Institution: University of Oxford

Unit of Assessment: 8 Chemistry

Title of case study: UOA08-04: Measuring chilli heat with electrochemistry

1. Summary of the impact

Richard Compton's group at the University of Oxford has developed an electrochemical sensor which uses multi-walled carbon nanotube electrodes to detect capsaicin molecules and related capsaicinoids – the chemicals responsible for the hot taste of chilli peppers. The technology, patented in 2008, has been licensed to the English Provender Company, which uses the sensors to perform quality assurance on 10 tonnes of incoming chillies every month, as well as monitoring reproducibility of finished products. In February 2013 Singapore-based Bio-X obtained an exclusive licence for the patent in Asia to develop, build and sell devices on a global basis. The science behind the technology has been the subject of significant outreach activities at UK schools, and has attracted extensive media interest.

2. Underpinning research

The 'heat' or piquancy of chillies is a result of the molecule capsaicin (8-methyl-N-vanillyl-6-nonenamide) and related compounds called capsaicinoids. Chillies are used ubiquitously throughout the food industry, and in order to ensure consistency there is a need to assess chilli strength – or, more technically, capsaicin content. Such measurements are traditionally performed using organoleptic (taste) testing: chilli pepper extract is diluted in sugar solution until the 'heat' is no longer detectable to a panel of five tasters. The degree of dilution gives a measurement on the Scoville scale, which dates back to 1912, and yields numbers which range from 0 to 16,000,000. By means of illustration, a Jalapeno pepper scores 2,500, Tabasco sauce 30,000-50,000, and pure capsaicin 16,000,000. The Scoville test can give accurate results when performed by expert tasters. However results are entirely dependent on the capsaicin sensitivity of the tasters, and the test is time-consuming and costly to perform since it requires a panel of five experts for every measurement of chilli heat. More consistent scientific methods of measuring capsaicin content involve cumbersome and expensive high-performance liquid chromatographs, which also require trained staff and involve time for sample preparation and results analysis.

In the mid-2000s Richard Compton and colleagues in the Department of Chemistry, University of Oxford began to undertake fundamental studies of adsorptive stripping voltammetry (AdSV) at multiwalled carbon nanotube modified basal plane pyrolytic graphite electrodes (MWCNT-BPPGE) – electrodes coated in carbon nanotubes. The Compton group applied the technique to a variety of analytes; with paracetamol [1] they obtained a detection limit of 10 nM, almost certainly the lowest limit of detection thus far reported for paracetamol using electrochemical techniques, showing that the technique was extremely sensitive. In a similar study on 4-hexylresorcinol (an anaesthetic and antiseptic used in pharmaceutical products) [2], Compton and colleagues experimented with MWCNT-BPPGE modified screen-printed electrodes, thereby demonstrating that the AdSV technique could easily be incorporated into a simple and inexpensive electrochemical sensor.

Compton and colleagues also studied food anayltes, specifically capsaicin molecules, which they observed to spontaneously adsorb onto a MWCNT-BPPGE. Further, when the voltage at the electrode was adjusted appropriately, the molecules were oxidised and stripped from its surface. This finding led to the development of an AdSV technique which built on the fact that, if deposition and stripping conditions were kept constant, the process could be calibrated to link the voltammetric stripping current to solution concentration [3]. The Compton group then developed an innovative electrochemical technique to reliably quantify the concentration of capsaicin in a sample.

The technique was refined by the group so that the multi-wall carbon nanotube-based electrode could be screen-printed and used with a hand-held sensor to assess capsaicin concentration

outside the laboratory. To use the device, food extracts containing chilli are diluted in a ratio of 1:100 with 0.05M Britton-Robinson buffer at pH 1.0 and then placed on the carbon nanotube electrode. The voltage is scanned, and the variation in voltammetric stripping current is recorded. The result is then converted into a measurement corresponding to the Scoville scale. The technology was patented in 2008 by Isis Innovation Ltd, the University of Oxford's technology transfer division [4].

The Compton group also developed a similar electrochemical technique using carbon-based electrodes to measure the strength of garlic by identifying the presence of disulfides [5]. This technology was patented in the UK, and by Isis Innovation, in 2010 [6].

Richard Compton has been on the academic staff of the Department of Chemistry from 1985 to the present day.

3. References to the research

Asterisked outputs denote best indicators of quality; University of Oxford authors are underlined.

1. Kachoosangi, R.T., Wildgoose, G.G., Compton, R.G. Sensitive adsorptive stripping voltammetric determination of paracetamol at multiwalled carbon nanotube modified basal plane pyrolytic graphite electrode. *Analytica Chimica Acta* 2008, 618, 54-60. DOI: 10.1016/j.aca.2008.04.053.

2. Kachoosangi, R.T., Wildgoose, G.G., Compton, R.G. Adsorptive stripping voltammetric determination of 4-hexylresorcinol in pharmaceutical products using multiwalled carbon nanotube based electrodes. *Electroanalysis* 2008 20, 1714-1718. DOI: 10.1002/elan.200804258

3. * Kachoosangi, R.T., Wildgoose, G.G., Compton, R.G. Carbon nanotube-based electrochemical sensors for quantifying the 'heat' of chilli peppers: The adsorptive stripping voltammetric determination of capsaicin. *Analyst* 2008,*133*, 888-895. DOI: 10.1039/b803588a
This paper reports the analytical protocol employed in the 'chillimeter'.

4. * Kachoosangi, R.T., Wildgoose, G.G., Compton, R.G. Chilli Sensor Patent, EP 09723101.3 , priority date 18 March 2008 (Isis Project 3670).
https://www.google.com/patents/EP2257795A1?cl=en
This patents the analytical procedure for the determination of capsaicin via adsorptive stripping voltammetry using carbon nanotubes.

5. * Martindale, B. C. M., Aldous, L., Rees, N. V., Compton, R. G. Towards the electrochemical quantification of the strength of garlic. *Analyst* 2011,*136*,128-133. DOI: 10.1039/c0an00706d
This paper reports work stimulated by discussion with the food technology industry into the electrochemical measurement of garlic flavour.

6. Aldous, L., Compton, R. G., Martindale, B. C. M., Rees, N. V. Garlic Sensor Patent, GB 1017625.3, priority date 19 October 2010 (Isis Project 7513).
https://www.google.com/patents/WO2012052755A1?cl=en

4. Details of the impact

Following the patenting of the Compton group's chilli sensor technology by Isis Innovation in early 2008, there were well over a hundred worldwide media reports on the potential of the new technique in newspapers, magazines, journals and online forums (including the Guardian, Nature, the Royal Society of Chemistry News, many chemistry journals and specialist chilli industry publications) [7]. Interest focussed particularly on the objectivity and precision of the measurements obtained, and on the greatly reduced costs in contrast to the traditional method of

using teams of tasters [8, 9]. Isis Innovation prepared the chilli sensor project for commercialisation, and the English Provender Company (EPC) subsequently expressed an interest in the technology for commercial purposes. Using EPSRC Pathway to Impact funds, prototypes were built and a PDRA employed to assist with field trials. The Compton group showed EPC staff how to use the tester on their food samples, and followed up with several visits to monitor progress. Isis licensed the technology to EPC on a non-exclusive basis in 2012 [10].

EPC, a company with a turnover of £ 63.6 M in 2012 and 323 employees [11], produces a wide range of sauces and condiments for use in domestic food, such as their Very Lazy range of pre-prepared ingredients, and is a key supplier of ready meals for the UK's major food retailers. It makes around 250 tonnes of cooked products (ready meals) each month containing chilli, and approximately 200,000 jars of chilli-containing foods. Typically it uses up to 10 tonnes of chillies every month, which vary dramatically in heat; there is thus a need to ensure consistency in both raw materials and finished product. Extensive tests by EPC in its production facilities confirmed that Compton's electrochemical device was accurate and reliable and matched Scoville Test results, a key part of validating the device and convincing the industry to use the method. Following this, EPC has routinely used the sensor as a quality assurance monitor on the incoming raw ingredients. The company can now offer its customers (which include Marks and Spencer, 2 Sisters Food Group and Greencore Group) improved consistency in the products it delivers, and this has been well received by the customers concerned [12]. According to the Head of Technical at EPC, *'the device [chilli tester] has a massive place in our future'*.

In February 2013 the Singapore-based company Bio-X (S) Pte. Ltd. obtained an exclusive licence for the chilli sensor patent in Asia-Pacific and a non-exclusive licence in the rest of the world (again through Isis Innovation). Bio-X works with technologies and products (including chemical sensors) targeted at the measurement of spiciness in food, and aims to establish standardisation for heat in the food industry. Following licensing, Bio-X is designing and building a new commercially available chilli sensor, and demonstration units have been sold prior to full-scale launch. Nestlé have been involved in a trial in which data from Bio-X's Chilli Tester matched with their own data from high performance liquid chromatography - an extremely promising outcome [13, 14]. The CEO of Bio-X has stated the advantages to them of the chilli sensor technology: *'Both suppliers and users of chillies can be assured of the quality by using a simple handheld device. Initially, we expect to see the Chilli Tester being used by food manufacturers to determine the quality of their raw materials and chilli farms to grade their products – but the real potential of the Chilli Tester will be realised in giving the consumer a number that they can use in deciding on sauces and other food products.'* [10]

The science behind the chilli sensors has been the subject of significant outreach activities at several UK schools. Since July 2012 a consortium of seven Yorkshire schools, led by Shelley College in Huddersfield and also involving the University of Huddersfield, has been engaged in an ongoing collaboration and outreach programme with the Compton group, aimed at increasing science applications from such students to research-intensive universities. Students aged 14-15 have successfully used research-level equipment based on the technology described in section 2 to investigate capsaicin levels in fresh chillies and chilli products. They have also used Compton's voltammetry techniques to investigate and quantify the amount of ascorbic acid in supplements and soft drinks. The programme has now expanded to incorporate a chilli-growing project, and selected students have taken part in residential visits to Oxford. The links with the GCSE chemistry curriculum are significant and include reinforcement of topics such as atomic structure and bonding, ionic compounds, covalent structures (carbon), electrolysis, redox reactions and nanoscience. In practical terms the activities give students the chance to improve skills in risk assessment, collection of data, processing and interpreting data, and development of hypotheses and explanations. In addition it assists in preparing them for aspects of the A level Chemistry syllabus, especially in the topics identified above.

The Director of Science at Shelley College confirms that the programme is encouraging more students to pursue chemistry seriously: *'The students from Shelley and the other schools involved in the project could not be more enthusiastic about taking their [chemistry] studies further. The*

whole nation recognises the importance of producing first class scientists for the future and this project is helping to further that aim in a way that should not be underestimated.' The outreach has been supported by funds from St John's College and the Department of Chemistry, University of Oxford, and from the Royal Society of Chemistry [15].

A second schools programme started in early 2013 at Cheltenham Ladies College in Gloucestershire, aided by a Partnership Grant from the Royal Society. Again, with active support from the Compton group, students have used the techniques described in the research section to devise their own experiments investigating capsaicin levels within chilli plants (the strength of dried versus fresh chillies, and which solvent will extract capsaicin more readily). The College's Head of Chemistry feels the project *'has helped push science to the top of the school agenda. A significant proportion of the school's cohort has become engaged with the project.'* In addition the Cheltenham students have particularly benefited from the high proportion of female researchers from the Compton group visiting the school, which has *'served as an excellent motivator for young female scientists'* [16].

Over 100 students in all have so far taken part across the schools involved, and both programmes are continuing.

5. Sources to corroborate the impact

7. A full list of media sources which ran the chilli sensor story is held on file.

8. http://www.nature.com/news/2008/080512/full/news.2008.817.html
May 2008 Nature News article detailing the advantages of the chilli sensor technique and the chemistry behind it.

9. http://www.theguardian.com/technology/2008/jun/12/news.sciencenews
June 2008 article in the Guardian describing Compton's development of the chilli sensor technique and its commercial potential.

10. Isis Innovation can corroborate the details of the licence with the English Provender Company and with Bio-X, details of patent-related income for the University of Oxford, and quotes from the Bio-X CEO on the benefits to them of the chilli sensor technology.

11. Details of English Provender turnover 2012 at http://www.worksmart.org.uk/company/company.php?id=02593588 .

12. The Head of Technical at English Provender Company can corroborate the use of the Chilli Meter in quality assurance of incoming raw materials, and the importance placed on the device by the company and its customers.

13. The Bio-X webpage at http://biox.com.sg/data-book/ confirms the link with the Compton research. The homepage shows the chilli sensor: http://biox.com.sg/ .

14. Bio-X June 2013 progress report to Isis Innovation (held on file), corroborating progress towards a commercially-available chilli sensor, and results of trials including the trial with Nestlé.

15. Letter from the Director of Science at Shelley College (held on file), corroborating details of Compton group outreach activities and impact on students.

16. Email from the Head of Chemistry at the Cheltenham Ladies' College (held on file), corroborating details of Compton group activities and impact on female students.