Institution: University of Oxford

Unit of Assessment: 8 – Chemistry

Title of case study: Electrochemical sensors for food and drink: Ensuring quality and safety

Period when the underpinning research was undertaken: 2008 - 2017

Details of staff conducting the underpinning research from the submitting unit:

Name(s):	Role(s) (e.g. job title):	Period(s) employed by submitting HEI:
Richard G Compton	Professor of Chemistry	1985 – present
Dr Gregory Wildgoose	Junior Research Fellow	2006 – 2009
Dr Christopher Batchelor-McAuley	Postdoctoral researcher	01/04/2012 – present
Dr Kristina Tschulik	Postdoctoral researcher	12/11/2012 – 31/08/2015

Period when the claimed impact occurred: 2017- 31 December 2020

Is this case study continued from a case study submitted in 2014? No

1. Summary of the impact (indicative maximum 100 words)

Professor Compton's research on electrochemical sensors led to development of the ChilliPot, GarlicPot and FoodSense, a series of handheld devices that measure analytes in food, drink and water (including chilli heat, garlic strength and ginger intensity), in collaboration with Zimmer & Peacock (ZP), a UK electrochemical sensor manufacturing company. Existing methods such as organoleptic (taste) testing and high-performance liquid chromatography (HPLC) are time-consuming and costly. Compton's electrochemical sensors enable near instant results and the devices can be used by anyone needing to ensure food quality and safety. Since 2017 the devices have been sold to **[text removed for publication]** companies across the globe, ranging from agricultural businesses and government research organisations, through medium-sized chilli sauce manufacturers to boutique chilli sauce producers. **[Text removed for publication]** The sensors have been instrumental to developing 'The Science of Chilli Heat', a schools outreach programme that has benefitted over 150 key stage 4 and 5 students through workshops and projects in collaboration with Compton's research group. One participating school went on to be recognised in the UK Parliament for its work in encouraging girls to study STEM subjects.

2. Underpinning research (indicative maximum 500 words)

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 next turned their attention to food analytes, initially focusing on chilli. The 'heat' or piquancy of chillies is a result of the molecule capsaicin (8-methyl-N-vanillyl-6-nonenamide) and related compounds called capsaicinoids. Compton's group observed that capsaicin molecules spontaneously adsorbed 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.

Compton realised that there was a particular practical problem that might be solved through this application of AdSV. 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 — but existing assessment methods such as organoleptic (taste) testing and high-performance liquid chromatography (HPLC) are time-consuming and costly. The electrochemical technique in [**3**] had the potential to provide a quick, cheap and reliable test.

The technique was refined by the group so that the multi-wall carbon nanotube-based electrode could be screen-printed and used with a handheld 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. Upon adsorption, capsaicin is electro-chemically oxidised to an ortho-benzoquinone molecule through two irreversible steps. This species then falls into a redox electrochemical loop with a catechol (1,2-dihydroxy benzene) species. It is the equilibrium between these two species which is detected at the carbon nanotube electrode, giving rise to a novel capsaicin detection method. The result is converted into a measurement corresponding to the Scoville scale, which expresses the relative strength of chillies against that of pure capsaicin (16 million Scoville units). The technology was patented in 2008 by Isis Innovation Ltd, the University of Oxford's technology transfer division (now Oxford University Innovation) [**4**]. The adsorptive stripping technique was then extended to the detection of ginger (gingerol), turmeric (curcumin) and vanillin [**5**].

The Compton group also developed an electrochemical technique using carbon-based electrodes to measure the strength of garlic by identifying the presence of disulfides [**6**]. This technology was patented in the UK by Isis Innovation in 2010. The garlic sensor uses a different electrochemical method to that used in the detection of chillies, turmeric and ginger. Bromide ions (Br-) are oxidised to bromine (Br_2) on the surface of a platinum electrode. Br_2 is then reduced by allicin (a pungent species which gives garlic its characteristic smell, making up around 70% of the thiosulfates present in freshly chopped garlic), forming a sulfonic acid and regenerating Br- ions, thus completing the catalytic cycle. The Br-/Br_2 redox system acts as a homogeneous redox mediator so that the recorded electrochemical signal is proportional to the organosulfur concentration present, enabling accurate detection of the garlic concentration.

3. References to the research (indicative maximum of six references)

1. Journal article: R T Kachoosangi, G G Wildgoose, R G Compton. 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. Journal article: R T Kachoosangi, G G Wildgoose, R G Compton. 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. Journal article: R T Kachoosangi, G G Wildgoose, R G Compton. 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
4. Patent: R T Kachoosangi, G G Wildgoose, R G Compton, Chilli Sensor Patent, EP 09723101.3, priority date 18 March 2008: https://www.google.com/patents/EP2257795A1?cl=en
5. Journal article: K Chaisiwamongkhol, K Ngamchuea, C Batchelor-McAuley and R G Compton. Electrochemical detection of gingerol species in ginger (Zingiber officinale) using multiwalled

carbon nanotube modified electrodes, *Analyst*, 2016, 141, 6321-6328. DOI: 10.1039/C6AN02254E

6. Journal article: E M Hall, K Tschulik, C Batchelor-McAuley, R G Compton. Electrochemical bromination of organosulfur containing species for the determination of the strength of garlic (A. sativum). *Food Science*, 2016, 199, 817-821. DOI: 10.1016/j.foodchem.2015.12.086

4. Details of the impact (indicative maximum 750 words)

Pathway to Impact

The research of the Compton group has given rise to electrochemical food sensors that can reliably and quickly test for a range of food analytes. Initially the focus was on developing a method of measuring the spiciness of chilli products without eating them. For over 100 years there was no quantitative procedure to measure the spiciness of chilli peppers, the standard method requiring the use of a panel of five 'expert' tasters. The Scoville organoleptic (taste) test (dating back to 1912) involved dissolving an exact weight of dried pepper in alcohol to extract capsaicin, the molecule responsible for the spice in chillies. Decreasing concentrations of extracted capsaicin were then given to the panel until a majority could no longer detect the heat. The test can give accurate results when performed by expert tasters, but is dependent on the capsaicin sensitivity of the tasters, and time-consuming and costly to perform. More consistent scientific methods of measuring capsaicin content involve cumbersome and expensive high-performance liquid chromatographs (HPLC), which also require trained staff and involve time for sample preparation and results analysis.

Professor Compton's research delivered a test which was quick, reliable and cheap to manufacture. The electrochemical chilli sensor was patented in 2008 [**4**]. In 2010 Beacon Foods (South Wales), an ingredient supplier, challenged Compton to develop a new method to objectively determine the strength of garlic, as opposed to the subjective organoleptic testing they were then currently using [**A**]. The resulting garlic sensor, patented in 2010 [**B**], was initially non-exclusively licensed to Beacon Foods who used it as quality assurance on the garlic that they bought in for processing. Subsequently it, along with the chill sensor and others, was licensed to Zimmer & Peacock (ZP), an electrochemical sensor manufacturing company.

Impact: Commerce and the economy

The development of ChilliPot, GarlicPot and FoodSense

In 2017 an exclusive worldwide licence was agreed between Oxford University Innovation (OUI) and ZP to produce the ChilliPot device, the world's first commercial quick test for measuring the heat of chillies and chilli products. The ChilliPot is marketed at chilli growers, farmers, producers, manufacturers, and food technologists. The success of the ChilliPot led to ZP signing a new licensing deal with OUI for garlic, ginger and turmeric sensors in 2018 [**C**], when ZP produced the GarlicPot, which had its first sale in South Africa later the same year. Around 15,700,000t of fresh garlic is produced worldwide each year and different batches vary significantly in the strengths of their flavour, so the development of such detection methods is important to ensure that garlic concentrations are standardised. In 2019 ZP licensed a 30-minute assay for the rapid detection of E. coli. ZP then utilised Compton's diverse research by creating FoodSense, a multi-analytical device capable of measuring capsaicin in chilli, diallyl disulfide in garlic, gingerol in ginger, curcumin in turmeric, vanilla aldehyde in vanilla, pH and E. coli. [**D**]

The ZP sensors have many advantages over existing sensing methods. All devices are handheld, accessible and easy to use, delivering results to an integrated smartphone app. The Compton Group's use of carbon nanotubes and disposable screen-printed electrodes are key to the accuracy, speed and low cost of the sensors. Readings are given within a minute, vastly quicker than the conventional method of using a panel of taste testers, and also quicker and cheaper than HPLC, which takes 3 days and costs in the region of GBP100 per test [**D**]. E. coli results are available within 30 minutes. This speed is especially important for detecting the presence of E.coli in food, as current methods of pathogen and microbial testing can take 3 days

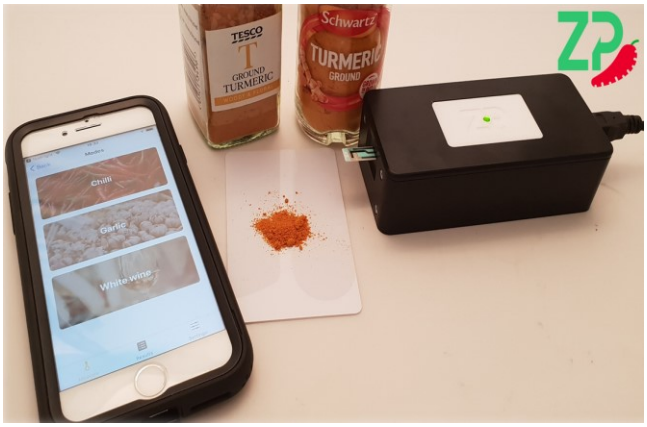
or more, in which time contaminated products risk being shipped to supermarkets and consumers. [**D**]

[Text removed for publication.] The company has additionally adopted the ChilliPot into its analytical service, offering testing at a cost of EUR85 per sample [**F**].

ZP reports very positive end user feedback:

- 'The ChilliPot means not having to pay 170 Euros per HPLC test.'
- 'There is no other practical way of assessing the crop whilst in the field.'
- 'The business can't afford HPLC but we can afford a FoodSense.' [**E**]

A Portuguese food company has used ZP's logo on the bottle of chilli sauce next to the Scoville score as a testament to the reliability of the heat rating. [**G**]



ZP's FoodSense device and accompanying smartphone app (© Zimmer & Peacock)

ZP's licensing of Compton's research has enabled the company to expand its operations into food sensing; it had previously specialised in biosensing and medical diagnostics. ZP's Director states: 'Our long-standing relationship with Professor Richard Compton at Oxford University has enabled us to benefit from his unique expertise in electrochemical sensing. This has given us a positive direction in the food quality and food safety testing market.' [**E**]

Professor Compton's innovative advances on the development of sensors for food technology contributed to him receiving the 2018 Robert Boyle Prize for Analytical Science by the Royal Society of Chemistry, awarded for 'innovative advances in the analytical detection of nano-entities, seminal contributions to the calibration free measurement of pH and the development of sensors for food technology and medicine'. [**H**]

Impact: Understanding, learning and participation among school students

Chilli sensors present an engaging way to encourage school students to participate in practical science activities, to work in collaboration with students from other schools, and with researchers at Oxford. Since September 2013 Professor Compton and his research group have attended schools and organised outreach events on 'chilli science'. The work produced by students at these workshops has included relating the colour of chillies to their heat, the relationship between the chilli anatomy (such as the pith, seeds and flesh) and the local heat content, and the effect of aging on chillies.

2 chilli conferences for schools were held in 2015 and 2016. Compton's research group planned a 6-month 'Science of Chilli Heat' research project for 24 key stage 5 students in collaboration with teachers at Highgate School in North London, acting as a hub for 7 local state schools. They created a programme with demonstrable links to the school curriculum, which also showcased the real-world work of scientists. The programme enabled students to design their own experiments using potentiostats provided by the Compton Group. In parallel, a similar programme was developed for 50 key stage 4 students at state schools in West Yorkshire. The students participated in weekly sessions at their local school or hub, supported by Compton's group, culminating in a conference held at Oxford's Inorganic Chemistry Laboratory where students presented their findings to fellow participants and Oxford researchers.

Over 2 years more than 150 students from 12 different schools benefitted from the 'chilli experience'. Feedback from Shelley College in West Yorkshire noted that the collaborative nature of the research project, in working alongside participants from other schools as well as researchers

at Oxford Chemistry, 'enabled students to develop the skills of collaboration, communication, resilience and often leadership. In addition to this, one student commented that once the presentation was over he felt like he could do anything! I believe this shows that the social development (above probably all else) has really been developed very well by participation in the project.' Students also reported being more interested in continuing to study chemistry at A-level: 'It has given [students] an appreciation that science has applications outside of the classroom and many of the students in the team have expressed interests in taking science at A-level following the project.' [**I**]

Feedback from 10 pupils at Woodkirk Academy, West Yorkshire, who participated in the chilli conference project in 2015 showed that '7 students are more likely to choose science and maths A-levels following the project and 6 are more likely to want a career in science.' [**I**] Woodkirk Academy went on to be recognised in a parliamentary debate on A-level STEM subjects for 'excellent work' in encouraging girls to study STEM subjects and promote STEM careers [**J**]. The Minister for Women and Equalities noted she 'was impressed to see at first-hand how the academies engage pupils in STEM subjects, demonstrating the application of science and maths and promoting STEM careers'. In their response to parliamentary recognition, Woodkirk cited the Oxford collaboration as a contributory factor of their success: 'Woodkirk is also involved in exciting research together with Oxford University. Over 40 students are currently working on a research project investigating what factors affect the spiciness of chillies with the Compton Electrochemistry Group at Oxford University. Academics from Oxford University travelled to West Yorkshire to conduct troubleshooting on these projects and inform students about cutting-edge research into nanochemistry and electrochemistry… South Devon Chilli Farm have also asked Woodkirk students to investigate the spiciness of their newest chilli varieties.' [**K**]

5. Sources to corroborate the impact (indicative maximum of 10 references)

A. Press release from Beacon Foods, corroborating their collaboration with Professor Compton's research group at Oxford University (11 Jul 2016)
B. Garlic Sensor Patent, GB 1017625.3, priority date 19 October 2010 (OUI Project 7513), subsequently granted as Singapore patent number 189458.
C. Press release: OUI announcement of ZP licensing of garlic, ginger and turmeric sensors (22 Jun 2018)
D. ZP website corroboration of products and performance (ChilliPot, GarlicPot, FoodSense and E.coli tests)
E. Letter from the Director of ZP confirming sales and customer feedback (27 Sep 2020)
F. ZP website showing ChilliPot being offered as part of ZP's analytical testing service
G. ZP website highlighting customer feedback (15 Sep 2018)
H. 2018 Robert Boyle Prize in Analytical Science awarded to Professor R G Compton.
I. Feedback and evaluation report from 2 schools participating in outreach programme, corroborating the benefits for students.
J. Hansard report on parliamentary debate on STEM subjects, corroborating the recognition of Woodkirk Academy in parliament (25 Feb 2016)
K. Woodkirk Academy website acknowledging the Oxford chilli collaboration as a key part of their STEM programme in response to recognition from the UK Parliament (2016)