

Institution: University of Nottingham

Unit of Assessment: 6; Agriculture, Veterinary and Food Science

Title of case study: Creation of new ingredients for the food industry

1. Summary of the impact

The University of Nottingham (UoN) has developed two novel food-allowed additives based upon xanthan gum. The generation of these structurally modified forms allow xanthan to be used more efficiently in food manufacturing applications and provide nutritional and health benefits. The invention of the new xanthans benefits the global food industry by facilitating new product development and formulation.

2. Underpinning research

Key researchers:

Prof John Mitchell: Professor of Biomaterials Processing (UoN;1974-2008) Prof Sandra Hill: Professor of Biomaterials Processing (UoN;1990-present) Dr Imad Farhat: Senior Lecturer (UoN;1994-2006) Dr Joanne Hort : Associate Professor in Sensory Science (UoN;2002-present) Dr Bettina Wolf: Associate Professor in Biomaterials Science (UoN;2006-present) Dr Tim Foster: Associate Professor and Reader in Food Structure (UoN;2007-present)

Dr Colin Melia: Associate Professor (UoN;1988-present)

Thickening agents are widely used in the food industry as additives to modify texture and taste properties. Xanthan gum is an important food hydrocolloid thickener, which was approved for use in food in the US in 1969 and the EU in 1982. The current annual market for xanthan gum is estimated to be US\$900M, split approximately equally between food and non-food use. This hydrocolloid is unique as its molecular solutions show weak gel properties, providing both suspension and flow behaviour e.g. allowing the suspension of herbs in a vinaigrette salad dressing. However, currently used grades of xanthan disperse poorly in water, a problem that the industry has battled with since the material was first commercialised. This prevents use of xanthan at high concentrations. Work at UoN has addressed two important problems that are associated with the use of xanthan gums.

Using extrusion technology UoN and CSM (a major manufacturer of baked products)-sponsored projects **[c]** showed the production of a physically modified xanthan **[A]** that is easily handled and readily dispersible. The physical modification aids the dispersion of the xanthan, but does not convert the parent hydrocolloid, as subsequent heating and cooling converts the dispersion to a solution matching the physical properties (viscosity and coil-helix transition temperature) of a control material **[1]**.

Increasing viscosity with the xanthan gums currently used by the food industry reduces the sensory perception of taste, requiring addition of higher amounts of salt or sugar to compensate. Salt reduction is an important target for many food producers and the use of extruded xanthan enables an improved tastant delivery, allowing reduced formulation levels. In work sponsored by Nestle and DEFRA [a] food scientists at UoN demonstrated that if food is thickened by swollen particles rather than a solution of a biopolymer flavour/taste delivery is retained at high viscosities [2]. A new extruded xanthan developed by UoN converts the polymeric macromolecule into a particulate form [1], which gives a product microstructure with improved salt release. Starch (energy density) reduction is also a growing requirement for industry to combat obesity and type-2 diabetes. The new extruded xanthan has functional properties on cooking performance (thickening on heating) and in-mouth dispersibility for mouthfeel and taste delivery **[a & d]**, allowing starch replacement. The processing of food biopolymers can be understood using principles that are well-established for synthetic polymers. The research, together with the above mentioned grants and the EU funded STEP-ITN programme [e], has created an understanding of control of amorphous-crystalline transitions [3]. This has led to the development of a new form of xanthan, produced by a novel structural rearrangement of the native helical linkages using melt extrusion cooking, a processing technology first developed for synthetic polymers (product name 'Hydraxan').



A second industry problem, which spans food and pharmaceutical industries, is that upon dilution (by bodily fluids during mastication and digestion) structures tend to lose texture and viscosity. In projects funded by Reckitt Benckiser a second invention **[B,C]** converted xanthan gum to a liquid crystalline form **[4 & 5]**. This thickens upon dilution as the xanthan concentration decreases **[6]**. Subsequent work involving a clinical trial, showed that this product (Thixate) suppressed appetite following consumption in a test meal. This led to a UK patent filing for Thixate as a satiety gel **[D]**.

3. References to the research

Evidence of the international quality of the research is indicated by publication in international, peer-reviewed journals. Food Hydrocolloids is ranked in the top 7% of food science journals (based on impact factor). Since its inception in 1965, Carbohydrate Research has gained a reputation for its high standard and wide scope which includes all aspects of carbohydrate chemistry and biochemistry, and the Gums & Stabilisers series is an important academia-industry conference where new concepts are debated – the conference series is in its 34th year.

- 1. Sereno, N.M., Hill, S.E. and Mitchell, J.R. (2007) Impact of the extrusion process on xanthan gum behaviour. *Carbohydrate Research* Volume: 342 Issue: 10 Pages: 1333-1342 DOI: 10.1016/j.carres.2007.03.023
- Ferry, A.L., Hort, J., Mitchell, J.R., Cook, D.J., Lagarrigue, S. and Pamies, B.V. (2006) Viscosity and flavour perception: Why is starch different from hydrocolloids? *Food Hydrocolloids* Volume: 20 Issue: 6 Pages: 855-862 DOI: 10.1016/j.foodhyd.2005.08.008
- 3. Paes, S.S., Sun, S., MacNaughtan, I.R., Ganster, J., Foster, T.J. and Mitchell, J.R. (2010) The glass transition and crystallization of ball milled cellulose. Cellulose 17(4), 693-709. DOI 10.1007/s10570-010-9425-7
- Boyd, M.J., Hampson, F.C., Jolliffe, I.G, Dettmar, P.W., Mitchell J.R. and Melia, C.D. (2009) Strand-like phase separation in mixtures of xanthan gum with anionic polyelectrolytes. Food Hydrocolloids Volume: 23 Issue: 8 Pages: 2458-2467 DOI: 10.1016/j.foodhyd.2009.07.008
- Lad M.D., Samanci, S., Mitchell, J.R. and Foster T.J. (2012) Viscosity development during competitive hydration of starch and hydrocolloids. Gums and stabilisers for the Food Industry 15, RSC, 126-136. DOI:10.1039/9781849730747-00126
- Foster, T.J. and Mitchell J.R. (2012) Physical modification of xanthan gum. Gums and Stabilisers for the Food Industry 16, RSC Publishing, 77-88. DOI:10.1039/9781849734554-00077

Underpinning research projects:

- a. 2003-2006: Relationship between rheology, mouthfeel and flavour perception of starch. PI John Mitchell. Sponsor Nestle, £60,000.
- b. 2002-2003: Influence of Alternative Product Formats on Key Ingredient Functionality. PI John Mitchell. Sponsor CSM, €85,000.
- c. 2003-2004: Characterisation of Biopolymer Blends. PI John Mitchell. Sponsor CSM, €60,000.
- d. 2005-2009: European Polysaccharide Network of Excellence (EPNOE). PI John Mitchell. Sponsor EU FWVI, €150,000.
- e. 2008-2012: Shaping and Transformation in the Engineering of Polysaccharides (STEP-ITN). PI Tim Foster. Sponsor EU FWVII, €860,000.
- f. 2001-2004: The phase behaviour of xanthan based biopolymer mixtures. PI C Melia (Pharmaceutical Sciences, UoN) Sponsor Reckitt Benckiser / FMC Corp £65,000.

Patented technology:

- A. Farhat, Hill, Mitchell, Scharf, Sereno and Stolz, Water-dispersible xanthan gum containing comnposition, WO2006/065136.
- B. Foster, Mitchell and Lad, Xanthan gum and swellable particulate containing composition and uses thereof, WO2010/122332.
- C. Boyd, Mitchell, Melia, Jolliffe and Dettmar, Thickenable compositions, WO2004/096906.
- D. Foster, Park and Gaunt, Satiety gel, GB1311027.5.



Professor Mitchell received the Food Hydrocolloids Trust Medal at the 2008 International Hydrocolloids conference held in Singapore, and Foster, Mitchell and Lad received the IChemE Award for Innovation and Excellence in the Food and Drink sector in 2009. Runner-up in the Food and Drink INet Innovation award (2009 & 2012). Foster was awarded an EPSRC Centre for Innovative Manufacturing in Food (2013-2018) worth £5.62m, in collaboration with University of Birmingham and Loughborough University.

4. Details of the impact

The use of thickening agents in packaged food materials is widespread and as the global market for pre-packaged foodstuffs is worth in excess of US\$3 trillion, developments in thickening technology are of considerable economic benefit to manufacturers and processors. The range of thickeners that is in current use is generally based upon polysaccharides (starches, vegetable gums including xanthan gum), or proteins (collagen, gelatin). Thickeners are selected depending upon their suitability in specific applications, related to differences in texture, appearance, taste and responses to chemical and physical conditions. The research of UoN has resulted in the creation of two new forms of xanthan gum (Hydraxan and Thixate) which will benefit industry by overcoming problems with unmodified xanthan gum, and enabling the formulation of foodstuffs with enhanced taste, texture and nutritional properties. This research has been enabled by the close partnerships that the University has with the industry.

<u>Hydraxan</u>

As a result of the recognised expertise of the Food Science group at the University of Nottingham, CSM supported three research projects to a value of £500k over the period 2002-2004 with a focus on optimum delivery of functional ingredients. UoN had a long-established interest in developing a new class of food additives/ingredients using expertise on extrusion and hydrocolloids and this led to the development of Hydraxan. Production trials with Dupont (a major ingredient manufacturer) held in 2008, were successful. In a statement, the Senior Principal Scientist, Hydrocolloids Science at Dupont stated that "*In 2008 Danisco (now DuPont) entered into an agreement with Nottingham University to evaluate proprietary technology relating to a novel formulation of Xanthan gum, cf. the patent application which is jointly owned by the University WO/2006/065136 regarding "Water-Dispersible Xanthan Gum Containing Composition". The objectives of the trials were to demonstrate that the novel readily dispersible xanthan could be made from DuPont xanthan, to see if the process could be scaled up and to provide kg quantities of the material for further application trials. These objectives were met in full' (Source 1).*

Work to scale up production has been a key focus of work with Hydraxan in the commercial sector. The patent was granted in 2010 in several countries, including the UK, and on 9th October 2012 was exclusively licenced to Biopolymer Solutions Ltd, an East Midlands SME. The technology has been given a boost as a result of further recent DEFRA LINK funding "Development of physically modified hydrocolloids for enhanced taste perception" (start date March 2012) and work is currently being carried out by CP Kelco, a US company with major expertise on xanthan. In a statement (2013), the Distinguished Research Fellow of CP Kelco said "Over the last six months" we have collaborated with the staff at the University of Nottingham to understand Hydraxan; a new form of xanthan developed by the University. CP Kelco intends to collaborate with the University of Nottingham and the French company Clextral for a large scale trial in early 2013. This trial is intended to provide a 100 Kg or larger sample of Hydraxan for commercial trials. The Hydraxan process developed at the University is intended to provide a different texture as compared to the unprocessed xanthan gum. Our work with rheological properties and optical microscopy indicates that a Hydraxan solution is composed of varying amounts of molecularly dispersed polymer along with another phase of swollen but still associated particles or soft bits of gel. Compared to other composites, Hydraxan is unique since the composite is formed of a single material; some of the thickening comes from molecularly dispersed xanthan and some comes from highly swollen small particles" (Source 2). Further process scale up work with Clextral (a major extruder manufacturer) (Source 3) to levels of 50kg/hr was successfully completed in January 2013. 250kg of xanthan was processed showing that the structural transformation discovered by the University had occurred in the process and that product structures can be tuned by varying the process conditions. Work on performance in sauces is being driven by Dovedale Foods, another East



Midlands SME, who are streamlining production processes, making high quality production accessible to the SME community (SMEs make up 90% of the UK food manufacturing sector).

Although the use of Hydraxan is at an early stage the food industry considers it to have major potential in both human and animal foods. A statement from the Physical Chemistry and Gels Manager at Mars Petcare confirmed that "*There are a number of factors that inhibit higher usage of unmodified xanthan in our processes and products. This modified raw material would help significantly to overcome those issues and that is why we are interested to pursue it further"* (Source 4).

<u>Thixate</u>

The catalyst for the development of Thixate was a request by Reckitt Benckiser (a pharmaceutical/healthcare company) to use UoN expertise on hydrocolloids to understand an observation of the unusual behaviour of xanthan in a major product system (underpinning research project f). While the initial work [C] has not yet been exploited the patented opportunity would have value in over the counter markets worth \$33bn. The initial work was extended to the very important and novel area of xanthan-particulate blends - termed 'Thixate'. It has been possible to integrate the two innovations by blending the "current" molecular xanthan with the novel particulate form prepared by the extrusion process. Patents have been filed by UoN on the work and in-product studies are currently being undertaken by Eminate (a wholly-owned subsidiary of the University of Nottingham). This collaboration has led to a third patent being filed and negotiations are underway for uptake by Dupont (Source 1). In a statement (2013), the Managing Director of Eminate stated "Over the last 24 months, Eminate has been working with Dr Tim Foster at the University of Nottingham to develop one of his patents (related to xanthan) into a commercial product. Building on the underpinning research work completed by Dr Foster and his research group on the fundamental science, the Eminate-led team has now developed a series of prototypes in a product range under the brand name Thixate. This range is a series of foods and drinks which contain the Thixate ingredient as described in the patent and have been shown to provide consumers with a feeling of satiety and reduced calorie intake at the next meal" (Source 5). Additional opportunities expanding the thixate technology into controlled release of nutrients and drugs have been identified, which product range is one of the core components of Eminate's commercial strategy, providing "commercially-viable, innovative ingredients for health" (Sources 5,6). The Thixate technology will be applied to use of concentrates for distributed manufacture of food, closer to the consumer: to cut down on waste and decrease distribution costs of foods containing high volumes of water. Work in this area resulted in the IChemE Award for Innovation and Excellence in the Food and Drink sector in 2009.

5. Sources to corroborate the impact

- 1. Senior Principal Scientist Hydrocolloids Science, Dupont Health and Nutrition. *Provides* corroboration for the trial production of Hydraxan for further applications. 2013.
- 2. Distinguished Research Fellow of CP Kelco. *Provides corroboration for the commercial production of Hydraxan in partnership with Clextral.* 2012.
- 3. Vice President Technology, Clextral, a division of Groupe Legris Industries, France. *Provides* corroboration for the commercial production of Hydraxan. 2013.
- 4. Physical Chemistry and Gels Manager, Mars Global Petcare. *Provides corroboration of the potential uptake of Hydraxan by industry.* 2013.
- 5. Managing Director of Eminate Limited. *Provides corroboration for the commercial production of Thixate and the potential use food ingredient to provide consumers with feeling of satiety and reduced calorie intake. Confirms the inclusion of Thixate technology in the commercial strategy of the company.* 2013.
- 6. <u>http://www.nottingham.ac.uk/Biosciences/documents/community/public-talks/eminate-23-jan-2013.pdf</u> Presentation by Research and Commercialisation Director, Eminate. *Provides corroboration that Eminate, a wholly owned subsidiary of UoN, are focused on Thixate as a product for licensing and market as a satiety agent.* 2013.