the chances of this occurring.

Flavour changes over time can occur for a number of reasons. Internal chemical reactions may take place within the food product, particularly if it is not stored appropriately. For example, fats may give rise to chemical changes as well as physical changes. Microbial interaction with the food product has also been known to cause flavour changes. This is less of an issue with confectionary since the high level of sugar used usually prevents this from happening.

Interaction of the product with the packaging can also be responsible for changing flavour. It has been known for compounds to migrate into the food product either from the packaging itself or from compounds in contact with the packaging resulting in undesirable off-flavours. For example, the type of paper used for packaging can give rise to a 'cardboard' or 'musty' off-flavour in confectionary such as cakes and biscuits. Such sensory descriptors can be indicative of a certain type of compound but may also be representative of several types of compounds.

To help find out which of the compound(s) identified as changing over time are responsible for off-flavour, again sensory work may be carried out to complement instrumental analysis. In the laboratory, gas chromatography-olfactometry (GC-O) may be employed. The gas chromatograph separates out the different volatile components in an extract and the human assessor is employed to describe the aromas as the different volatile components elute. For the most helpful results, sensory trials of the food product over time must always be carried out as part of shelf-life tests by trained sensory panels.

Once marker compounds for off-flavour have been identified in a food product the levels of the markers should be related to consumer acceptability to determine the cut off points for shelf-life.

Capabilities at Leatherhead Food Research

The Chemistry Research team at Leatherhead Food Research have both a GC-MS-TOF and an LC-MS-MS, enabling the analysis of both volatile and non-volatile compounds using mass spectrometry. The mass spectrometer on the GC-MS can be used as a standalone mass spectrometer for direct probe analysis.

About the Author

Angela Calder has worked in analytical chemistry for over a decade. Prior to joining Leatherhead Food Research as a GC-MS Chemist, she worked in the tobacco industry.

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Addressing new questions with LC-MS

Anyone familiar with American detective dramas on TV, especially the CSI programmes, will have seen their laboratories produce answers to virtually every question posed. From the TV producer's perspective, all that needs to happen is for a sample to be fed into a black box (or a white one) and moments later the computer delivers some crucial piece of evidence that helps to solve the case.

It is fiction, of course, but modern analytical instruments, linked to powerful processing software and extensive databases, are closing the gap between fact and fantasy. In the field of food science, high resolution and high accuracy liquid chromatography-mass spectrometry (LC-MS) is starting to prove its worth in answering questions about food products that technologists would never have dreamed of asking only a few years ago.

These include questions about the impact of chemical composition on flavour or texture or appearance; questions about the impact on the quality of the final product of changes to processes or ingredients; questions about authenticity or purity or contamination; and questions about the impact that our food might have on treating (or causing) diseases. Not that these are the only questions. The beauty of high accuracy/ high resolution LC-MS is that it raises the possibility of posing questions in food science that few people have yet thought to ask.

Benefits of high accuracy/high resolution

As its name implies, mass spectrometry is a technique for detecting and identifying molecules based on their molecular mass. The technique involves ionisation of the molecules and their fragments, and the instrument ultimately measures the mass to charge ratio of the ions produced.

Where mass spectrometry has advantages over other detection techniques is that it is capable of identifying many chemicals simul-

taneously and without necessarily having to know in advance what chemicals to look for. It is applicable to large complex molecules like proteins and oligosaccharides, as well as smaller, simpler molecules like vitamins and other metabolites.

Of course, many foods are extremely complex mixtures of hundreds or thousands of chemicals. That being the case, it is of no surprise that molecules that are very different in terms of structure and composition can have very similar molecular masses. It is the ability of high accuracy, high resolution mass spectrometers to differentiate between the similar but different masses, that makes them so powerful in determining the chemical composition of complex mixtures, and perhaps finding unexpected contaminants / impurities that no-one expected to be there.

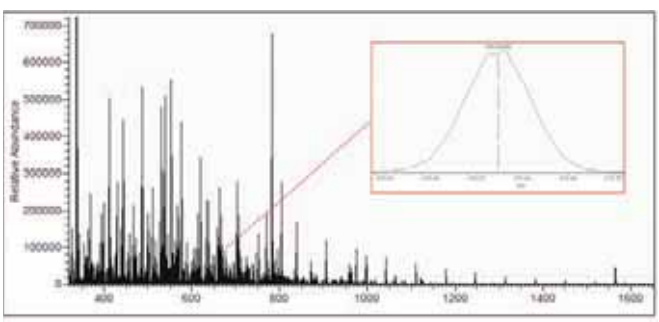

Figure 1: A high resolution instrument is needed to pick out peaks of interest from within the 'noise'

Similarly, high resolution refers to the ability of a mass spectrometer to distinguish between ions of different mass-to-charge ratios. Only a very high resolution mass spectrometer can determine that two molecules of very similar mass are actually distinct and different. Figure 1 shows how a high resolution instrument can pick out a peak of interest from within the noise of many other peaks.

Application areas of LC-MS

LC-MS is interested in the non-volatile chemicals, including the very large molecules such as proteins and DNA. Hence LC-MS has become a powerful tool in the burgeoning science of proteomics (the large scale study of proteins, particularly their structure, function, amount, location in a biological system) and metabolomics (the systematic study of the complete set of small molecule metabolites found in a biological system).

Together, proteomics and metabolomics represent powerful analytical strategies that, in part, could be applied to food science and nutrition, unveiling more detailed information on food composition beyond traditional food component analysis.

In both cases, LC-MS can be used as a screening technique (fingerprint analyses), as well as to focus in on a specific class of molecules (profile analyses), discovering perhaps what happens to a protein under different process conditions, or whether suspected contaminants are present or not.

Proteomics

A combination of developments has enabled scientists to study proteins in different systems, including food products. These include an increase in accuracy, resolution, and scan speed of bench-top mass spectrometer instruments, built with powerful detectors such as the Orbitrap. There have also been improvements in informatics tools to quickly analyse raw data in detail.

Many sections of the food industry are only just starting to question the role of proteins in determining characteristics of a food product such as structure, texture and taste. LC-MS gives researchers a way to understand more about how proteins, and their modifications such as glycation and phosphorylation, affect product performance and quality.

The most basic question to ask is; what proteins are most abundant in any given food product or ingredient? Thereafter, one might also wish to consider how any given protein content or modification affects sensory characteristics, and how these modifications are related to different processing regimes.

Protein analysis

Protein analysis with LC-MS often involves a bottom-up approach, in which the proteins are initially broken up into smaller fragments (peptides) by proteolytic digestion using an enzyme (most probably trypsin). These peptide fragments can be detected in the mass spectrometer, and fragmented in a way that is specific to the instrument used, to generate distinct fragment ions. As an example, if higher-energy collisional dissociation is used to fragment the peptides then fragments along the peptide backbone between the carboxyl carbon and amino nitrogen form a series of so-called b- and y-ions (Figure 2). These ions are then analysed with high accuracy and resolution in the mass spectrometer and the resulting spectrum is compared against those of a protein database that theoretically digests the proteins into peptides and likewise theoretically fragments the peptides.

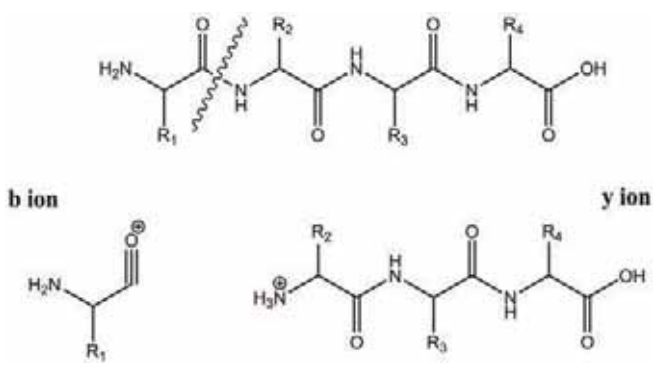

Figure 2: Fragmentation of peptides into b- and y-ions

Ultimately this process yields a probability score that a particular protein is present, or not, in the product. This allows researchers to start forming conclusions about the role of certain proteins in determining product characteristics. For example, one can envisage that a bread baked with one type of flour performs differently from a bread baked with another flour, and that the difference could be attributed to presence or absence of a particular protein or a modification of it due to the process. Or perhaps the creamy texture of a cheese is altered by milk from different cattle species, and that different cows on different diets produce different proteins in their milk. The technique of LC-MS provides a means of asking and answering such questions.

Of course, one must be careful here not to overstate the simplicity of

this procedure. Sample preparation is crucial, and the results from peptide matching will require some interpretation, but compared to other forms of protein analysis, LC-MS is a powerful and rapid tool for researchers looking for a way to understand the proteins in their products and ingredients.

Metabolomics

The same kinds of observations in respect of speed and applicability can be made in relation to the study of metabolites.

The plant metabolome is considered to comprise roughly 10,000 different chemical compounds. Of these, 2,000 are presumed to have some nutritional benefit, whereas 8,000 are not. Ongoing research may result in changes to these calculations.

In any event, there are potentially hundreds of different molecules within any given food, and understanding how the relative chemical compositions of different foods and ingredients impact on aspects of flavour, function, structure, texture and so on is a challenging task.

LC-MS offers up a means of making comparisons, and understanding differences, perhaps when the same product is produced in different factories, or using new formulations, or under different processing regimes and so on. It is also useful in investigating contamination issues; spotting 'rogue' molecules where none were expected.

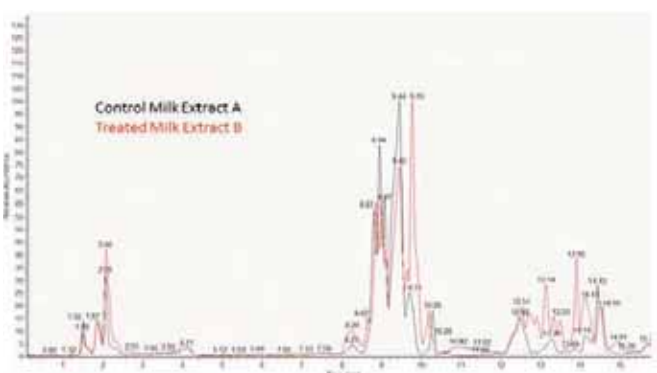

Figure 3: Statistical differentiation of signals can help differentiate between two chromatograms (in this case of dairy samples)

On this last point, we can reflect on some of the major contamination scandals of recent years. For example, when melamine was added to milk, it was done so with the express purpose of 'fooling' protein analysis that simply looked for nitrogen as an indicator of protein content and hence milk quality. The tests applied at the time were blind to the fact that the nitrogen came from a non-protein source, so the deceit went undetected for some time. Had there been a regular LC-MS screen of milk products in place, the melamine would have stood out on the spectrum compared with an uncontaminated product. Similarly, LC-MS can quickly spot the use of artificial dyes in spices (refer to the use of Sudan red), without the analyst needing to know what type of dye has been used. If contamination is merely suspected, LC-MS is likely to find it.

In fact, LC-MS as a screening technique is capable of detecting and identifying any non-volatile chemical that has been added to an ingredient / food. It is also capable of detecting adventitious migration of chemicals from packaging, for example. By the same token, it is also capable of determining that specific markers are absent. So, in the case of vitamin supplementation, for example, LC-MS offers a means of substantiating claims that vitamins are present in the finished product, and of investigating reasons for their absence should that prove to be the case.

Returning to the more positive aspect of using LC-MS to determine how different chemicals affect product quality, **Figure 3** shows how two total ion chromatograms of milk extracts could be differentiated and the sensory characteristics of one of the two samples explained. Just by looking at the overlapped chromatograms it would be very difficult, in fact, to make assumptions of what signals are most abundant in one sample compared to the other.

However, the screening of the overall mass spectrometer files, component extraction, statistical differentiation of signals and hopefully their identification, would help scientists clarifying the changes in chemical composition that could be related to the observed sensory / quality attributes. In this particular example, extract B happened to be bitter, and after differentiation, a series of di- and tri-peptides were identified that could explain its taste. This demonstrates how these tools give researchers / product developers some chemical targets to work towards producing higher quality ingredients / products.

Of course, how easy all this is depends on the food matrix. Many foods are complex mixtures of hydrophilic and hydrophobic molecules. Hence there is always a challenge in developing appropriate sample preparation techniques to extract the chemicals of interest and avoid matrix effects.

Conclusion

The above only gives a taster of the potential for LC-MS to address new questions in food science. That is not to say that the technique is the 'all-knowing' black box of US crime dramas. For example, it is not always possible to determine the precise structure of a chemical from its spectrum. Nonetheless, it does offer a powerful means of addressing compositional questions that are difficult to answer in other ways. Whereas many other analytical techniques are directed towards detecting only specific chemicals, or chemicals with particular structures / chemical bonds, a mass spectrometer 'sees' nearly everything.

Proteomics and metabolomics are fledgling sciences, but growing ones too. They are increasingly being applied in clinical studies to discover if and how different proteins and metabolites impact on human health. With the commercial potential for functional foods, there is a strong case for the food industry to adopt similar methods in understanding and developing its own products. They will help the industry to become better informed, and more able, in future, to substantiate health claims on behalf of products.

About the Author

Gianluca Dimartino gained his PhD in Chemistry from the University of Birmingham. Since 2005, the focus of his work has been in the food industry and includes contributions to the development of new, rapid method for analysing vitamin D in foods and supplements. He also developed the LC-MS method for melamine, subsequently adopted by the International Dairy Federation and published in the final ISO standard. In his current role at RSSL, he leads a team pioneering use of new instrumentation and techniques.

22 – 24 January 2014
Crystal Gateway Marriott, Arlington, VA, USA

Fifth Annual Food Quality, Safety & Analysis Symposium: PAT Applications in the Food Industry

There is an increased global focus on food quality, safety and analysis in the food industry and increased monitoring needs are emerging. The use of Process Analytical Technology (PAT) applications to monitor food quality and food safety is an important area that will be covered during this symposium, including new policies, technologies, case studies and a look to the future of the food industry.

This premier event is being held from 22 – 24 January 2014 in Arlington, Virginia at the Crystal Gateway Marriott, just minutes from Washington, D.C.

IFPAC has a history of providing a high quality programme with an extensive range of topics and expert speakers. This conference will be forward thinking with representatives from the FDA, CFSAN, USDA, NIST, ECBC, USP, industry leaders and academia from across the globe in attendance.

Food Symposium topics will include:

Emerging Technologies with Focus on Traceability and Contamination

Food safety incidents have reduced consumer confidence in the food industry and supply chain, prompting increased government intervention and regulation on food safety and food defence. Rapid analysis methods – particularly in the area of microbial and chemical threat agent detection – are being developed at an ever accelerating pace. This session will focus on emerging technologies in analytical / detection methods, rapid bio-monitoring and genetic fingerprinting in a response to recent contamination issues.

Food Safety

Food pathogens create an enormous social and economic burden on communities and health systems continue to be a source of concern for consumers, federal government and industry. The US Government has identified a number of previously unrecognised food vehicles as causes of foodborne disease, and the development of control strategies for pathogens and contaminants needs to address a wide range of foods. This session will focus on detection technologies for foodborne pathogens addressing sensitivity, specificity, speed of testing and use under a variety of conditions to ensure safe food.

PAT for the Dairy Industry

Application techniques, case studies and the benefits of implementing PAT for the dairy industry and in infant formula will be presented. Traditional dairy and pharmaceutical approaches and technologies will be discussed, also taking into consideration the nutritional arena.

PAT as a Quality Control Tool

This session will include discussions on process analytical technologies capable of providing quality control during food manufacturing. The business merits and strategies of PAT adoption will be discussed. The potential of PAT to support food regulation strategies will be addressed.

The Food Symposium also bridges with IFPAC® 2014, offering comprehensive tracks for the pharmaceutical, biotechnology and related industries; and the Onsite Analysis event with topics including field analytics, microbial detection, and the latest handheld instrumentation providing further opportunities to hear lessons learned that can be applied in the food industry. Please visit www.ifpacpat.org for details.

The poster session and social events will provide numerous opportunities for networking and informal meetings.

IFPAC provides a unique opportunity to interact with other industries that have been using PAT for nearly 60 years!

IFPAC® 2014 exhibition

A comprehensive exhibition showcasing of latest PAT instrumentation and services will be available.

It's an opportunity to see and be seen by old and new colleagues and customers. Those companies interested in exhibiting at IFPAC® 2014 are invited to contact conference management as soon as possible.

Please visit:
www.ifpacpat.org/food
for more detail

RECAPT: Retailer and Consumer Acceptance of Promising Novel Technologies and Collaborative Innovation Management

Novel food and drink products have traditionally been developed by the food industry, supported by input from food science. However, innovation is a task for all actors in the food chain, including retailers and caterers, who perform an important bridging function, identifying consumer demands and linking them to food producers and food scientists who possess the technical know-how for the development of innovative food products and services filling those consumer demands. Furthermore, retailers and caterers are central to the diffusion of new food products, services or technologies to consumers.

Building on this idea, the RECAPT project (running from 2011 to 2014) aims to strengthen collaboration around the management of innovations along the food supply chain, such that research findings can be effectively integrated into the development of innovative and sustainable products that meet consumer acceptance.

The core element of the RECAPT project is the Collaborative Food Innovation Forum (CFIF), a unique platform that brings together food scientists, the food industry, caterers and retailers. It allows the different actors to exchange information, facilitates trust building and promotes effective cooperation among all actors. The CFIF discusses issues related to promising novel food technologies, consumer acceptance of new products based on novel technologies, retailer and caterer adoption of new products as well as innovation management processes in the food chain.

Through this exchange, RECAPT will contribute to a higher degree of innovativeness across the food supply chain, thus contributing to the global competitiveness of the European food sector.

Promising novel food technologies

RECAPT has identified 15 novel food technologies (**Table 1**), which have either not yet been implemented in the food industry or that are implemented but have the potential for improved and/or extended application. Most importantly, we consider these technologies to have the potential to provide significant benefits for consumers, retailers and/or caterers. The 15 technologies can be divided into four categories: texturising methods, mild processing, electromagnetic methods and advanced packaging methods.

All technology candidates were evaluated regarding their contribution to processing, preserving and decontaminating food products[1]. Criteria included the status quo of the implementation level, benefits and shortcomings for consumers (e.g. 'fresh products', 'no additives', 'enhanced taste') but also for retailers and caterers (e.g. price margins, increased shelf life, extension of assortment or added value), and issues related to the adoption of such technologies (e.g. number / range of products a technology can be applied on, costs per treatment, expected market share and possible restrictions).

During the first CFIF workshop held in conjunction with SIAL 2012 in Paris, these novel technologies were introduced to an audience of food retailers and caterers. Each technology was discussed in detail, with regards to development status, level of current acceptance and indicators for future acceptance or reluctance by consumers.

Consumer acceptance of new technologies and products

In addition to selecting promising novel technologies, RECAPT focuses on the analysis of factors that determine consumer acceptance of such new technological processes. The three main areas cover acceptance of specific food and drink products to which such technology has been applied, the role that retail and catering play therein, and what strategic options emerge from this on the impact of the retail sector on (selective) adoption of food and drink innovations.

The work proceeded in three different steps. The first step was to identify emerging consumer wants and needs of relevance for technology acceptance[2]. Five emerging trends were identified that are particularly relevant to food technology development and acceptance.

Table 1: Overview of promising novel technologies

Texturising methods	**Mild processing**
• Hydrodynamic pressure technology (shock wave)	• High pressure processing (HPP)
• Ultrasonic cutting	• Infrared heating
• High pressure homogenisation	• Super critical fluid extraction (SCFX)
Electromagnetic methods	**Advanced packaging methods**
• Pulsed electric fields (PEF)	• Intelligent packaging
• Electron beam irradiation	• Radio Frequency Identification (RFID)
• Ohmic heating	• Edible coatings
• Cold plasma	• Active packaging
	• Biodegradable packaging film

These are: health, convenience, pleasure, sustainability and authenticity. For each of these trends, it was analysed 'what the trend will mean for actors in the food chain'.

Importantly, the report identified that these 'new trends' do not replace established food choice motivation such as 'good taste at a reasonable price', but rather they just add to the requirements of the demanding consumer that 'wants it all'. Next to this more holistic trend of 'want it all', a second holistic trend of 'relationship building' was identified, which acknowledged that technology plays an important relationship building role in terms of interactions with the consumer about products and services. During the discussions with the CFIF-Amsterdam meeting, a first outline of the guiding model was developed.

In a second step, the scientific literature on consumer acceptance of new food technologies was reviewed, and organised around the RECAPT model of Food Technology Acceptance[3]. The model identifies three paths through which technology attributes can influence end-user choice. The first path is the tangible product attributes modified by a technology, resulting in changes in the product quality as perceived by the end user. An example would be tenderising technologies that improve meat texture. The second path involves technology features that impact on retail and catering service attributes (an example would be mild processing technologies that increase shelf life, or allow uncooled presentation of products without impacting on taste). The third path involves associations with the technology in itself, resulting in socio-political technology attitudes that can create resistance against, or support in favour of the new technology as a whole. The model states that end-consumer acceptance of products produced with new technologies can be only be understood if all three routes (the consumer benefit road, the retail / caterer service road, and the technology attitudes road) are considered together and in interaction.

The model was then applied to four sets of technologies (mild processing technologies, electromagnetic methods, texturising technologies, and novel packaging and storage technologies) for which the scientific literature on consumer acceptance was reviewed, and organised around the product attributes, socio-political attributes and retail / catering value. The amount of consumer evidence differs considerably between the technologies, confirming that they are at different stages in their life cycle; or that the relation to end-users received different levels of attention.

Overall, the report finds support in the literature for the proposed model, but concludes that the three routes toward end-user acceptance have largely gone un-integrated in the existing literature. Future scientific research should focus more on the integration of and integration between the three routes of how technology features may affect end consumer acceptance.

This conclusion of course also holds for the more practical implications of the model and from it a checklist was derived allowing a novel product to be checked against the general knowledge in the field related to each of the three paths. The checklist focuses on the following three questions:

1. Does the technology change (experience or credence) product attributes?
2. Has the technology the potential to create strong socio-political technology attitudes:
   a. Has the technology perceived dread elements? (e.g. many affected people, chance of mortality, long term effects?)
   b. Is the technology perceived more technological than natural?
   c. Is the technology otherwise socially or politically sensitive?
3. Does the technology change the way products are presented/ delivered to the consumer in retail or catering?

In a third step, strategic options for CFIF were extracted for selected streams of food technologies identified in RECAPT's work package 3[4]. This was achieved by mapping on the RECAPT model the available scientific information on the four groups of technologies identified in WP3 as promising (electromagnetic methods, texturing methods, mild processing, and advanced packaging methods). These were further discussed in detail with the stakeholders present at the CFIF-meeting in Paris (October 2012). Together, this information has formed the basis for the formulation of strategic options for each of the technologies.

Although all four groups of technologies were perceived promising, the consensus was that advanced packaging methods and mild processing technologies are expected to show the greatest potential in terms of consumer acceptance. The overall conclusion was that for this potential to be realised in the market place, scientists and practitioners have to communicate any new technology and products based on that technology in a friendly and easy to understand way, to ensure that consumers can make an informed choice.

Where do we go from here?

In RECAPT, we are now exploring the decision-making processes of retailers and caterers for new products and technologies and conducting an investigation of current innovation models in the food industry, particular those focusing on open and collaborative innovation.

To learn more about the RECAPT project, visit the project website (www.recapt.org) or contact Dr Lars Esbjerg (lae@asb.dk), who is the Executive Project Director of RECAPT.

This project is supported by the European Commission under the Food, Agriculture and Fisheries, and Biotechnology theme of the 7th Framework Programme for Research and Technological Development.

CFIF Workshop

The next CFIF workshop will be held on 3 April 2014, at the Hotel Plaza Catalunia in Barcelona, Spain. Register now to join the discussion on how you can benefit from and organise collaborative innovation with suppliers or customers! Places are limited and travel expenses will be reimbursed. Contact Viviane Glanz (Glanz@asb.dk).

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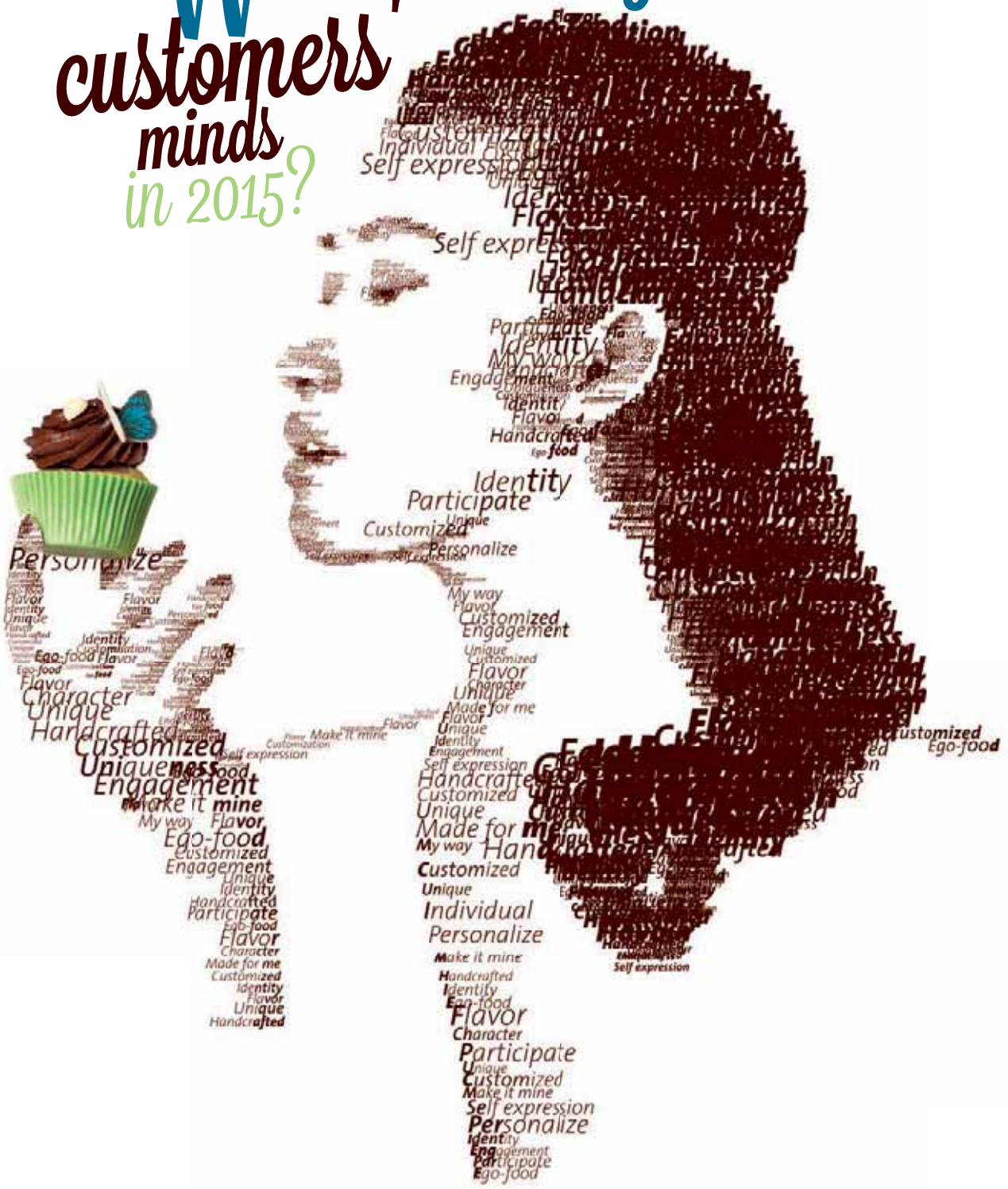
What's on your customers' minds in 2015?



Our lives are changing faster than ever. And what about consumer expectations? Discover what consumers really want tomorrow and how our range of cocoa, chocolate, fillings, decorations and nuts keeps you at the front of the customer's mind. Visit InsightOut, the second Barry Callebaut trend experience at ISM 2014.

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FOR FOOD MANUFACTURERS

The purest way to express ideas:
The Cocoart™ Collection

Making fine-quality cocoa and chocolate has always been something of an art form. But one small company in the west of Belgium – backed by the technological prowess of the world's largest chocolate manufacturer – has definitely taken things to the next level! They call it Cocoart™, we call it sheer genius. From the most intricate pattern or print to logos, text or even illustrations… Cocoart™ instantly transforms anything from chocolate to confectionery, bakery products and even ice cream into a genuine work of art! And while the idea of edible image transfers is not new, the Cocoart™ Collection's 100 per cent natural, E-number-free version absolutely is because it is miraculously made from 100 per cent cocoa powder using colour variations achieved via completely natural means. This is, by no stretch of the imagination, a revolution in decoration. One that is nevertheless sure to stretch the imagination of many a chocolatier, baker and pastry chef!

Broad pallet

Such a huge range of designs: spanning the spectrum between classic and contemporary, trendy and timeless, customised and standardised. And a surprisingly broad pallet of colours: from brown, black, purple and even red!

The Cocoart™ Collection is the brainchild of IBC, a subsidiary of Barry Callebaut based in Kortrijk, Belgium, using Bensdorp® cocoa powders, another Barry Callebaut brand representing over 170 years of expertise in cocoa. That combined expertise gives rise to a wide variety of colour intensities, which means that the possibilities for adding a touch of originality and luxury to your products are limited only by your imagination!

Natural ingredients

The real innovation behind the Cocoart™ Collection is of course the fact that it is made from 100 per cent natural ingredients. No artificial food dyes! No e-numbers! Just 100 per cent natural cocoa powder made from 100 per cent natural cocoa beans, grown under the equatorial sun. It's enough to inspire the artist in anyone! And with consumer demand on the rise for authentic, natural foods, the Cocoart™ Collection caters to a growing number of people for whom artificial additives are a major turn-off!

But how can you create such a broad pallet of colours from just one plant? The secret lies in meticulously controlling a number of different parameters: such as selecting the right variety of cocoa to begin with or altering the temperature and duration of roasting. The colour palette ranges from brown to black and from purple to deep unique red, depending on the mixture of Bensdorp® cocoa powders that have been obtained by patented cocoa processing (patent number WO2009/093030). Revolutionary element in the collection is the world's first natural, e-number free red colour, which places the Cocoart™ Collection in a unique position in the market.

Indeed, Cocoart™ offers manufacturers an endless array of possibilities for personalising or branding their products, producing seasonal variants or even inspiring entirely new products and applications. For retailers, it opens up avenues for a more innovative chocolate and confectionary line-up and increased revenues via a broader premium segment. And last but certainly not least, it answers the growing consumer demand for foods that match their style and individuality without adding to the list of ingredients. Indeed, as consumers search for more natural and less processed foods, the Cocoart™ Collection offers a unique alternative, one that virtually comes with its own ready-made marketing story.

Has your artistic side been awakened? Cocoart™ has already captured the imagination of several chefs and chocolatiers and has been nominated for the category: Confectionery Innovation of the year at the Excellence Award at Food Ingredients Europe 2013. And as seasoned art critics with something of a sweet tooth, we can but wholeheartedly support this truly artful invention! Bon Appétit and above all, Vive l'Art!

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K. Niranjan University of Reading

Creating food structure out of thin air!

Bubbles are always perceived to represent the best in food and drink. Their presence and characteristics have dominated our perception of the quality of traditional products such as bread, champagne, ice creams and let's not forget the good olde beers! In recent years, there has been a constant flow of new bubble-containing snack foods into our supermarkets; whipped cream, chocolate, wafers, cakes, meringues, extruded snacks and sparkling drinks, all of which have very novel structures and mouth-feels (Figure 1). Some products are perceived to offer lighter alternatives in terms of calories. Most products manage to gain a positive market image by highlighting bubbles, because bubbles tend to be associated with celebration and luxury.

Incorporating bubbles can be challenging in any food system, simply because gas is three orders of magnitude less dense than the food matrix into which it is added, and inevitably, the dispersion is unstable. It is even more challenging to create the right bubble-containing structure that will give the product the mouth-feel that its consumer desires. In addition to the food matrix, a bubble containing structure is typically characterised firstly by the fractional volume of the product occupied by bubbles and secondly, the bubble size distribution. Thus, ingredient formulation as well as the processing technique employed have to work in tandem in order to result in a product that is desirable and stable over its shelf life. In terms of product formulation, it is imperative to have agents in the ingredient mix which exhibit what is commonly known as gas-liquid interfacial activity. Such agents normally hold the bubbles within the matrix and stabilise the dispersion by lowering interfacial energy. In practice, however, a number of components in a product formulation will possess interfacial activity, and it can be extremely complex to analyse which component or components have succeeded in migrating to the interface and in what proportions.

Figure 1: A selection of commercially available bubble containing products

Techniques such as confocal laser scanning microscopy are only just being applied to unravel such phenomena. The technology employed to incorporate bubbles into food systems is also equally important, if not more. A variety of methods are used commercially, but the most common method involves admitting the food system at a temperature

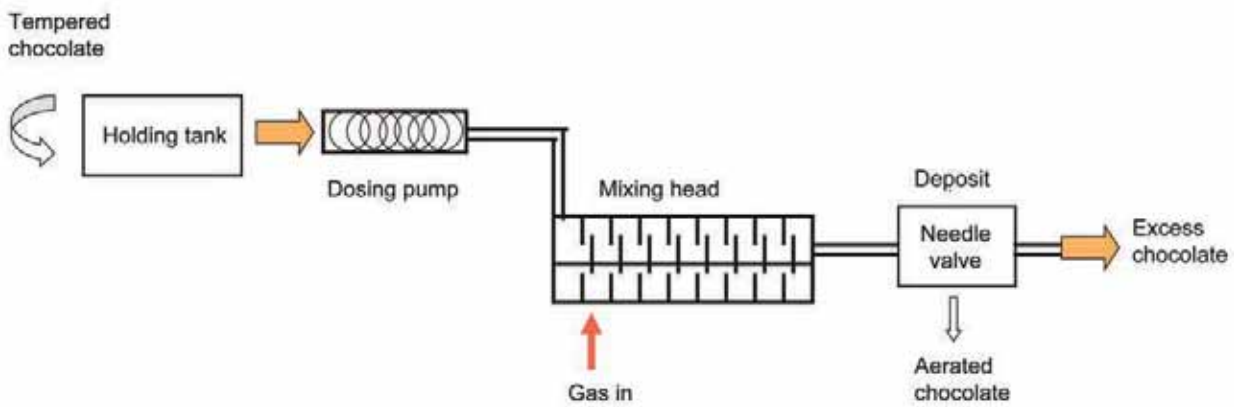


Figure 2: Flow diagram of a typical process used to incorporate gas into liquid chocolate (Taken from: Bubble-Included Chocolate: Relating Structure with Sensory Response J. Hadelt, S.T.Beckett, and K. Niranjan, Journal of Food Science, Vol. 72, Nr. 3 (2007) doi: 10.1111/j.1750-3841.2007.00313)

where it is predominantly in the fluid state, together with a gas (not necessarily air), into a mixing head maintained under a positive pressure and driven by an impeller. The dispersion formed is then drawn out and restored to atmospheric pressure, with the temperature adjusted to a value that is needed for further processing of the dispersion (Figure 2).

The mechanisms responsible for the formation of bubble-containing structures is complex, but worthy of some discussion here. Intuitively, engineering science suggests that the rapid impeller rotation (normally in the orders of magnitudes of hundreds to thousands of revolutions per minute) creates high levels of turbulence in the fluid inside the mixing head, which breaks and disperses the gas phase; the interfacial energy, on the other hand, counteracts the forces of turbulence, and a balance between turbulent dissipation and interfacial energies determines the resulting bubble size distribution. Although this mechanism has been proven to be valid in the case of chemical and biochemical reactor operation, recent research has shown that it may not be the sole, nor indeed the dominant mechanism responsible for the formation of bubble-containing structures in food systems such as chocolates or foamed creams used in cream biscuits. If the balance between hydrodynamic and interfacial energies determined bubble size distribution, then the chemical nature of the gas phase should have been immaterial, and regardless of whether air or carbon dioxide or any other gas for that matter were employed, the dispersion structure should have stayed the same in a given system for a given set of mixing conditions. This is clearly not the case, as is well known in the food industry. The chemical nature of the gas plays a critical role in characterising the dispersion, and the gas phase employed, indeed defines the structure, texture, and eventually, the mouth-feel of the product – a fact that was not hitherto appreciated.

How does the chemical nature of the gas act to characterise the dispersion? It is believed that the gas acts through its solubility in the food system, which, in turn, depends on the pressure employed in the mixing head. Contrary to the hypothesis that the gas is merely broken down by the impeller into bubbles, it is now clear that the gas, to a very large extent, dissolves in the fluid phase inside the mixing head. When the pressure is subsequently released downstream, gas desorption occurs to form the bubbles. Thus the number of bubbles and the bubble size distribution depends on the level of supersaturation generated by pressure release.

Until recently, the role played by the physico-chemical nature of a gas in forming chocolate foams was not fully appreciated. It was thought that similar foams would result if different gases were used in a process, under otherwise identical conditions. In a study published in 2007, bubble-containing chocolates were made by sparging four different gases – CO_2, N_2O, He and N_2 – separately into a given base recipe under otherwise identical operating conditions. When the properties of the product formed were investigated, it was clear that the more soluble gases in chocolate, i.e. CO_2 and N_2O, gave a foam structure that contained greater fraction of bubbles which were also larger in size. In other words, these gases gave 'macro-aerated' chocolates. On the other hand, the gases with relatively lower solubility – He and N_2 – gave micro aerated chocolates; these products contained fewer bubbles which were also smaller.

2D Image analysis of X-ray images

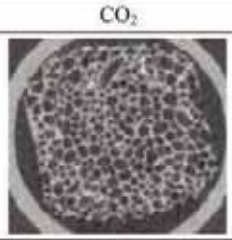
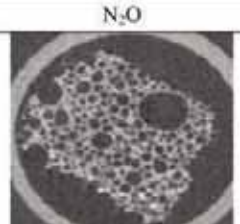
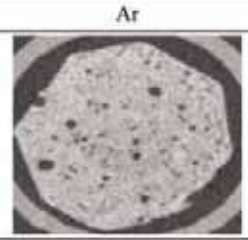
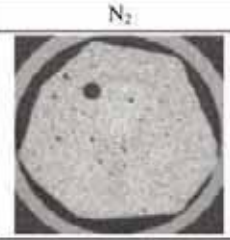
Chocolate type	CO_2	N_2O	Ar	N_2
2D reconstructed X-ray image				
d_{mean} (mm)	0.51 +/- 0.32	0.41 +/- 0.41	0.19 +/- 0.18	0.13 +/- 0.13
No. of bubbles considered	227 +/- 3	222 +/- 27	234 +/- 5	218 +/- 8

Figure 3: Chocolate structures formed by the different gases
(Taken from: Bubble-Included Chocolate: Relating Structure with Sensory Response, J. Hadelt, S.T.Beckett, and K. Niranjan, (2007) Journal of Food Science, Vol. 72, Nr. 3)

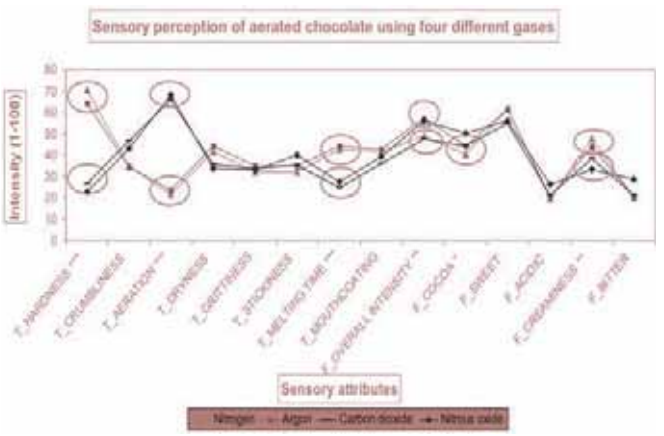


Figure 4: Sensory panel response (Taken from: Bubble-Included Chocolate: Relating Structure with Sensory Response, J. Hadelt, S.T.Beckett, and K. Niranjan, Journal of Food Science, Vol. 72, Nr. 3 (2007); Differences being tested at: * 5% significance level; ** 1% significance level; *** 0.1% significance level)

Figure 3 (page 46) summarises the chocolate structures formed by the different gases, with the images shown being generated by a technique called x-ray tomography which is widely used in hospitals for imaging tumours. This technique relies on the difference between the extents of x-ray attenuation caused by the gas and chocolate phases to produce the necessary contrast in an image, which can subsequently be analysed using image analysis software to deduce information such as bubble size distribution. It may be noted that the image produced is essentially two-dimensional, and a 3-D replica of the bubble containing chocolate can only be obtained by scanning a number of parallel layered 2-D sections and stacking the images together using standard software.

Figure 3 (page 46) clearly shows that the structures of the chocolate foams formed from the same base recipe depend on the gas used. The response of a sensory panel presented with samples made from the four gases is shown in **Figure 4**. It is clear that the products were perceived to be significantly different in terms of hardness, aeration, and melting time. They were also found to differ in terms of overall flavour intensity, cocoa flavour, and creaminess. No significant differences were perceived between the other attributes. This does not imply that products are the same with respect to such attributes; it only means that a difference cannot be detected for the attributes concerned.

It is therefore evident that different bubble-containing chocolates can be produced from the same base recipe by simply changing the gas used. It is necessary to note that the gas used to form the structure is not trapped inside the chocolate. By the time the product is consumed, the gas has essentially escaped and the voids are replaced with air. Therefore the gas used in the process is essentially a structure developing aid, and does not directly come into contact with the consumer. In other words, if helium, for instance, is used to make the chocolate and a consumer bites into the product, the chance of the consumer's voice turning squeaky is fairly remote.

The use of a gas within a recipe, thus, gives the opportunity to create novel food structures and mouth-feels. The use of different gases in the same recipe also enables the creation of perceptibly distinguishable products. Bubble-containing products, in general, possess a favorable ratio of volume to the amount of material and involve the use of food approved gases which do not interact directly with human health. The use of such gases can enable manufacturers to confer upon the products novelty but not calories.

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Alexis Freier Research & Development and Technical Services Manager, Dakota Growers Pasta Company

Pasta production

The quality of a pasta product is evaluated by dry appearance and cooked texture. 'Good Pasta' is defined as having uniform amber colour with an absence of black, brown or white spots, a smooth surface free of streaks or cracks, and a texture that when cooked is neither chewy nor mushy but 'al dente'. Three key factors determine success or failure in pasta production: raw materials, processing technology and the presence of skilled employees throughout the manufacturing process.

Raw materials

There are six classes of wheat grown in the United States: Hard Red Spring, Hard Red Winter, Soft Red Winter, Durum, Hard White and Soft White. Each class has its own characteristics and recommended use. For example, Soft Red Winter Wheat is associated with low protein content, low water absorption, and is recommended for cakes and pastries[7].

Durum is the only wheat that has all the necessary qualities required to make pasta[5]. Durum wheat produces kernels much harder than all other wheat. Durum is also the only wheat that has yellow pigments distributed throughout the entire endosperm rather than only the outer layers of the kernel[1]. This means that milled durum produces yellow, granular semolina while all other wheat produce white, powdery flour. The gluten (wheat protein) found in durum is much more pliable than gluten in other wheat which leads to easier extrusion. Durum also has lower water absorption, an important factor in the pasta drying process. Pasta products made from durum hold their shape better and have a firmer texture when cooked.

It is important to remember that not all durum is created equal. Vitreousness is a key element of durum evaluation and grading. Vitreous kernels have a translucent or glass like appearance with a flinty texture. Starchy kernels are the opposite of vitreous; yellow, soft and possessing a crumbly texture. As the amount of vitreous kernels decrease, semolina extraction decreases, flour production increases and finished pasta quality decreases leading to inferior products. In the United States, durum is categorised into three subclasses: Hard Amber Durum (75 per cent or more vitreous kernels), Amber Durum (60 - 74 per cent vitreous kernels), and Durum (less than 60 per cent kernels). The best quality pasta is sourced from Hard Amber Durum.

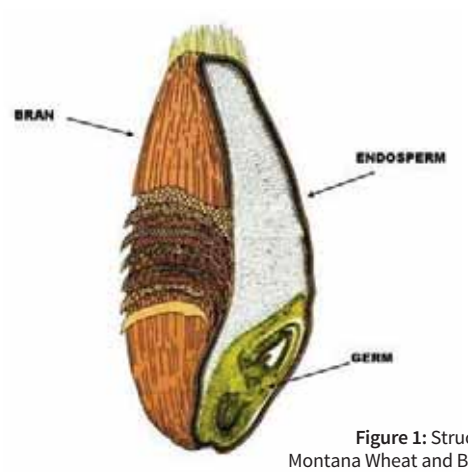

Figure 1: Structure of a wheat kernel. Montana Wheat and Barley Committee. 2012. http://wbc.agr.mt.gov/wbc/Consumer/Diagram_kernel

Kernel structure and milling

A kernel of durum wheat has three components (**Figure 1**). The bran, or outer layers, constitutes 12 – 15 per cent of the kernel and is high in fibre and ash. The germ is the embryo of the plant. This is three to five per cent of the kernel and contains most of the lipids and other nutrients needed to uphold germination. The endosperm is 80 - 85 per cent of the kernel, and contains high amounts of starch and gluten.

Milling is the reduction of wheat kernels to smaller particles that can be made into more palatable products1. The finished product of durum milling is semolina. This is made by removing the bran and germ, then breaking the endosperm into coarse, granular pieces. The efficiency of a durum mill is measured through maximum semolina extraction,

minimum flour production and the quality of semolina produced. An ideal semolina product will have uniform particle size or granulation, minimum starch damage, and show consistency between shipments.

The pasta production process

Large scale pasta production began as a manual, discontinuous batch process where production was very labour intense and somewhat disjointed. Modern industrial pasta production is a continuous, automated process. This is advantageous because it creates pasta that is consistent in quality, uses equipment with lower space and labour requirements, and permits a more hygienic processing environment[2]. Pasta production is composed of three unit operations or steps: mixing, extrusion and drying. We will first cover traditional mixing and extrusion, then follow with a new technology - the Polymatik system.

Mixing

The purpose of the mixing step is even distribution of water throughout the semolina, promoting equal hydration of all particles[4]. The mixing operation is composed of three stages: the pre-mixer (or high speed mixer), the main mixer and the vacuum mixer. The entire mixing process ranges from 10 - 20 minutes depending on the size and model of the line being used.

To begin the process, calculated amounts of semolina and water are fed into the ingredient doser (or feed). The ingredients flow into the high speed or pre-mixer where small blades quickly rotate to promote rapid and even hydration. The flour and water mixture then flows into the main mixer where dry spots are eliminated and dough balls are formed through the use of large paddles. The dough balls then flow into the vacuum mixer where air is extracted out of the mix prior to extrusion.

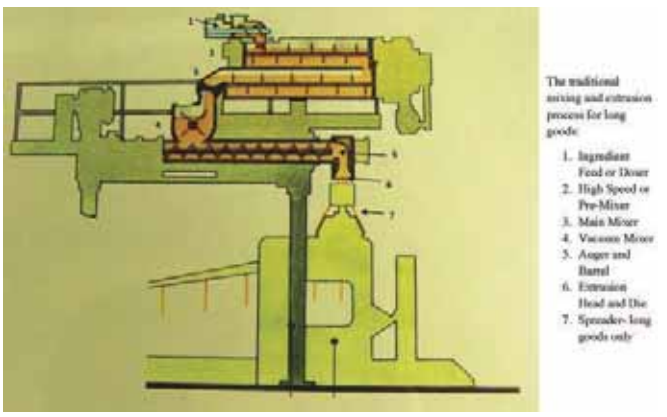

Figure 2: Traditional mixing and extrusion diagram. Courtesy of Braibanti

Air in the mix is unfavourable for two reasons. As the dough is extruded air is dissolved in the dough and after exiting the die, small bubbles are formed in the product[3]. This defect is known as vacuum burn and causes the product to be opaque and spotty, rather than the clear yellow appearance desired by consumers. The second reason for air removal is the lipoxygenase enzyme. In the presence of oxygen, the enzyme will interact with free fatty acids in semolina and bleach the semolina's yellow caretonoid pigments[3]. This causes poor colour in finished product.

The moisture content of the dough after mixing is 29 - 32 per cent. Proper moisture content is determined by the size of dough balls entering the vacuum mixer. Ideal moisture content makes balls about

one inch (2.54 centimetres) in diameter[4]. If the dough is too wet, large balls or a bulky mass will form, causing product defects in the drying process. In extreme cases, a wet mix will plug up the vacuum mixer causing the line to go down. If the dough is too dry, there will be very small balls and dry pieces will plug up the die causing product defects and insert damage. Excessively dry mixes will jam up the extrusion auger forcing the line to go down.

Extrusion

Extrusion is often called the heart of the pasta-making process. In this step, the balls of dough are kneaded and shaped into a pasta product using an auger, extruder barrel, press head and dies. The extruder barrel is jacketed and cooled with water to counteract unwanted heat produced during the extrusion process[3].

The dough balls formed during the mixing process flow from the vacuum mixer to the extrusion auger where the dough is kneaded and formed into a continuous, homogenous mass. While the dough is kneaded it is pushed through the extrusion barrel toward the press head. The press head receives the dough from the extruder and uniformly distributes it on and through the die[6].

The production of long and short pasta products requires different equipment; therefore a line is built either to be 'long' or 'short'. To produce long goods, a special straight head is installed on the press. This head is designed to hold rectangular dies for the extrusion of long pasta shapes arranged in a straight line and divided into one or more parallel curtains[2]. An automatic spreader cuts and drapes these curtains onto special metal poles[2]. To produce short goods, a round head and dies are used. This process uses a continuous rotating knife at set speed[2].

The pasta die is one of the most important components of the manufacturing process because it gives the pasta its shape. Dies are composed of a die block and inserts. The block is made of bronze and holds the inserts. Inserts may be brass or Teflon and shape the pasta product. Teflon is the most popular insert material because it makes pasta that is smoother, more yellow in colour and helps reduce friction during extrusion, increasing line capacity. Brass inserts produce a chalkier product and create more dust in the factory setting.

Unlike snack foods or cereal processing, very little expansion (puffing) is noted as the product exits the die. This is due to dough temperature maintained at less than 45°C and a relatively low moisture content[3]. This temperature also prevents damage to the gluten network and maintains final product integrity and cook quality.

Traditional versus Polymatik systems

The above mixing and extrusion scenario is the common, traditional procedure used in factories today. A new technology available to pasta makers is the Buhler Polymatik system (**Figure 3**). Product is fed into auger, where it is mixed and kneaded using a twin screw under vacuum, then extruded. Advantages of this system include the entire process is under vacuum and it is entirely enclosed.

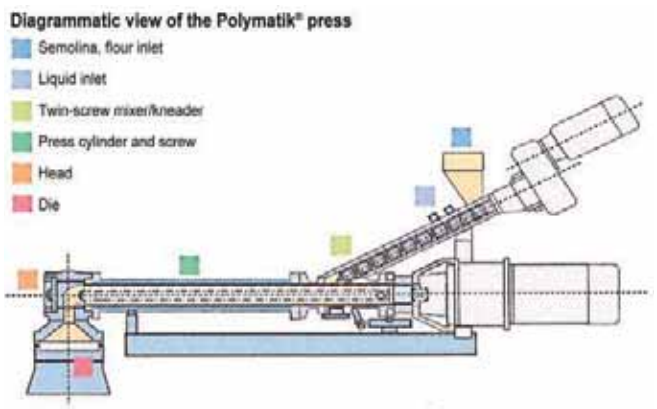

Figure 3: Polymatik mixing and extrusion system. Courtesy of Buhler Group

Figure 4: Long good drying diagram. Courtesy of Braibanti

Drying

After the dough is extruded through the die, moisture is approximately 28 - 32 per cent. The drying step will lower product moisture to about 11 - 12.5 per cent; finished product moisture should never exceed 13 per cent. The goal of every drying operation is to prevent checking the formation of numerous hairline cracks in the product that make it appear opaque and decrease its strength[3]. This is achieved through several drying steps or zones at different temperatures and humidity.

Humidity in the dryer is a result of moisture leaving the product and evaporating into the air of the dryer. This causes the inside air to become saturated with water molecules so air inside the dryer is exchanged with outside air at a slow rate. Air exchange is controlled using adjustable inlet and outlet doors, or dampers, found on the roof of the dryer. If moisture inside the dryer becomes too high, the dampers are opened. If it becomes too low, the dampers are closed. Outside air is either pulled from the outside or pushed from the inside by the use of a blower fan. The fan can force air in and out of the dryer with the opening and closing of damper doors.

Heat inside the dryer is produced through heating coils positioned throughout the dryer to contact the circulating air from fans. The coils are typically connected to a hot water source which is circulated through the coils to provide the heat to dry the pasta. The hot water in the coils is controlled by opening and closing valves.

The initial pre-drying step is given to the pasta immediately after extrusion to maintain product structure. For long goods this is a blast of hot air immediately after the spreading and cutter step. For short goods a ventilation group is used as pasta leaves the die followed by shaking step to prevent sticking during conveying[2]. Long goods travel through each phase of drying on poles (**Figure 4**). Short goods use a series of conveyor belts to move through each dryer (**Figure 5**).

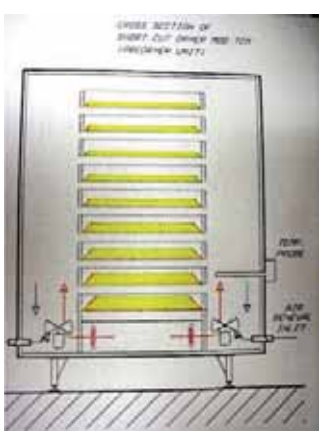

Figure 5: Cross-section of short good drying diagram. Courtesy of Braibanti

Pre-drying is the most sensitive step of the drying process. The pre-dryer utilises rapid temperature increases, air ventilation, and controlled humidity to quickly remove moisture from the product. Stabilisation stages or zones are alternated with rapid drying to prevent product burning and other defects. After the pre-drying step product moisture ranges from 15 – 20 per cent, depending on the shape.

When product enters the final dryer it has a firm structure and no capillarity[4]. This step focuses on lowering product moisture to ~12.5 per cent and balancing residual water content on the inside and outside of the pasta shape[2]. This is achieved by using longer rest or stabilisation periods to lower the product temperature gradually to avoid stressing the pasta.

After the final dryer the pasta passes through a cold air treatment, or cooler, to stabilise the product prior to packaging[4].

Drying time depends on the product shape, temperature, and equipment model being used. In general, short goods take four to six hours and long goods take 8 - 12 hours.

New technology has allowed for the use of higher drying temperatures. There is not sufficient room in this article to cover the extent of gains found by using high temperature drying. A summary includes: reduced drying time, reduced microorganism levels in finished product, improved pasta quality due to better holding qualities during cooking and improved colour of dried product[2].

Conclusion

Pasta is processed using the steps of mixing, extrusion, and drying. Quality pasta production is achieved through the use of proper raw materials and processing technology.

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About the Author

Alexis Freier is the Research & Development and Technical Services Manager for Dakota Growers Pasta Company and is based in New Hope, MN. Her key responsibilities include creation, development and implementation of new products relating to pasta operations, enhancement of quality and operational execution of existing products, and providing technical support for customers and the sales, marketing, and operations groups.

Alexis has been a guest speaker at several Northern Crops Institute short courses and covered topics such as pasta die maintenance and management and pasta quality control. She represents Dakota Growers on the National Pasta Association Technical Affairs Committee and is a member of the Institute of Food Technologists.

Alexis is a graduate student at Kansas State University, Manhattan, KS pursuing a Master of Science in Food Science. She received her Bachelor of Science in Food Science from North Dakota State University.

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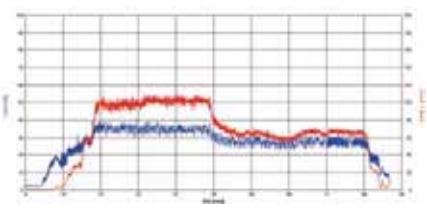

Calculation of costs / benefits for the investment in a LABORATORY EXTRUDER

Calculation basis:	*Production extruder*
Raw material costs (with 4 test series)	approx. – € 8,000.00
Costs for interruption of production	approx. – € 24,000.00
Restarting of production	approx. – € 2,000.00
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Gary Tucker and Graham Duckworth
Baking and Cereal Processing Department, Campden BRI

Temperature and humidity effects on baking

The production of baked goods such as bread, cake, biscuits and pastry requires a number of processing steps between which there exists significant interaction. This article considers the influence of temperature and humidity control in industrial or plant bakeries and how this can impact on product quality. The stages of mixing, forming, proving, baking and cooling are considered. Bread is most sensitive to changes in temperature and humidity and so this article discusses these effects on bread but makes reference to other baked products as required.

There are numerous methods of making bread, ranging from the highly labour intensive craft techniques using sourdough cultures, sponges and brews, to the high volume industrial bread processes which are more time and cost efficient. Despite the variety of methods, most use common processing steps comprising mixing, resting, moulding, proving, baking and cooling. Each of these requires considerable skill levels on behalf of the craft baker or high levels of automation and process control for the plant baker. Both craft and plant bakers intend on producing bread with a specific structure and both take immense care in their processes to ensure that the product meets the needs of their consumers. This requires an understanding of each stage and how it is influenced by external factors in the bakery such as temperature and humidity.

Mixing

The process of mixing is a critical element of any bread making process. For no-time dough, such as that typically produced in plant bakeries, the mixing element is wholly responsible for delivering dough with the required structure and rheological properties for further processing. It is often stated that the dough is at its optimum quality immediately after mixing and that further processing can only serve to damage the delicate gas cell structure. This means that the dough must be optimal to minimise damage during transfer and moulding.

A rise in dough temperature during mixing is a natural consequence of the work expended in shearing and deforming the dough within the mixing chamber. The final dough temperature should be controlled because it has a direct effect both on how the remaining parts of the process (such as proving) proceed, as well as how the dough will transfer and mould. Warm ambient conditions, as found during the summer in the northern hemisphere, will result in increased ingredient temperatures and a higher dough temperature from the mixer. This also happens with bakeries that have poorly insulated ovens that warm the air around the ingredients and mixing area. Solutions are to cool the dough by using ice or a glycol cooling jacket, or to minimise this effect by controlling the air temperature around the mixer. If dough gets too warm it will become soft and sticky, and this will lead to quality problems

as well as increased waste from contact with surfaces during the subsequent processing stages.

Dough transfer, resting and moulding

After mixing, it is common to divide bread dough into unit pieces and let it rest for a few minutes. This allows the gluten network to relax so that moulding can take place with fewer problems but it also can be used to change the internal structure of the baked bread. Longer resting periods at ambient bakery temperature tend to open up the structure, a benefit that is used in producing French baguettes. This resting period takes place in the bakery environment and so is not temperature or humidity controlled.

High dough temperature can result in softer dough that flows more easily and with a stickier surface. This can lead to a greater chance of the dough sticking to equipment surfaces. High bakery temperatures are more prevalent during summer months when ambient temperatures increase, giving rise to more instances of soft and sticky dough. Sheeting rollers of a moulder are the most likely equipment to collect sticky dough because of the shear forces and high surface area in contact with dough. This leads to line stoppages, product waste and costly clean down.

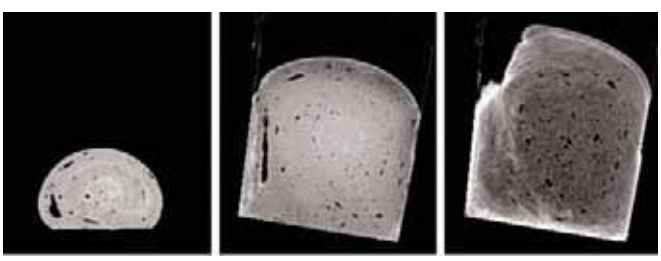

Figure 1: X-ray image of bread dough during proving and baking, showing dough at the start and end of proving and at the end of baking

If dough is too cold it can also experience damage during moulding and this tends to show itself as elongated holes where the sheeted dough has not joined together properly. A cold dough will be tight and will not prove up as effectively as one that is warmer and softer. This will result in lower dough heights after proving and force too much oven spring during the baking process. Excessive oven spring is seen as large tears on the sides of a tinned loaf and is undesirable. The ideal situation is to have a controlled amount of oven spring, which requires that the dough proves to the correct height.

Biscuit and cake manufacturing do not tend to rely on moulding or transfer systems in which the dough makes contact with fresh equipment surfaces. Biscuit dough is transferred as separate units on conveyors to the oven and cooler, whereas cake batter is deposited into tins that are moved without the batter contacting other surfaces. This makes the influence of surface softness and stickiness a different one to that with bread.

A small number of modern bakeries are now designed with air conditioning as a means to control temperature and humidity. Traditional alternatives to this are to use flour dusting of contact surfaces or air jets targeted on dough surfaces to create a skin on the dough. Neither is ideal because they dry out the dough surfaces and this makes it more difficult for the dough to increase in volume during proving. However, they are less expensive than air conditioning.

Proving

Proving is a time-consuming process step during the bread making process. The objective in proving dough is to generate carbon dioxide from yeast activity and in doing so increase the dough volume. Typically, 90 per cent of bread volume is achieved in the prover with a further 10 per cent from oven spring. There are two basic requirements of the proving operation, which are to operate at dough temperatures in which the yeast is very active (34-36°C) and to utilise high humidity (70-80%RH) levels so a skin does not develop on the dough. Most bread provers are set at 38-42°C because of the need for higher temperatures than the dough to gain benefit from a temperature driving force that maximises heat transfer into the dough. Without humidity control the dough surfaces will lose moisture and develop a skin that restricts expansion of the dough. Bread dough typically has a natural relative humidity above 95%RH and so during proving the dough pieces will lose a small amount of water to the environment. This is of the order of two to three grams in a 900 gram dough piece.

Optimising proving is effective providing that interactions between mixing, proving and baking are understood. During mixing, it is necessary to create a network of small gas cells in the dough so the yeast cells can utilise oxygen from the cells and inflate each of the bubbles. This gives rise to a fine bread crumb structure and requires the use of minor ingredients such as enzymes, emulsifiers and oxidising agents. One of the innovative ways used by Campden BRI to investigate proving (and baking) is with X-ray imaging. Figure 1 shows static images taken from a movie clip of proving and baking for a bread dough piece. It shows clearly the gas cell structure and how this changes over the proof time.

Some baked products that use bacterial and yeast starter cultures, such as sour dough bread and croissants, prove at lower temperatures than conventional bread. The aim here is to encourage a different group of microorganisms to grow in addition to the yeast. Typical proving temperatures can be around 25°C and can be for much longer than the 45-55 minutes for 800 gram tin bread. Bacterial groups such as *Lactobacilli* and *Acetobacter* will favour growth at a lower temperature and generate metabolites that confer benefits to be product in terms of flavour, texture and preservation. This is where the craft sector and plant bakeries overlap in their use of mixed microflora.

External ambient conditions can exert an influence in controlling the prover temperature and humidity. Independent control of temperature

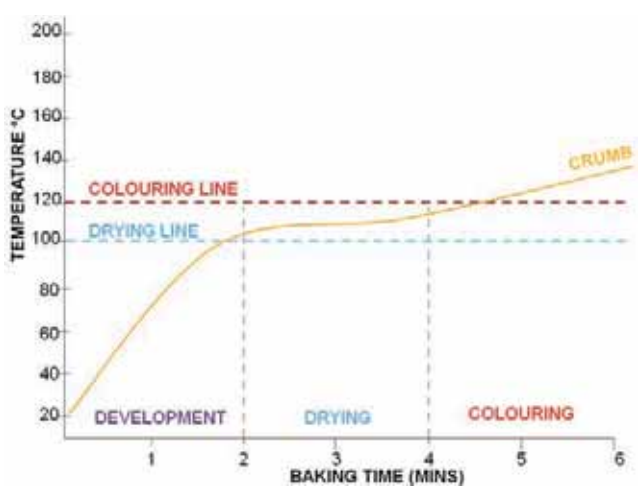

Figure 2: Core temperature rise during biscuit baking showing the three oven stages

and humidity is not always possible, particularly with provers that use steam to generate the humid environment. Warm and humid summers are often an issue in bakeries and can cause prover settings to go out of control. This can be a problem for products that are proved at low RH such as English muffins and other products with very wet dough.

Baking and cooling

Baking follows proving and is by far the most energy intensive process step in the manufacture of baked goods, and therefore the most costly. Bakery ovens can be either directly fired by gas burners or indirectly heated by gas or electricity. Radiation, convection and conduction are all used in bakery ovens, with any one type of oven using at least two of these heating modes. Typical oven temperatures for bread are between 200 and 270°C with baking times of 20-25 minutes for 800 gram loaves. For cakes, the range of temperatures and times is greater because of the size differences with products and the high levels of sugar that can generate browning at faster rates than with bread. Oven temperatures as low as 150°C are used for large cakes but with longer baking times of two to three hours. For biscuits it is usual to set ovens at 250-300°C and bake for five to ten minutes.

Analysis of the changes that take place during biscuit baking are useful for helping understand the requirements of the baking process for any baked product. **Figure 2** (page 55) plots the temperature change measured at the core of a biscuit during baking and shows the three stages of baking, which are structure development, drying and colour formation. Each has its own requirements for the oven settings. In **Figure 2** (page 55), they are shown to be approximately equal in time but this will differ for products of varying size and heating rate.

Development stage: During this stage, the dough changes from its raw state into an aerated structure in which the proteins and starches have started their chemical and physical transformations. Raising agents (or yeast as with bread) release carbon dioxide, which, together with gases released from solution, thermal expansion and steam, will create lift in the dough. High humidity is best suited to this stage so that the surface remains soft and can accommodate the rise without cracking. Ovens with dampers are usually operated with the dampers fully shut during the development stage to provide a build up in humidity caused by water vapour from the biscuit dough and from combustion products with direct fired ovens. Humidity during the first stage of baking is also important for bread and cake to avoid surface cracking and resistance to the rise.

Drying stage: This is more specific to biscuit baking because bread and cake require water to be retained within the structure. Biscuit dough temperature will reach 100°C at the start of this stage. Water is driven off at a rapid rate and it is important that the oven can remove the water vapour from the biscuit surface. This allows more water to be evaporated and so keeps the drying rate high. Much of the volume increase has already been achieved in the development stage and the surface can now be dried without risk of expansion cracks. High oven temperature, fully open dampers to reduce humidity and high rates of air turbulence are all ideal to remove water.

Colour formation stage: This is applicable to all baked products. There are two main colour reactions that give rise to the desirable brown crust and paler crumb colour. Maillard browning takes place above approximately 105°C and requires the presence of a reducing sugar (e.g. glucose, maltose or lactose) together with an amino acid, the type of which determines the colour and flavour. The second colour reaction is caramelisation of the sugars at 160°C and higher. A lower oven temperature (than with the drying stage) is desirable to control the biscuit surface temperature so it does not end up with a blackened surface. Cake baking is particularly sensitive to colour formation because of the high sugar concentration. It is common to reduce oven temperatures but to bake for longer to avoid excessive darkening of the surface.

The X-ray technique can also be used for improving our understanding of cake baking. **Figure 3** shows a fruit cake batter before baking, after 30 minutes of baking, and at the end of a 60 minute bake. It can be seen in **Figure 3** that low humidity during baking has caused the cake surface to crack and may also have restricted the rise. It is also apparent that the fruit, highlighted by high density white areas, has gradually sunk towards the bottom of the cake.

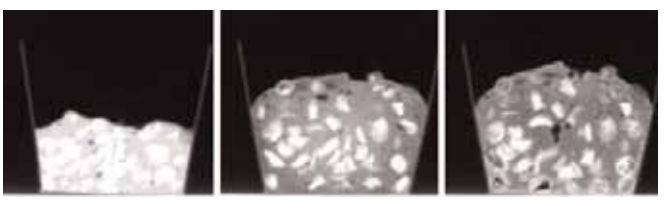

Figure 3: X-ray images of a fruit cake batter, after 30 minutes baking and 60 minutes baking

Cooling

Cooling of baked goods can be a difficult process to implement and one in which the risks of post-bake contamination are high. Humidity levels and air temperature are usually ideal for microbial growth in the cooling chamber because of the water released from hot bread. Attention to cooler hygiene is critical in order to minimise the number of mould spores in the air. The high residence times of the products within these environments provide plenty of opportunity for airborne micro-organisms such as mould spores to settle on the product surfaces.

Typical cooling times for 800 gram loaves of bread are around 90 – 120 minutes if ambient air is used. Cooling times can be reduced through the use of refrigerated air towards the end of the cooling process, typically taking cooling times down to 60 – 90 minutes. However, the introduction of a refrigeration system will result in areas of condensation and potential for mould to grow.

A reduction in cooling time can also be made using saturated air because of the increase in cooling rate that can be achieved with increased humidity. Saturated air also has the advantage that water loss is reduced, leading to higher bread weight or the possibility to reduce dough scaling weight. High humidity can be achieved by blowing the air through a water curtain.

Significance

Temperature and humidity control during the manufacture of baked goods is important with most of the processing steps, from mixing to cooling. Dough is very sensitive to changes in both factors and it is necessary to understand how changes can affect the baked product quality. Campden BRI has researched their influence on final product quality and process efficiency. Some examples are given in this article.

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Comparison of applicable technologies to secure safe water supplies

In the past few years, it has become evident that there are topics that deserve attention, but do not easily fit into existing publications, such as regular scientific journals and scientific book series. The European Federation of Food Science and Technology (EFFoST) therefore decided to initiate a new series of publications, 'EFFoST Critical Reviews'. It is meant to fill the gap between the journals and the books. This short communication given below is a summary of the EFFoST Critical Review 1 on 'Securing Safe Water Supplies – Comparison of Applicable Technologies'.

The full version is available as paperback or eBook. The review discusses the availability of potable water in the world and technologies to make water safe, in particular under difficult circumstances, such as after natural disasters. The review, however, covers technologies that are applicable everywhere, in disaster areas as well as in developing and developed countries. The review is the result of a thorough study by the authors of the global situation with respect to the availability of potable water as part of the EU Project NovelQ.

Introduction

Water disinfection is a crucial step in the production of safe drinking water as well as in the generation of water used during the processing of food. Different methods and technologies exist and need to be selected depending on the type of application and food industry sector. Whereas sophisticated solutions can be realised in industrialised countries, the situation becomes more challenging in developing countries and disaster areas where access to safe water is limited. According to Kofi Annan, former UN Secretary General (1997 – 2006), no single measure would do more to reduce disease and save lives in the developing world than bringing safe water and adequate sanitation to all.

In the UN Millennium Development Goals, signed in 2000 by 189 countries, target 7c states that, by the year 2015, the proportion of people without sustainable access to safe drinking water and basic sanitation should be halved (from 65 per cent to 32 per cent). This goal includes improved access to sanitation, drinking water sources, better hygiene and improved water quality by municipal or home water treatment. A classification of possible types of interventions is given in **Figure 1** (page 58)[1].

Climate change is one of the greatest environmental challenges facing the global community, and will have a worldwide effect on water sustainability. Water stress and scarcity will increase because of changes in seasonal rain and snow patterns, coupled with increased demand due to continued population growth. Currently, 1.1 billion people lack access

to safe water and 2.6 billion people lack access to proper sanitation. As a result, more than 4,500 children under five years of age die every day from easily preventable diseases such as diarrhoea[2].

There is emerging evidence that climate-induced changes in temperature and precipitation patterns will increase water-related infectious diseases, especially diarrhoeal diseases. In addition, there is already evidence that climate change is resulting in rising sea levels, increased cyclonic activity, and increased intensity and frequency of floods and droughts. Hence, in addition to the need for the continuous improvement of water access and quality in the developing world, there is a need for water decontamination in disaster areas. Natural disasters occur worldwide and very often produce an immediate need for clean, safe drinking water because of the damage to existing water supply systems, contamination of water sources and the creation of refugee camps.

Experience shows, however, that access to improved water supply facilities is no guarantee for safe and reliable water, especially in developing countries where tap water can be unsafe because of inadequate treatment and recontamination during transport or storage or at home. Disinfection is of unquestionable importance in the supply of safe drinking water and the destruction of microbial pathogens is essential[3]. Point-of-use and small-scale treatment is a promising strategy to improve access to safe drinking water, particularly for the poor. Easy-to-handle home water treatment devices could help to prevent many of disease outbreaks and save many lives, especially when combined with improved sanitation and hygiene, since many pathogens are waterborne.

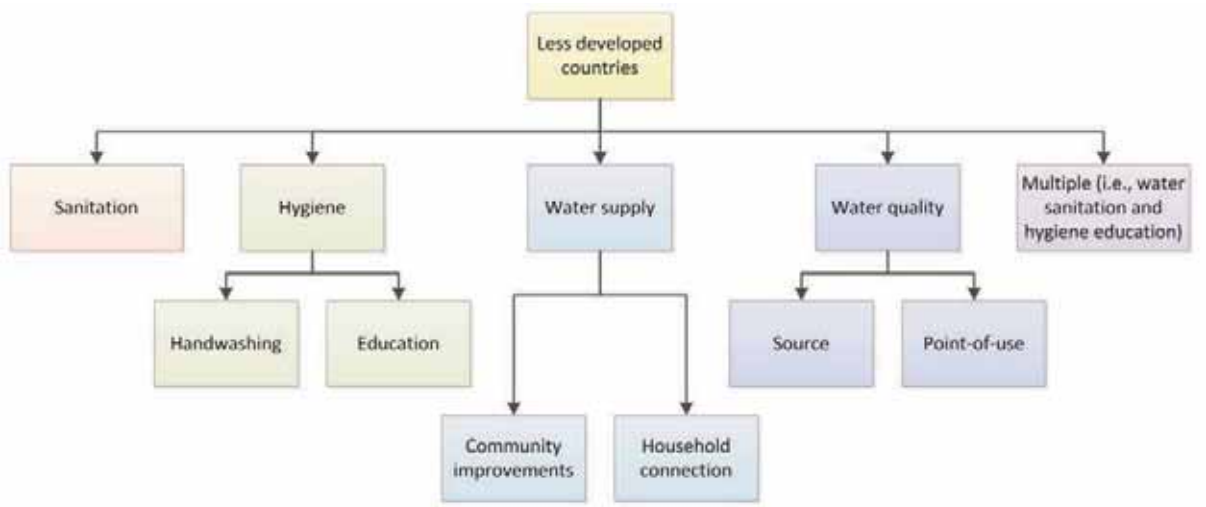

Figure 1: Classification of interventions to reduce diarrhoea in less developed countries

Sustainable supply, storage and transport of clean freshwater are a main challenge involving food process engineering and the food industry directly or indirectly. New concepts will be required considering the abovementioned aspects of water handling and linking it to food processing as well as to the agricultural production of food, considering that more than two-thirds of the fresh-water used on earth is currently needed to produce food and animal feed.

Basic methods for water disinfection

Boiling or heat treatment of water is effective against the full range of microbial pathogens and can be employed regardless of the turbidity or dissolved constituents in the water. However, the considerable amount of energy required (wood, charcoal, fuel) and associated costs including time are leading to alternatives.

Chemical disinfection reduces the overall risk of disease, but may not render the supply safe to drink. Chlorine is used in various forms and its antimicrobial effects are based on pronounced oxidation and rapid binding to proteins including enzymes and nucleic acids. However, the efficiency of chlorine depends on the presence of organic substances since these compounds react with the disinfectant and reduce its efficiency. Chlorine disinfection of drinking-water has limitations against the protozoan pathogens – in particular Cryptosporidium – and some viruses. Another common method of disinfecting water is ozonation, also known as ozone disinfection. Ozone is generated by electrical discharge through dry air or pure oxygen on site because it decomposes rapidly to elemental oxygen. Chemical flocculants such as ferric sulphate are used as a pre-treatment to remove solids from the water and improve subsequent disinfection. The use of chemical disinfectants in water treatment usually results in the formation of chemical by-products. However, the risks to health from these by-products are extremely small in comparison with the risks associated with inadequate disinfection. It is important that disinfection is not compromised by attempting to control such by-products. Some disinfectants such as chlorine can be easily monitored and controlled as a drinking-water disinfectant, and frequent monitoring is recommended wherever chlorination is practised[3].

Mechanical means such as filtration are also used in order to remove solid pollutants. Ceramic filters with small pores, often coated with silver for bacteriostasis, have been shown to be effective at removing microbes and other suspended solids. Filters need to be cleaned regularly. Reverse osmosis (RO) is best known for use in desalination (removing the salt and other minerals from seawater to get freshwater), but since the early 1970s, it has also been used to purify fresh water for medical, industrial and domestic applications. Reverse osmosis can be used to improve water for drinking and cooking, and portable reverse osmosis water processors are sold for personal water purification in a variety of locations.

Solar disinfection (SODIS) is a simple water treatment method, which uses solar radiation (UV-A light and temperature) to destroy pathogenic bacteria and viruses present in the water[4]. It can be used to

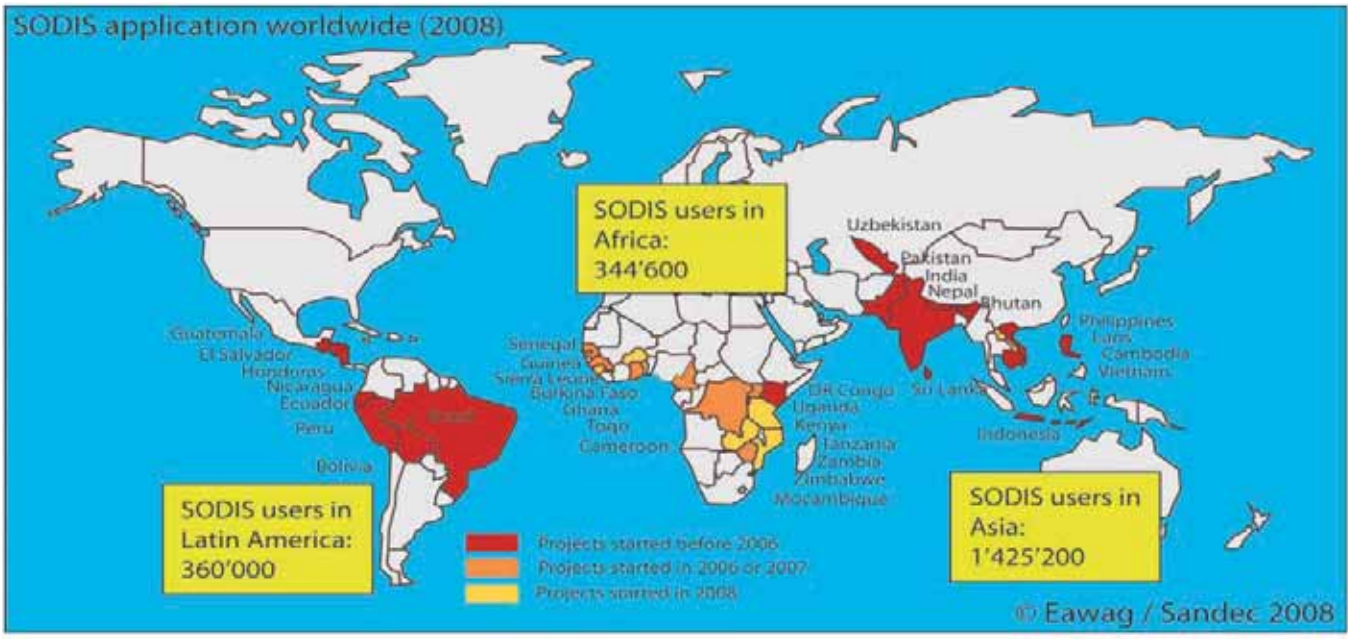

Figure 2: The estimated number of users of SODIS applications worldwide, including starting period of projects and countries in which SODIS is implemented as local house water treatment

disinfect small quantities of water with low turbidity. An overview on SODIS applications worldwide is given in **Figure 2**. Similarly, ultraviolet light is suitable for oxidation and reduces contamination in water. UV disinfection is a physico-chemical process, and the light is generated using special UV lamps. Ultraviolet light is also suitable for oxidation and therefore for the reduction of undesirable substances in water. For example, UV light with a wavelength of 185 nm oxidises hydrocarbons, chlorine, N-nitrosodimethylamine (NDMA) and reduces total organic carbon (TOC) values. In combination with ozone and peroxide UV-light can additionally be used for advanced oxidation processes (AOP) for degradation of persistent pollutants from drinking water.

Pulsed electric fields technology (PEF) is also a non-thermal method for decontaminating liquids. The treatment is effective for the inactivation of vegetative microbial cells. The power requirements depend on the scale of the system. The electric field strength applied to inactivate microorganisms is in the range of 2 – 4 kV/mm and can be generated for a small unit manually or by solar power.

For regions difficult to access or with low colonisation, it is more reasonable to implement several household-size systems. For refugee camps, highly colonised areas etc., it is more feasible to operate large systems. Uncomplicated, robust and easy-to-use technologies, which work in most climatic environments, are optimal for operating water sanitation systems in disaster areas. Not all of the methods listed above are convenient in rural areas of developing countries, because they may require skilled staff, electricity or fuel and the reliable supply of spare parts and consumables, like membrane filtration. Hence, depending on local conditions and advantages and disadvantages of the method, a selection needs to be made. Decisions about which system to choose and how much training effort has to be invested includes not only technical criteria but also local cultural beliefs and education. The development of adjusted processes by employing technical based knowledge as well as using modern tools in order to increase process efficiency is a key point in this context and will require input from a food engineering perspective.

Commercial water disinfection systems available on the market

The EFFoST Critical Review 1 on 'Securing Safe Water Supplies – Comparison of Applicable Technologies' contains an extensive list of commercial water disinfection systems comparing different aspects such as working principle, capacity, energy requirements, robustness and costs among others. There are many devices and systems available on the market. Furthermore, there are a number of competing commercial companies and several commercially available systems of municipal size, which are designed to be user-specific and purpose-built. However, significant deterioration of water quality may occur between water collection and consumption.

Unhygienic handling of water during transport or within the home can contaminate previously safe water. In such situations, investing in conventional water treatment at-source will not prevent diarrhoeal diseases. Household Water Treatment Systems (HWTS) and safe storage are cheaper and more effective. Household-level intervention can be very effective in preventing disease if used correctly and consistently. A high percentage of people could, therefore, benefit from effective household water treatment and safe storage practices.

As with sanitation, the promotion of water treatment at household level requires a behavioural change approach, which goes beyond the mere provision of technology. Householders must have the motivation to treat their drinking water, and the technologies and consumables must be affordable and easily available[5,6].

Although household water treatment is important, ensuring water quality at-source remains crucial. Unfortunately, this is an area of increasing global concern. Systems in industrial / municipal size are mainly designed for hospitals, schools, refugee camps and water sources of (municipal) communities up to 40,000 people. Working principles mainly include multistage processes and a modular conception consisting of flocculation, filtration and disinfection.

Conclusion

Water is a scarce source in temporal and regional terms and water shortages as well as water crises affect many people suffering from limited access to safe water as well as basic sanitary services. An effective overall water management strategy incorporates multiple barriers including at-source water protection and appropriate treatment as well as protection during storage and distribution in conjunction with disinfection to prevent or remove microbial contamination. Sustainable water management also plays a major role for food production and food processing which depend on the availability of high quality water as well.

Available water disinfection systems rely mainly on large-scale filtration and a combination of filtration (to remove solids) and subsequent chemical disinfection. This approach has proved to produce water of acceptable quality. Important points for use in household systems are low complexity, few training requirements, and easy transportation and distribution as well as a sufficiently high acceptance by the user.

The use of a Pulsed Electric Field (PEF) system in disaster areas will require water with a low turbidity unit to allow proper operation of the system or the application of higher power levels. The lack of performance indicators and the risk of insufficient inactivation going unnoticed by the user need to be compensated for with reliable technical performance. The advantages and disadvantages of this alternative system should be considered carefully and, based on the results, its usefulness and further development for specific applications decided.

As an important part of improving water treatment, training and behaviour change need to be taken into account and education and promotion need to be implemented in order to reach the target group at the points of sale. Printed materials along with face-to-face communication should actively promote safe water systems and their benefits.

The EFFoST Critical Review 1 on 'Securing Safe Water Supplies – Comparison of Applicable Technologies' aims to provide relevant background information to allow a knowledge-based decision on the suitability of available, and the need for the development of, improved water sanitation systems.

Acknowledgements

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Application Notes

Monitoring the frying process with FT-NIR

Fast, non-destructive and cost-effective analysis of used frying oils

Frying is an ancient way of food processing, which can be traced back to the Egyptians. Today, people from all cultural backgrounds love the taste and the mouth feel of crispy food like fried meat or potato products.

In recent years, frying food has been stigmatised due to health concerns claimed in various studies, like cardiovascular disease, obesity or diabetes. The fact is however that the quality of the frying fat heavily influences the quality of the finished product. This makes the importance of a thorough quality control imperative – in fast food restaurants and huge frying operations alike. Whereas small frying operations mostly rely on experience and occasional quick test solutions, it is of utmost importance to constantly monitor the frying medium in large scale industrial fryers with high turnovers. Any deviation from the guaranteed quality will lead to loss in consumer trust and potential claims. Moreover, the tight control of the oil quality helps to cost-effectively manage the replenishing.

There are several parameters documented in the literature that correlate well to the quality of the used frying oil:

- Acid Value (AV)
- Anisidine Value (AnV)
- Total Polar Components (TPC)
- Polymerized Triacylglycerols (DPTG)

However, no single parameter is able to sufficiently explain the frying process and finally the food quality. Ideally, a combination of the indices, giving a complete picture of the chemical substances formed during frying would be desirable. All these parameters normally require a time-consuming analytical method, e.g. chromatography or titration. FT-NIR spectroscopy is on the other hand a fast, non-destructive and cost-effective technique which is already routinely used for the analysis of fresh fats and oils.

A study was carried out to establish validated methods based on a large data set using FT-NIR spectroscopy in order to analyse both the fresh and the degraded frying oils for the relevant quality parameters. The samples were analysed for the relevant parameters using the standard DGF (German Society of Fat Sciences) methods and analysed by FT-NIR in transmission (**Figure 1**). A mathematical relationship between FT-NIR spectra and the analytical parameters was established using multivariate calibrations.

Figure 1: Measurement of frying oils with FT-NIR spectroscopy

The statistical evaluation indicates that the measured FT-NIR spectra show strong correlations with the official lab methods (**Table 1**). The fact that the most important parameters of oxidation and polymerisation can be determined simultaneously makes FT-NIR a fast and reliable tool for companies and health authorities alike to check the degree of degradation.

This was acknowledged by the DGF who issued the Standard Method "FT-NIR Spectroscopy: Screening analysis of used frying fats and oils for rapid determination of polar compounds, polymerized triacylglycerols, acid value and anisidine value [DGF C-VI 21a (13)]" in September 2013.

Table 1: Result overview of FT-NIR calibrations

Property		Data Set			Performance	
Name	Unit	Number	Min	Max	R^2	RMSEP
Acid Value	mg KOH/g	459	0.05	5.7	97.51	0.18
Anisidine Value	-	378	0	172.6	95.24	7.88
Total Polar Compounds	%	410	2.03	44.2	97.05	1.63
Di-and Polymerised TAGs	%	500	0	31.3	99.15	0.66

n = Sample number; R^2 = Coefficient of determination; RMSEP = Root Mean Square Error of Predicition

Can LC-MS/MS be used in horsemeat detection?

Following the Food Standards Agency's (FSA) announcement earlier in the year that horse and pig DNA had been identified in beef products sold in supermarkets, further testing across Europe and beyond has revealed widespread incidences of the contamination. Most testing methods, however, are based on detection of species-specific DNA in meat, using the polymerase chain reaction (PCR) – which does not detect or identify proteins. This is a concern because DNA can be easily disrupted or removed during standard meat processing and food manufacturing. As a result, horse tissue or other contaminants remain undetected in food samples, despite strong presence of the contaminating proteins. An alternative protein-based method, ELISA (enzyme-linked immunosorbent assay), can also be used to complement DNA testing, but this method has limitations, including that it detects only one part of the protein and not multiple protein markers.

AB SCIEX has developed an LC-MS/MS method that offers a more accurate and reliable approach to meat speciation than PCR or ELISA-based techniques, and also allows for the simultaneous detection of veterinary drug residues in the same analysis. The method utilises an Eksigent ekspert™ microLC 200 UHPLC system coupled with an AB SCIEX QTRAP® 5500 LC-MS/MS system. The method is able to detect specific peptide markers for horse as well as providing peptide / protein sequence information to further confirm the species identity. This approach provides greater confidence for food testing by distinguishing between species at the amino acid level as well as in this example, being able to detect prohibited substances, e.g. the non-steroidal anti-inflammatory drug (NSAID) phenylbutazone (BUTE).

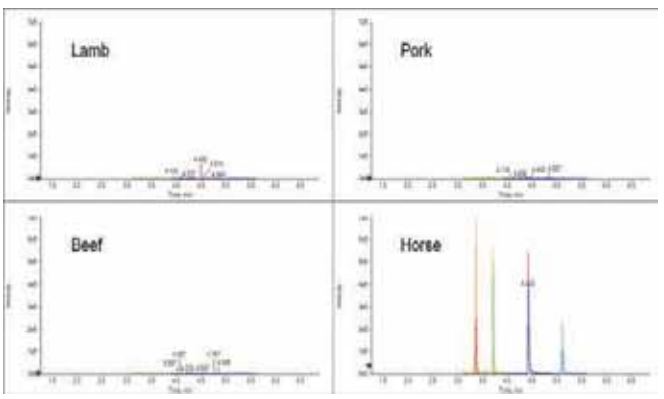

Figure 1: Unique peptide signatures for horse meat detected and analysed for in a variety of meat extracts

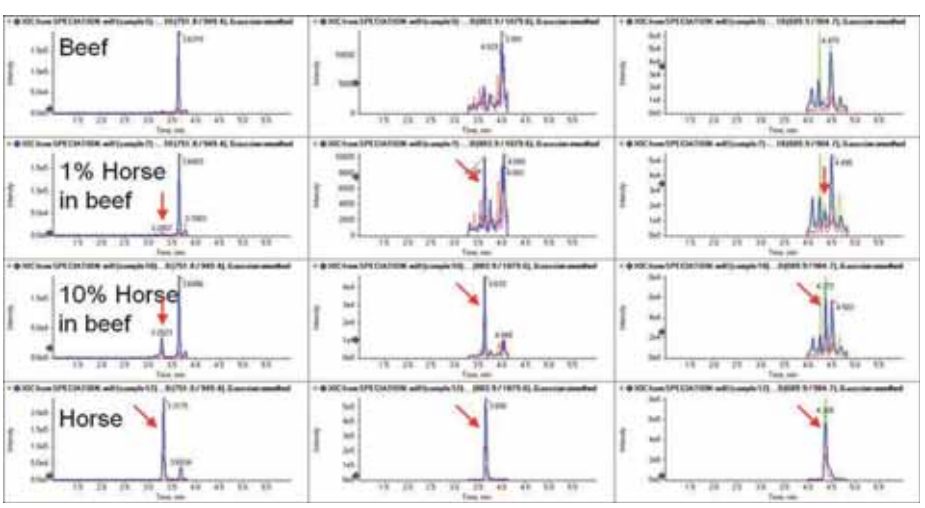

Figure 2: Detection of characteristic peptides of horse meat in beef at different levels, highlighting that horse meat can be detected at a 1% level by LC-MS/MS

Results and discussion

During method development, care was taken to make sure that peptides chosen were unique to the meat species. The list was further consolidated by removing peptides that could be susceptible to modification during food processing, e.g. undergo post translational modification or the Maillard reaction. This reduced the number of peptides used as triggers for detection and generation of peptide fingerprints of species. **Figure 1** shows a comparison of horse, beef, pork and lamb extracts where four unique peptides for horse are shown from a method which contains additional markers for other species. **Figure 2** shows the comparison of beef and beef reference material which had been spiked at 10 per cent and at one per cent horse (current detection limit for PCR). In this figure, three of the four peptides have been extracted and it shows clearly that horsemeat can be detected at a one per cent spike level. The fourth peptide was detected at 10 per cent level but it was below the LOD limit at one per cent horsemeat in beef. In order to confirm these results, extractions of samples were performed multiple times and in each batch, one per cent horsemeat could be detected in beef.

Summary

LC-MS/MS has the potential to offer a rapid, robust, sensitive and specific assay for the simultaneous detection of a series of meat species as well as veterinary drug residues in a single analysis. Sensitivities achieved were equivalent to sensitivities of some currently available methods based on ELISA and real-time PCR. The LC-MS/MS approach has the additional advantage of being a potential multi species screen unlike ELISA where individual meat species are detected by separate kits.

Rapid automated determination of possible migrants from paperboard packaging material by solid-phase micro extraction coupled to gas chromatography-mass spectrometry

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Packaging is a key part of food technology. It provides protection for food against external influence; keeps it fresh, safe and attractive. The use of packaging materials for food is regulated in the European Union (EU) by EU Framework Regulation EC 1935/2004. There are a number of other regulations, the most recent being Regulation EU/10/2011 which focuses on plastic materials and articles intended to come into contact with food. The packaging legislation stipulates which chemicals should not transfer from packaging material to food, which chemicals should not be used for production of food packaging material at all, and it also establishes limits for compounds that cannot be present either in food or packaging material itself.

A problem that often arises from inappropriate packing is a change in sensory properties of food. Paperboard can be produced from virgin paper, from recycled paper, or a mixture of both. Recycled paperboard can contain a wide range of dangerous contaminants caused by the degradation of paperboard components including printing inks, coatings, and adhesives. In this article, an analytical method for the determination and quantification of 12 possible representative migrants (phthalates, photoinitiators, phenols and off-flavors) from paperboard is presented. Solid phase microextraction (SPME) coupled to gas chromatography mass spectrometry (GC-MS/MS) has been applied to detect volatile and semi-volatile compounds of interest, as volatile compounds will more likely migrate from packaging to food[1].

Table 1: Validation results: method recovery (%), method repeatability expressed as RSD (%) and limits of detection and quantification (LOD and LOQ)

Compound	Spiking level (µg/kg)	LOD (µg/kg)	LOQ (µg/kg)	Repeatability (%)	Recovery (%)
1-hexanole	750	100	300	8	76
1-methoxy-2-propanol	75	20	60	17	107
2,4-di-tert-butylphenol	7.5	0.3	1	11	87
2_ethyl-1-hexanol	75	20	50	17	106
Allyl benzoate	7.5	0.3	1	14	87
Benzaldehyd	75	2	5	14	97
Benzophenone	7.5	16	50	8	91
Dipropylenglykol-monomethyleter	7500	2500	7500	22	76
Dimethylphthalate	75	8	20	9	96
Ethyl benzoate	7.5	1.5	5	14	88
Hexanal	7500	35	100	21	98
2,4,6-trichloroanisole	7.5	0.03	0.1	17	103

Experimental

The principle of the method is very simple. A small amount of sample is placed in a 20mL headspace (HS) vial and an optimised amount of extraction solvent is added. A paperboard sample is cut into small pieces (2 mm x 2 mm) and one gram is weighed and directly added to the HS-vial. Then, 8mL of extraction mixture (13 per cent methanol in water) is added and the vial is placed in the tray for automated SPME analysis.

Figure 1: Thermo Scientific TSQ 8000 Triple Stage Quadrupole Mass Spectrometer

The instrument used is the Thermo Scientific™ TRACE™ 1310 gas chromatograph coupled to the Thermo Scientific™ TSQ™ 8000 triple-stage quadrupole mass spectrometer equipped with Thermo Scientific™ TriPlus™ RSH Autosampler (**Figure 1**).

The vial with the sample is first transferred to the heated agitator where it can be incubated. The fibre in the special sheath is immediately immersed into the headspace of the vial and is extracted at 65°C for 45 minutes. During the extraction period, the target compounds are adsorbed on the fibre's polydimethylsiloxane coating (PDMS, 100µm). To optimise the extraction, the agitator swirls the vial in addition to heating. After extraction, the fibre is removed and inserted in the injection port for desorption (seven minutes) and GC-MS/MS analysis is initiated. In the meantime, during the analysis of the first sample, the fibre is conditioned for 20 minutes and the extraction of the next sample begins.

Results and discussion

This method benefits from a significant decrease in extraction solvent and reduced sample preparation time, so it is cost effective and environmentally friendly. It is recommended for routine use in monitoring the content of packaging migrants in paperboard. After optimisation of all important features, the method was successfully validated in-house (**Table 1**) and applied in a small survey covering paperboard samples of various qualities including both virgin and recycled paperboard.

Reference

1. Arthur CL, Pawliszyn J (1990), Anal Chem 62:2145-2148

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In a nutshell

An interview with
Rohit Shroff, Application Specialist, Tecan Schweiz AG

Tecan is a global provider of instruments and solutions for laboratory automation in various markets with a broad portfolio for the food testing industry. Founded in 1980 in Switzerland, Tecan now has over 1,100 employees and owns production, research and development sites in both Europe and North America; maintaining a sales and service network in 52 countries. An original equipment manufacturer (OEM), Tecan is also a leader in the development and manufacture of OEM instruments and components that are distributed by partner companies.

Safe and healthy food is a fundamental necessity for all people, and while advances in storage and transportation technologies and a globalised food supply chain provide a constant supply of food products to consumers, it also poses a significant challenge to manufacturers and regulatory agencies to ensure that the supply is free from harmful substances. To combat this challenge, the food industry has adopted a 'farm-to-fork' food safety approach which involves monitoring of raw materials, semi-finished and finished products. In addition, recent food scandals, such as melamine in milk, have implicated that the regulatory environment is getting tougher, with an ever-increasing number of compounds to be tested before the release of a product batch. "This is where mass spectrometry fits – in line with the need of food safety analysis," says Rohit Shroff, Application Specialist at Tecan. "It allows for specific identification and quantification of compounds in a broad variety of food matrices.

"Mass spectrometry delivers the sensitivity, specificity, short turnaround times and high-throughput that a food testing laboratory requires, and consequently liquid chromatography mass spectrometry (LC-MS) is a widely used tool in today's food safety arena. LC-MS is used to test for pesticides, antibiotic drug residues, contaminants, allergens, adulteration, authenticity profiles, vitamins and nutritional analysis. Major developments in the LC-MS technology also enable the detection of hundreds of different compounds in a single analysis; providing significant advantages over several other analytical techniques."

What are the current hurdles for food scientists working with LC-MS? "Sample preparation prior to LC-MS analysis has emerged as the major bottleneck for food scientists," reveals Shroff. Sample preparation is critical in obtaining sensitive, reproducible data from LC-MS instruments. And while sample preparation can be as simple as sample distribution to a new tube or plate format, it can also include various extraction techniques to be able to enrich or extract specific compounds required for further downstream analysis.

"As food matrices are heterogeneous, these extraction techniques can range from simple sample dilution prior to injection into an instrument to solid phase extraction (SPE), liquid-liquid extraction (LLE) or protein precipitation / purification techniques," Shroff explains. "Sample preparation is carried out manually in large parts by laboratory technicians, but manual sample processing can lead to a comparably low throughput, a higher number of repeat tests and overall lower quality of data, which ultimately results in higher variation depending on the expertise of the technician carrying out the tasks."

Tecan's range of automated solutions can help to address the issue of manual sample preparation. "Tecan's Freedom EVO® platforms seamlessly automate sample preparation protocols such as SPE, LLE and protein precipitation and purification. The instruments can prepare samples in different formats ready for injection into LC-MS devices. They also allow generation of customised output files which can be fed into LC-MS instruments as batch lists, reducing manual interactions and documentation to a minimum," says Shroff.

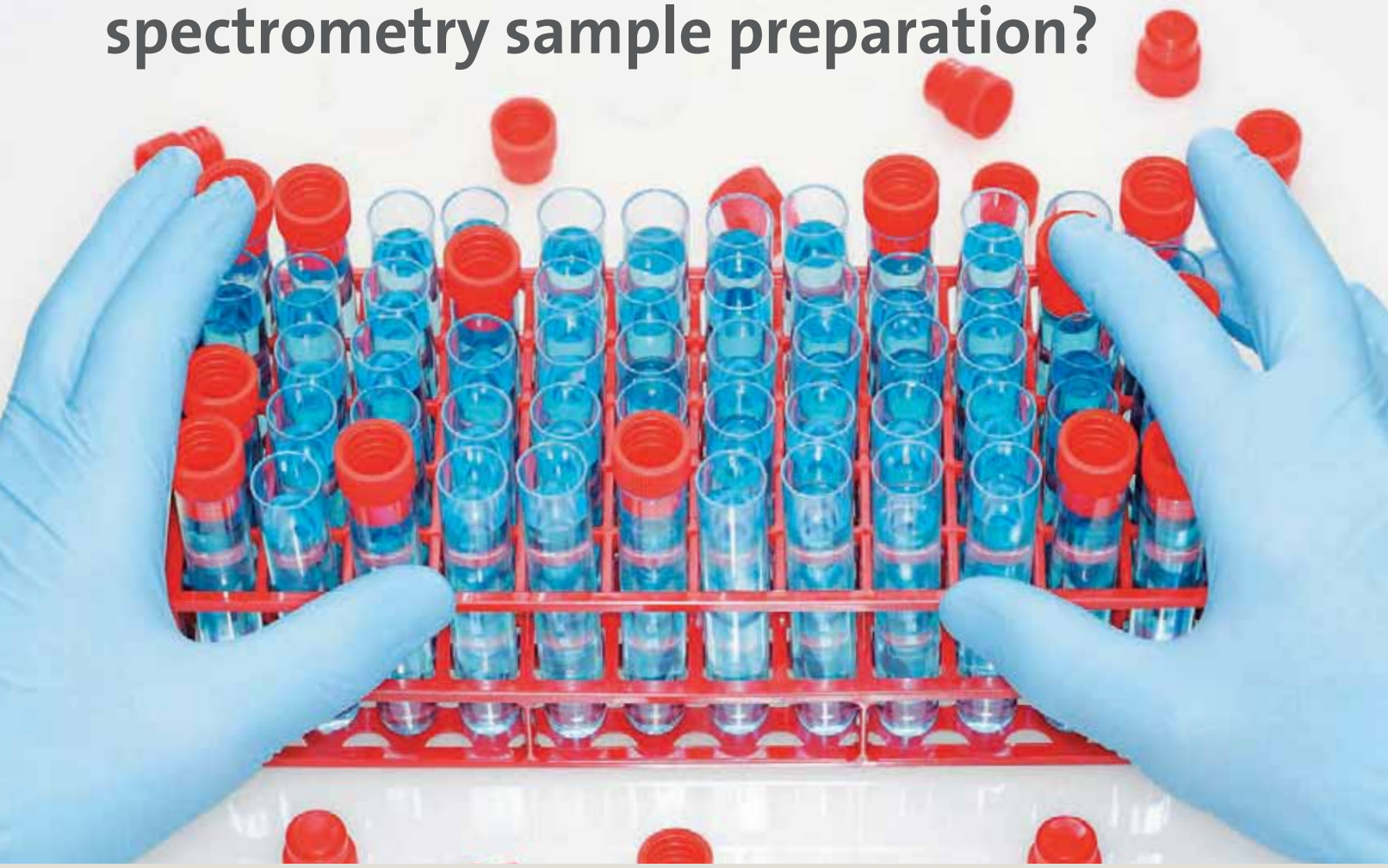
"Full process automation allows for extended walkaway times and even for unattended overnight runtime, which allows our customers to achieve their throughputs and enables staff to attend to other tasks. Tecan is also committed to providing easy-to-use solutions, which can be seen in the TouchTools™ software, which allows the customer to run applications by working their way through a graphic user interface, without having to learn liquid handling software.

"The ability to transfer sample preparation and assay steps to an automated liquid handling platform allows larger facilities and companies to share common procedures between subsidiaries or departments, greatly reducing the training effort to implement the procedures with new employees and different facilities."

Tecan provides highly flexible and modular platforms which can be tailored to the specific needs of a customer, beyond sample preparation for mass spectrometry. This includes chemical analysis, ELISA testing, microbiology and genomic testing.

"The Freedom EVO® platform is available in three sizes and can be integrated with a variety of modules, depending on the customer's application of interest," says Shroff. "Tecan understands that laboratories need to be dynamic – the protocols they are running may change over time. The modular worktable of our instruments allows our customer to adjust to these changing needs in food testing; providing longevity of the automation solution and, consequently, a high return on investment."

Had enough of tedious mass spectrometry sample preparation?



Sample identification → Sample reagent, and standard pipetting → Sample extraction → Output to analyzer format

Sample preparation is not glamorous. However, reproducibly performing this cumbersome step is critical to getting the best data, and highest return, out of your mass spectrometry investment.

To help you do this, Tecan offers Freedom EVO®-based end-to-end process automation simplifying even the most challenging protocols, liberating you from the tedium of manual sample preparation.

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