Food Institutional Research Measure

Final Report

‘Smart packaging systems containing novel optochemical O₂ and CO₂ sensors for the food industry’ (SMARTPACK)

DAFM Project Ref No: 11F015
Start date: 01/01/2013
End date: 30/09/2016

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Prof. Joseph P. Kerry, School of Food and Nutrition Science, University College Cork

Please place one ‘x’ below the appropriate area on the research continuum where you feel this project fits

<table>
<thead>
<tr>
<th>Basic/Fundamental</th>
<th>Applied</th>
<th>Pre Commercial</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>2</td>
<td>3</td>
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<tr>
<td>4</td>
<td>5</td>
<td>X</td>
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<td>6</td>
<td>7</td>
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</tbody>
</table>

Please specify priority area(s) of research this project relates to from the National Prioritisation Research Exercise* (NRPE) report;

<table>
<thead>
<tr>
<th>Priority Area (s)</th>
<th>I - Sustainable Food Production and Processing; L - Manufacturing Competitiveness; M - Processing Technologies and Novel Materials</th>
</tr>
</thead>
</table>

Key words: Optical oxygen and CO₂ sensors, food safety, quality control, non-destructive measurement, modified atmosphere packaging, new sensor materials and technologies
1. **Rationale for Undertaking the Research**

**SENSOPACK** was aimed at solving real problems of the packaging industry, especially food packaging. Consumers want good quality and safe products; retailers expect long shelf-life; food inspectors require safe and hygienic manufacturing practices, adequate product labelling and compliance with national regulations, whilst food producers demand efficient sensor technologies to meet consumer and regulatory expectations and to reduce product costs. Non-destructive real-time monitoring of food quality throughout the retail chain is principally possible, but still has not been implemented on a significant scale. The most important food quality and safety parameters which require monitoring in packs are: residual O₂, CO₂, humidity. This however requires adequate tools that can analyse non-destructively, quickly and cost-effectively a large number of packs having different shape, size and appearance, and provide quantitative and interactive readout.  

**SENSOPACK** was set to develop novel optochemical sensors for O₂ and CO₂, which possess new functionalities, smart features, green chemistries and fabrication technologies tailored to large-scale food packaging applications. Such high-tech sensors for key parameters of packaged products, their components and know-how built into such materials will provide an efficient and affordable means of assessing the freshness of foods, thus enhancing protection of consumers and producers and safety of food products by timely detecting deteriorative changes. Currently, neither consumers nor retailers have effective means to test the quality of the packages in store and at the point of purchase. We also planned to demonstrate performance of these new sensors with a range of different food products and prove their efficiency in improving the control and quality assurance of packaged food products. A number of such sensors have been developed in recent years under FIRM projects ‘Application and further development of non-destructive oxygen sensing technology within the Irish food and food packaging industry’ (06/R&D/C/82), New analytical systems for quality and safety assessment of packaged foods based on optical oxygen sensing (00/R&D/C/468), Development of a novel CO₂ sensor for food packaging applications (Smartpack, 08/RD/C/642). The utility of these prototype sensor systems has been demonstrated in trials with foods, however they could not make way to a wider practical use and commercialisation, due to their non-optimal properties, operational performance, compatibility with the packaging systems, high costs and lack of dedicated instrumentation. Nonetheless, the current size of the food packaging market is $60 Billion p.a. shows their high potential and need for development.

2. **Research Approach**

Several new sensor technologies, particularly polymer extrusion and solvent crazing, were researched with the view to produce new types of O₂ and CO₂ sensors and materials dedicated for intelligent food packaging. These materials were expected to utilise ‘green’, safe, environmentally friendly and scalable sensor chemistries, polymeric materials and fabrication technologies, be compatible with existing instrumentation, such as the Optech® reader for O₂ or visual colour change for CO₂, and operate in various conditions and be integrated in various packs and product types. This strategy has addressed the main bottlenecks of the existing sensor materials allowing development of new materials with improved scalability, processability and reduced costs.  

The main objectives of the SENSOPACK project were:
- To develop new optochemical sensors for O₂ and CO₂ based on hot polymer extrusion processes;
- To develop nanostructured polymer fibre materials functionalised with O₂ sensors using solvent-crazing technology;
- To apply sustainable and environmentally friendly polymers in such sensor and packaging materials;
- To fully characterise the new smart packaging materials for use with packaged foods;
• To assess the new sensors and smart packaging materials in laboratory and industrial trials with packaged food samples, such as muscle-based meats and other products produced by Modified Atmosphere Packaging (MAP).

These five major SENSOPACK objectives were realised through six scientific tasks and one task focusing on Dissemination.

3. Research Achievements/Results

The team has developed and characterised new types of optochemical sensors tailored for food packaging applications, namely: O₂ sensors based on high density poly(ethylene) (HDPE) films impregnated by local solvent-crazing, microporous polymeric fabric materials impregnated by swelling or discrete spotting of solvent cocktails; O₂ sensors without noble metal based indicators based on halogenated boron-dipyrromethene (BODIPY) dye embedded in polysulphone by spot casting or in microporous polypropylene (PP) by solvent swelling; extrudable O₂ and CO₂ sensor materials fabricated by polymer extrusion at Queen’s University of Belfast (QUB); discrete HDPE and polyphenylenesulphide (PPS) O₂ sensors produced on an industrial automated puncturing machine by solvent-crazing method. We also screened and evaluated comparatively a range of green and sustainable polymer materials (poly(lactic acid), PPS, poly(caprolactone)) for their use in O₂ and CO₂ sensors and smart food packaging systems. Studies were carried out to assess production scalability for these sensors and their integration into conventional packaging materials and new smart packaging systems. Several small and medium scale laboratory and industrial trials with packaged raw meat products and beverages, were carried out with our industrial partners, to whom the O₂ and CO₂ sensor technologies were presented and demonstrated. Thus, O₂ sensor systems were applied to test performance of their packaging systems and processes, perform troubleshooting and process optimisation, quality control/quality assurance (QC/QA) and benchmarking with alternative systems currently in use.

Experimental results on these new O₂ and CO₂ sensor systems provided the basis for 17+ papers in high-impact journals, 9 international conferences and one invention disclosure on extrudable sensors filed at UCC. A number of contacts were made with industry on demonstration and evaluation of these sensor technologies and on the possibilities of their further development, commercialisation and adoption by industry.

4. Impact of the Research

SENSOPACK provided a new technological platform for smart multi-functional packaging materials incorporating optical O₂ and CO₂ sensors, custom-tailored for non-destructive assessment of packaged foods for residual O₂ and CO₂ content. This project generated a large amount of new knowledge and IP, which facilitate rapid dissemination and commercialisation of these sensor technologies. However, these ultimate goals still require an additional R&D effort and resources. SENSOPACK outcomes have the potential to transform current food packaging practices (estimated at $60 bn p.a.), by providing a previously unachievable 100% control and quality assurance of various MAP foods for residual O₂ and/or CO₂ levels, at very affordable costs. This will provide the customer (i.e. retailer and consumer) enhanced reassurance of the safety and freshness of the contents of any food package. Therefore, SENSOPACK results and practical outcomes span the many priority areas listed in the FIRM Programme and address many of the anticipated impacts.

4 (a). Summary of research outcomes:

(i) Collaborative links developed during this research

1. UCC team established collaboration with researchers from the Polymer Chemistry Department of Moscow State University on polymer solvent crazing technology which was transferred to Cork and developed further with respect to new sensor materials and processing methods. It
was actively used in project work for the fabrication of novel O₂ sensors, their optimisation and up-scaling for food packaging applications. Three joint papers were generated.

2. Collaborations on the use of BODIPY dyes in O₂ sensors were established with Siberian Physical-Technical Institute of Tomsk State University, Tomsk, Russia and Complutense University of Madrid, Spain.

3. Collaboration was established with BioPlasma Research group, School of Food Science and Environmental Health, Dublin Institute of Technology on the application of phosphorescent oxygen sensors in in-package dielectric barrier discharge plasma environment.

(ii) Outcomes where new products, technologies and processes were developed and/or adopted

1. Control and optimisation of in-house bulk vacuum packaging process used for raw meats with disposable phosphorescence based O₂ sensors and Optech® reader (Mocon, USA). Shown to be a convenient non-destructive QC/QA tool for mapping local O₂ levels in multiple different locations in bulk vacuum packaging of raw beef

2. Non-destructive monitoring of residual O₂ levels and dynamic changes throughout shelf life in retail-grade meat cuts packaged under industrial settings, using disposable phosphorescence based O₂ sensors and Optech® reader

3. Performance evaluation of packaging materials containing new anti-microbial coatings under industrial vacuum packaging conditions with medium size batches meat cuts, disposable O₂ sensors and Optech® reader (Mocon, USA)

4. Performance evaluation and comparison of the different MAP machines and packaging lines (old, new and custom-modified packagers) using industrial meat samples, disposable O₂ sensors and Optech® reader (Mocon, USA)

5. High-throughput non-destructive quality assessment of several bottle closures and packaging processes used by an Irish beverage company (SME) with their products, and subsequent optimisation of these closures and process parameters for the given product and storage conditions using disposable O₂ sensors.

6. Testing of microbial contamination of non-carbonated beverages using the colorimetric CO₂-sensitive materials by a large beverage company in Europe.

(iii) Outcomes with economic potential

1. Three trials on sensor use were conducted jointly with an Irish industrial partner: i) on bulk meat vacuum packaging; ii) shelf life studies of packaged meat cuts; iii) performance evaluation and cross-comparison of their three different vacuum packaging machines and packaging lines; iv) use of anti-microbial coatings to extend product shelf life. This work enables process control and optimisation.

2. Trials with an Irish beverage company using sensors to assess performance and quality of their packaging containers (bottles) and screw-cap closures currently used with their products. Joint troubleshooting and optimisation of these packaging materials and process conditions.

3. The new CO₂ sensor technology was presented to a large MNC and discussions about possible joint projects are on-going.

4. A joint project (shared BSc student) was arranged with an Irish SME on the evaluation of the new CO₂ sensors for detection of microbial growth.

5. Targeted project was conducted with industrial partner (Irish SME) on the optimisation and control of dedicated bottle closures for their products.

(iv) Outcomes with national/ policy/social/environmental potential

None

4 (b) Summary of Research Outputs
(i) Peer-reviewed publications, International Journal/Book chapters.


(ii) Popular non-scientific publications and abstracts including those presented at conferences

2. Papkovsky DB, Imaging of tissue oxygenation with phosphorescent oxygen (nano)sensors, European Conference on Optical Chemical Sensors and Biosensors, April 12-16, 2014, Athens, Greece. (poster presentation)
8. Papkovsky DB, Dmitriev RI, Zhdanov AV, Sensing and imaging of cellular oxygen with phosphorescent nanosensors, Proceedings of 16th International Meeting on Chemical Sensors, July 10-13, Jeju, South Korea. (invited talk)
9. Yusufu D, Mills A, Highly CO$_2$ sensitive, extruded fluorescent plastic indicator film based on HPTS, Europrotrode-2016, March 20-23, Graz, Austria (poster).

(iii) National Report
None

(iv) Workshops/seminars at which results were presented

1. SENSOPACK researchers participated in ‘Meat Gateways’ conference at Teagasc, Ashtown, Co Dublin, May 21, 2014, demonstrated our sensor platforms at the exhibitor booth to multiple food companies (~30 of local companies and MNCs) and participated in networking session.
2. The new O$_2$ sensors and smart packaging materials were demonstrated to three instrumentation and packaging companies from Ireland, Europe and USA.
3. Technology Workshops with industry were conducted in April 2015, November 2015 and September 2016. The new active and smart packaging technologies developed in this project were initially presented (in general terms) to Industry during the development of the Meat Technology Centre (workshop in 2015, Ashtown). One Irish company responded, with whom the team organised a targeted workshop at their premises in 2015/2016. As an outcome of this workshop, two joint pilot trials were planned with company process specialists. The trials have been successfully completed by UCC researchers. Their outcomes were discussed at another workshop at Dawn Charleville, with participation of technologists and QC/QA specialists from company’s other plants. This work was co-ordinated with UCC School of Food Science and Nutrition working on another FIRM project.

(v) Intellectual Property applications/licences/patents

1. An IDF on extrudable O$_2$ sensors, filed on October 26, 2016 (Ref. UCC-16-67). Now under consideration by UCC TTO for patenting and licensing.

(vi) Other

The new IP and sensor materials developed under SENSOPACK were presented to six companies (food/beverage packaging, analytical equipment and sensors), who are now evaluating the sensor technologies with respect to their commercial application and licensing. The improved performance, flexibility, production costs, as well as the EU labelling laws forcing companies to look more closely at the entire supply chain (from farm to fork), make these sensor solutions attractive.
5. **Scientists trained by Project**

Total Number of PhD theses: __2__

1. Caroline Kelly (UCC) ‘Novel luminescent oxygen sensor systems for smart food packaging’, PhD awarded in March 2017

Total Number of Masters theses: __0__

6. **Permanent Researchers**

<table>
<thead>
<tr>
<th>Institution Name</th>
<th>Number of Permanent staff contributing to project</th>
<th>Total Time contribution (person years)</th>
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<tbody>
<tr>
<td>UCC</td>
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<td>QUB</td>
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Total 3 0.58

7. **Researchers Funded by DAFM**

<table>
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<tr>
<th>Type of Researcher</th>
<th>Number</th>
<th>Total Time contribution (person years)</th>
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<tbody>
<tr>
<td>Post Doctorates UCC</td>
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<tr>
<td>Post Doctorates QUB</td>
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<td>Researchers</td>
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<tr>
<td>PhD students UCC</td>
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<td>3.17</td>
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<tr>
<td>PhD Students QUB</td>
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<td>3.00</td>
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<tr>
<td>Masters students</td>
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<td>Temporary researchers</td>
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<td>Other</td>
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Total 6 9.8884

8. **Involvement in Food Graduate Development Programme**

<table>
<thead>
<tr>
<th>Name of Postgraduate / contract researcher</th>
<th>Names of and dates of modules attended</th>
</tr>
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<tbody>
<tr>
<td>Caroline Kelly (PhD)</td>
<td>1. Hot Topics in Agri-Food Research – October 2015</td>
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<tr>
<td></td>
<td>2. Agri-Food Career Management – October 2015</td>
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Caroline Kelly also attended Advanced Study Course on Optical Chemical Sensors, Porto, Portugal (July 2015), which contained informative lessons on many different aspects of sensor design, application and commercialisation, in addition to encouraging inter-disciplinary co-operation by way of team projects and trouble-shooting groups. C. Kelly was in a team which won the competition of projects and received a fee waiver for Europtrode-2016 conference in Graz, Austria.

9. Project Expenditure

<table>
<thead>
<tr>
<th>Total expenditure of the project</th>
<th>€446,506</th>
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<tr>
<td>Total Award by FIRM</td>
<td>€444,493</td>
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<tr>
<td>Other sources of funding (specify)</td>
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In-kind contribution by the industrial partners involved in SENSOPACK was in excess of €25,000. Thus, our food company partner provided large quantities of meat samples, and analytical and Biotech company partners provided sensor materials and access to their imaging instrumentation, which were actively used in the project. We also received access to polymer solvent-crazing technologies and products from Moscow State University (Russia), to plasma treatment equipment at DIT (Ireland) and to new O₂-sensitive dyes in Spain and Russia – all at cost-free basis (estimated in-kind contribution of approximately €6,000).

**Breakdown of Total Expenditure**

<table>
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<tr>
<th>Category</th>
<th>UCC</th>
<th>QUB</th>
<th>Institution 3</th>
<th>Institution 4</th>
<th>Total Project</th>
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<tr>
<td>Contract staff</td>
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<td>n/a</td>
<td>147,320</td>
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<tr>
<td>Temporary staff</td>
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<td>n/a</td>
<td>n/a</td>
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<tr>
<td>Post doctorates</td>
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<td></td>
<td>n/a</td>
<td>n/a</td>
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</tr>
<tr>
<td>Post graduates</td>
<td>69,890</td>
<td>67,663</td>
<td>137,553</td>
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<tr>
<td>Consumables</td>
<td>27,168</td>
<td>24,541</td>
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<tr>
<td>Travel and subsistence</td>
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<tr>
<td><strong>Sub total</strong></td>
<td>251,897</td>
<td>93,840</td>
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<tr>
<td>Durable equipment</td>
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<td>n/a</td>
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<tr>
<td>Other</td>
<td>75,569</td>
<td>25,200</td>
<td>100,769</td>
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<td><strong>Total Expenditure</strong></td>
<td>327,466</td>
<td>119,040</td>
<td>446,505</td>
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</table>

10. Leveraging

Project PI received funding from the Science Foundation Ireland: €500k over six years starting in 2013 to run of the platform projects at the Irish Photonics Integration Research Centre, IPIC (Tyndall-UCC) as a PI, and also SFI Catalyst Award in 2015 (€16k) to build research networks and consortia for H2020 projects. These funding and R&D activities are synergistic to those of the FIRM project.
11. Future Strategies

The team is determined to continue this research. We are constantly looking for other sources of funding for this work, including the 2017 FIRM call, H2020 program, industrial funding and other means. Thus, UCC Co-PI has recently received Teagasc Internally Funded Walsh Fellowship award "Novel ingredients and processing technologies (INGRETECH) for the improved quality of processed meat products", which will fund a PhD student working on related topic. We retain active membership of the Meat Gateways consortium/network, the Irish Photonics Integration Centre (UCC/Tyndall) and also continue active engagement with our industrial partners. We regularly run feasibility projects and trails with industry, plan and prepare new R&D projects on optical sensor development and application. Contacts with the Irish food industry and multinationals were very active during the project (see above) and remain at this level after its completion. Commercialisation of our extrudable O2 sensor technology (IDF filed in 2016) is under evaluation by UCC TTO. Several companies are now considering these technologies for use with their products and processes. Additional R&D funding is being sought to develop them further and make more attractive for industry and commercialisation.