White Paper

Analytical Solutions for Solving Food Product Problems

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Introduction

Anyone involved in food production recognises that there are many factors that can compromise the quality and/or safety of the end product. They can, however, largely be categorised as unexpected issues with raw materials, failure of staff to follow standard operating procedures, and failures of process equipment.

The last two, staff and equipment, are to some extent more easy to recognise. If a fruit cake is missing its cherries because someone neglected to add them to a hopper, it is relatively easy to identify who was at fault. If the cake has the wrong texture because an oven has failed to reach the appropriate temperature, then process monitoring devices can be expected to alert the issue at the time, or at least explain the problem once a quality assessment has taken place.

Problems arising from unexpected issues with raw materials (in this paper, we consider raw materials to be ingredients, as well as packaging and chemicals used on or around the process environment) can be much more difficult to identify and explain. Indeed, they can be difficult even to characterise since the outcome of a contamination incident, for example, might manifest in different ways in different products. The same issue with an ingredient might produce a taint/off-flavour in one product, but a discolouration in another. It might result in both outcomes or neither.

Viewed from the reverse perspective, which is the one that emerges in real-life, when a problem with a product is reported, it is never possible to point immediately to the root cause. The product that 'smells bad' may be explained by a chemical contamination, but equally, the root cause might be microbiological. If chemical, the contaminant might come from packaging or from the environment, from an external source or from decay of an ingredient, or perhaps from a combination of processing factors that are in-spec yet unexpectedly combine together to create an out-of-spec product.

Every incident/problem is different, hence problem solving requires different approaches. For the purposes of this paper, we categorise problems by the broad definitions shown in Table 1. However, as will be clear from the above, these categories are somewhat arbitrary. A given problem is seldom so easily classified, and any given incident might feature an element of one or more of these categories.

<table>
<thead>
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<th>Table 1. Broad classification of product problems</th>
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<td>• Appearance/foreign body - the product looks 'wrong' or has something in it that should not be there</td>
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<td>• Taste/smell - the product tastes or smells 'off' or 'wrong'</td>
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<td>• Performance - the product doesn't behave as intended, either in its packaging or in use eg. a sauce won't pour, a biscuit is too crumbly, a gum is not chewy</td>
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<tr>
<td>• Authenticity/purity – ingredients are suspected to be adulterated or diluted</td>
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<tr>
<td>• Unspecified customer complaint related to illness - the customer reports illness, especially vomiting or allergic reaction (which may include factors related to the above)</td>
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Any of the problems featured in Table 1 will merit further investigation, whether identified within the production environment or reported by customers. Indeed, the problem may not be directly associated with one's own products. It may be the case that an industry wide issue (horsemeat in beef, melamine in milk, acrylamide in fried products) leaves a manufacturer with the problem of proving that their own products are not similarly affected.

Analytical approaches to problem solving

There is a range of powerful analytical techniques available to the modern laboratory that can answer many of the key questions about what has gone wrong with a product. Powerful need not always mean sophisticated, as there are occasions when a relatively simple test can provide a great deal of useful information. That said, it is important to emphasise that any given problem may have a number of factors working together to create the 'perfect storm' and therefore, any investigation of a problem may often involve a multi-disciplinary approach in order to reach a satisfactory conclusion. Moreover, any investigation should not look merely for an
exploration, but also to offer remedies to prevent a recurrence. Clearly, it is not sufficient to identify that the hairs found in a product are mouse rather than rat, then do nothing towards improving rodent control. We also need the investigation to identify the vulnerability in the processing factory or supply chain, and rectify any issues.

Taking each category in turn from Table 1, we now examine some of the techniques/strategies available for investigation. Though a discussion of sampling is beyond the scope of this paper, it is necessary to note that the way in which samples are taken, packaged and sent for an analysis is hugely important. Errors made at the sampling stage have the potential to damage or destroy evidence and negate the significance of any analysis.

**Appearance/foreign body**

Blemishes and discolouration may be the result of chemical contamination, or perhaps microbial spoilage. The chemical techniques referred to in the discussion of taints may be appropriate to use in this instance too. Other cosmetic/aesthetic problems, such as sediments, a visible separation of emulsions and such like will be investigated by the physical techniques applicable to performance problems. Foreign bodies are a problem category in their own right.

Foreign bodies can be broadly classified as extrinsic or intrinsic. The former are materials that have no place in a food product, coming from external sources either by deliberate or accidental means. They might include swarf from process machinery, insects or other animal body parts, pieces of glass from an item broken in the factory, or any other item, such as a razor blade introduced maliciously, perhaps by a consumer attempting an extortion. Intrinsic foreign bodies include ingredients such as bones and gristle in a meat product, or a leaf or stalk in a pack of frozen vegetables, or an ingredient in an unusual/unexpected state. They might also include fragments of packaging or a blemish on an otherwise perfect surface, e.g. a smear of raspberry jam on a white iced cake. If the last example seems trivial, the point is that when the foreign body is first noticed, it may not be at all obvious what it is. To one consumer, the red smear will be interpreted as jam and probably ignored. To another, it could look like blood, and when their complaint comes in, that is the complaint that has to be investigated, proved or disproved, and then dealt with.

It is always sensible to investigate a foreign body incident thoroughly, even if it is assumed to be a one-off. A thorough investigation demonstrates a commitment to 'due diligence' and should help in restoring consumer confidence.

A broad spectrum of technologies is routinely used in foreign body investigations, reflecting the diverse nature of potential contaminants. However, relatively simple light microscopy is often the starting point of any investigation, as it can be used to determine features that are typical or characteristic of the likely candidates, thereby directing the scientists to the more sophisticated methods that will provide ultimate confirmation.

Different techniques are required for different types of contaminant. So for example, a scanning electron microscope fitted with an energy dispersive X-ray (EDX) detector is useful for identifying the elemental composition (and hence providing a positive ID) for items such as glass fragments. On the other hand, plastic fragments, which may look like glass, require confirmation using a technique known as FT-IR (Fourier transform infra red spectroscopy). For the purposes of this article there is no need for detailed discussion of the technologies. The point is that even the smallest contaminant can usually be identified, provided the lab doing the work has the appropriate expertise and equipment.

It is worth noting that identification goes way beyond merely describing the contaminant as a piece of plastic, a slice of glass, or a fragment of bone etc. It matters to know, and it is usually possible to tell, what type of plastic, what type of glass and what kind of animal.

Taking glass as the example, a proper investigation can differentiate between the different types of glass used in a domestic or commercial setting. It can differentiate between sheet glass and spun glass, borosilicates (Pyrex), leaded glass and specialist glasses, such as those used in laboratories. Moreover, it is also sometimes possible to tell whether the foreign body has been processed alongside the food, added afterwards, or come into contact with any other ingredient or item that might indicate where it came from and how it got into the product.

A key resource in identifying contaminants is the availability of reference materials against which to check the sample provided. This might, for example, expose a maintenance issue with a piece of factory equipment, or rule out the possibility that a given foreign body...
arose within a particular production environment. In deciding, for example, whether a particular ingredient was the source of a foreign body, it may be necessary to take this investigation much further down the supply chain.

**Taste/smell**

A bad or inappropriate taste or smell in a product is usually due to the unexpected/unwanted presence of any number of chemicals that have the potential to be tasted or smelled. That need not always be true. It may be that there is an absence of chemicals (e.g. bitterness blockers), or failure of a taste-masking system (e.g. encapsulation of oils), that might otherwise be expected to keep the unwanted flavours in check.

Nor is it necessarily the case that the offending chemicals arise from direct contamination of the product. It might be that the packaging is the root cause of the problem, or the airspace in a drinks bottle. The unwanted chemical may have been introduced into the product due to microbial spoilage, or perhaps from reaction of ingredients within the product.

The potential for a chemical to cause a taint is indicated by its flavour threshold (taste or odour). This is the concentration at which the chemical would be perceived (smelt or tasted), in a given matrix, by 50% of a population of consumers. There are several observations to be made from this statement. In the first instance, it should be clear that for any given chemical, its ability to taint a product will depend on the strength of other flavours present, and on the specific sensitivity of individual consumers to that particular taint. Hence a natural yoghurt might be more vulnerable to taints than a highly flavoured fruit yoghurt. However, given that some potent taints have a flavour threshold as low as 20 parts per trillion (ppt), it is not safe to assume that a taint will be masked automatically by other flavours or that the majority of consumers will be insensitive to it.

To give some idea of how potent some taints can be, imagine that sugar had a flavour threshold of 20 ppt. It would be possible to take one grain, dissolve it in the water of an Olympic size swimming pool, take a glass of water from the pool and taste the sweetness. Taints as potent as this can be hard to disguise from consumers regardless of other flavours present.

Unfortunately, consumers are generally more sensitive to some taints than even the most sophisticated analytical instruments. Therefore, isolating and identifying a tainting chemical is often a challenge. The description of the taint given by the customer, or by a specially trained sensory panel if necessary, is often vital in directing the efforts of the expert chemist towards identifying the offending chemical.

Certain groups or families of chemicals give rise to specific flavours/odours that an experienced chemist will be able to recognise. Hence cheesy and rancid flavours tend to be indicative of short chain fatty acids; soapy flavours with longer chain fatty acids. A musty, damp off-flavour is often associated with a group of particularly potent tainting species known as haloanisoles, but this short list is by no means exhaustive. There are thousands of potential tainting chemicals in widespread use.

An important point to note is that a taint or off-flavour can originate at any point in the lifetime of a product, from production of the raw materials through to eventual consumption. Hence it may be necessary to trace the source of a taint in a yoghurt back to the farms that produced the milk or the fruits, to the tankers/trailers that collected them, to the dairy that processed the product, to suppliers and carriers of other ingredients, or to the company that supplied the packaging for the finished goods. In some cases it may even be traced to a certain retailer, or even the consumer's home.

Investigation of taints and off-flavours will usually involve an initial assessment by human senses. If the offending chemical is believed to be organic (i.e. carbon-based) there will follow a chemical extraction procedure, separation via chromatography (either liquid or gas) and identification of the chemical concerned using mass spectrometry or possibly nuclear magnetic resonance spectrometry. A metallic contaminant will be detected using inductively coupled plasma (ICP) mass spectrometry or ICP-atomic emission spectroscopy.

This simple description belies the complexity that might be involved. As noted above, taints can be present in parts per trillion concentrations. There is huge skill involved in extracting the taint, at detectable levels, free of other chemicals that might interfere with the analysis, and (in the case of volatile odours) without simply losing the chemical to the atmosphere before it can be identified.
Performance

There is a range of performance issues that affect products, and similarly a wide range of physical chemistry methods that can be used to investigate problems. Problems to do with flow can be addressed using rheological instruments. These measure the shearing forces that determine how materials flow against different surfaces and internally. Such measurements are relevant also to address processing problems as well as problems with finished products. The resistance to flow by powders within hoppers, for example, can lead to clogging, or an accumulation of out-of-date material. Similarly, with liquids processed in a factory, or transported by tankers, resistance to flow can create major headaches for manufacturers, (whether that is within process equipment or when delivered as a layered component of the product) which may be solved by some fundamental investigation/research.

It is surprising how often a performance issue comes down to a problem with particle sizes or shape. The instability of an emulsion, or the gritty mouthfeel of a product is often dictated by particle size. This can be investigated using laser diffraction instruments, often in combination with microscopy techniques. The size and shape of particles can also be measured using the technique of static image analysis, which is a useful alternative to manual microscopy measurements.

It is sometimes possible to observe how ingredients are distributed within a product, based on their elemental composition, by using X-ray diffraction techniques along with the scanning electron microscope. This can give useful information about why a product performs badly. Even simple light microscopy, used with staining procedures can identify some of the microstructural features that are giving rise to performance problems.

The modern laboratory also has access to a range of instruments that can load products with crushing or stretching forces to investigate structural weakness (or strength).

Customer complaint/illness

Customers may attribute illness to a particular food item that shows no other signs of being at fault. Clearly, a manufacturer will want to investigate a complaint of this kind, with the possibility of allergen or pathogen contamination being a particular cause for concern.

Microbiological testing can deal fairly certainly with the latter. Allergen testing can be more complicated depending on the nature of the complaint and the likelihood, or otherwise, that specific allergens might have come into contact with the product or its ingredients/packaging.

Tests for allergens usually rely on enzyme-linked immunosorbent assay (ELISA) techniques directed at specific allergenic proteins, or DNA techniques that can detect trace amounts of DNA associated with allergenic ingredients. In both cases, but especially with ELISA, it is important to be aware of the potential for interferences that can lead to false positive or false negative results. Indeed, the potential for false positives/negatives is such that any of these tests must be properly validated before results can be relied on, or further action taken.

Authenticity/purity

High profile scandals (horsemeat in beef, melamine in milk, sudan red in spices) remind us that not every player in the supply chain is honest or legitimate. Whether there has been a specific incident, notification of a wider industry-concern, or merely a desire on behalf of the manufacturer to protect their interests (perhaps with a new supplier), there may be analytical approaches that can be taken that will assist in determining the authenticity of a particular supply. However, this is very much dependent on the ingredient in question, and authenticity is not always an easy matter to prove.

Testing for authenticity in the case of meat is relatively routine, using DNA methods that can target gene sequences found in one species but not in another. Hence, a competent, experienced laboratory should be able to detect trace amounts of one meat when mixed with another. Similarly, DNA methods are applicable to fish speciation, and to certain strains of rice, and a few other ingredients such as nuts.

Other authenticity issues are more complex. Olive oil is one food that has well defined acceptable ranges for a variety of naturally occurring compounds giving the analyst a set of parameters that can be measured when assessing the authenticity of the oil.

Similarly, there are recognised bio-markers for several other ingredients, but not always a defined acceptable range, meaning that low levels of a given biomarker in a fruit juice, for example, is not always definitive proof that the juice has been diluted. Natural variation may be
the more logical explanation, and it is an assessment of many variables that allows an analyst to reach a conclusion about authenticity.

Where adulteration is suspected, new methods may need to be developed as a matter of urgency to address the specific problem. In the melamine in milk example, melamine, a nitrogen-rich chemical, had been added to milk to fool a test that judged milk quality by its nitrogen content. That particular test had no means of detecting the source of nitrogen, and an entirely different way of testing milk was needed for this fraud to come to light. If there is suspicion that an ingredient is being adulterated, it will help the analyst considerably if the suspicion is backed by some idea of what the adulterant might be.

Conclusion

It is impossible to give a definitive list of problems and solutions, even in a very broad sense. Each problem needs to be assessed and investigated on its own merits in order to be understood. Similarly, the solution will have to be tailored to the specific incident.

It is often the case that the solution involves a review of SOPs and an element of staff training. However, whereas generalist food safety training may be sufficient to deal with a hygiene related incident, more specialised training (of management and factory-floor staff) may be necessary for dealing with issues such as HACCP and allergens. Similarly, specialist consultancy is available to help manufacturers address specific issues such as allergen management, and the expert input should help to avoid the 'coming together' of circumstances that lead to problems.

It goes without saying that it is impossible for every manufacturer to equip itself with the resources and expertise that would be needed to investigate every potential problem that exists, or is lying in wait. Rather it is important that they take action in advance of a problem, to identify the analytical support that might be needed should any kind of incident arise. When the crisis strikes is not the time to be searching for a laboratory partner.

Of course, it is hard to know what support might be needed until an incident actually occurs. However, it is in the interests of every manufacturer to identify in advance, a multi-disciplinary laboratory, with in-house or partnership expertise in chemistry, microscopy, physical sciences and microbiology, and crucially with proven experience in trouble-shooting/problem solving. At least that will ensure that when the problem occurs, whatever it happens to be, there is help at hand to identify what has gone wrong, and what needs to be done to put it right.

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Jane has worked in the food industry for 28 years in roles spanning various sectors and functions including research & development, quality and analytical science. With her Food Technologist background and many years spent working primarily in Research & Development, undertaking challenging development projects for the global food industry, Jane has built up a huge knowledge about the problems that arise whilst bringing products to the market place. In addition, Jane is now involved in supporting the industry identifying which technical solution is best placed to help resolve and identify the root cause of many of the technical problems they face.

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